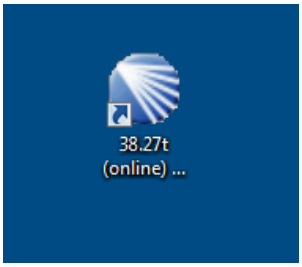
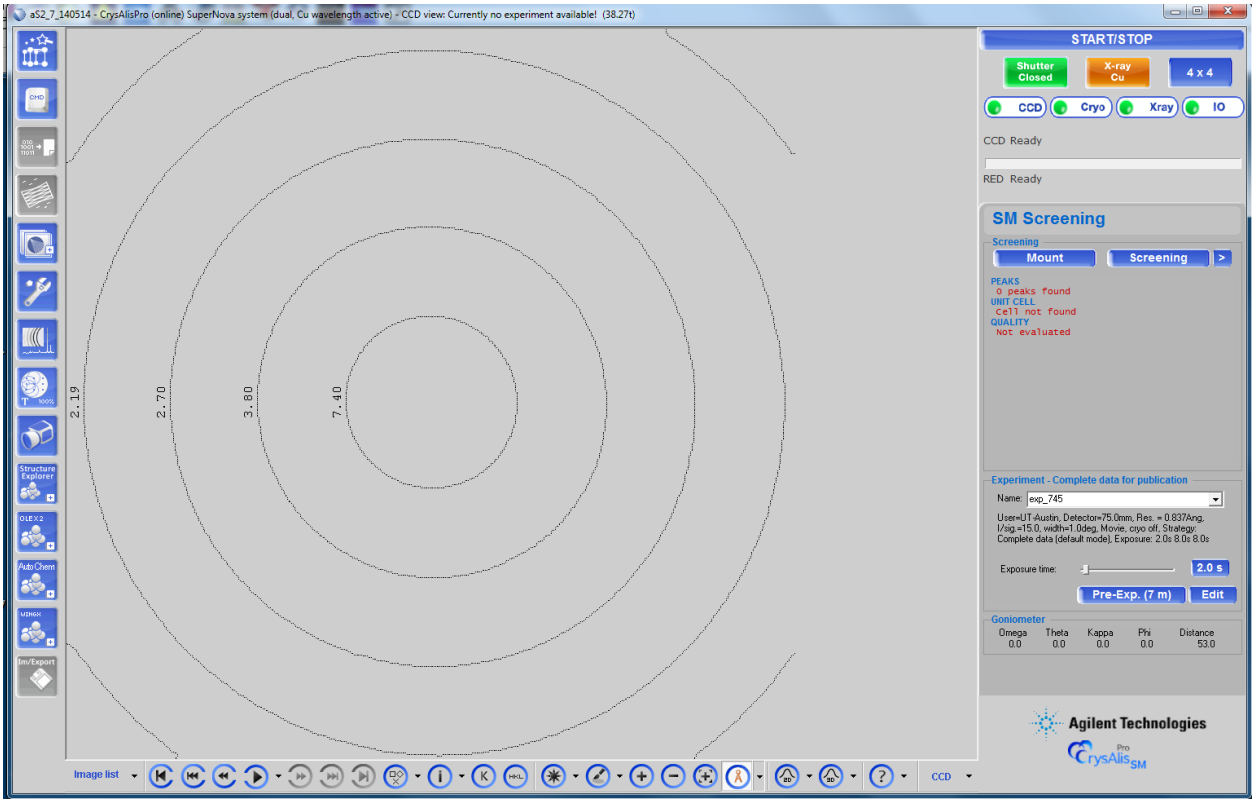
Using the Agilent for Single Crystal Work

Generally, the program to access the Agilent, CrysalisPro, will be open. If not, you can start the program using the shortcut on the desktop.

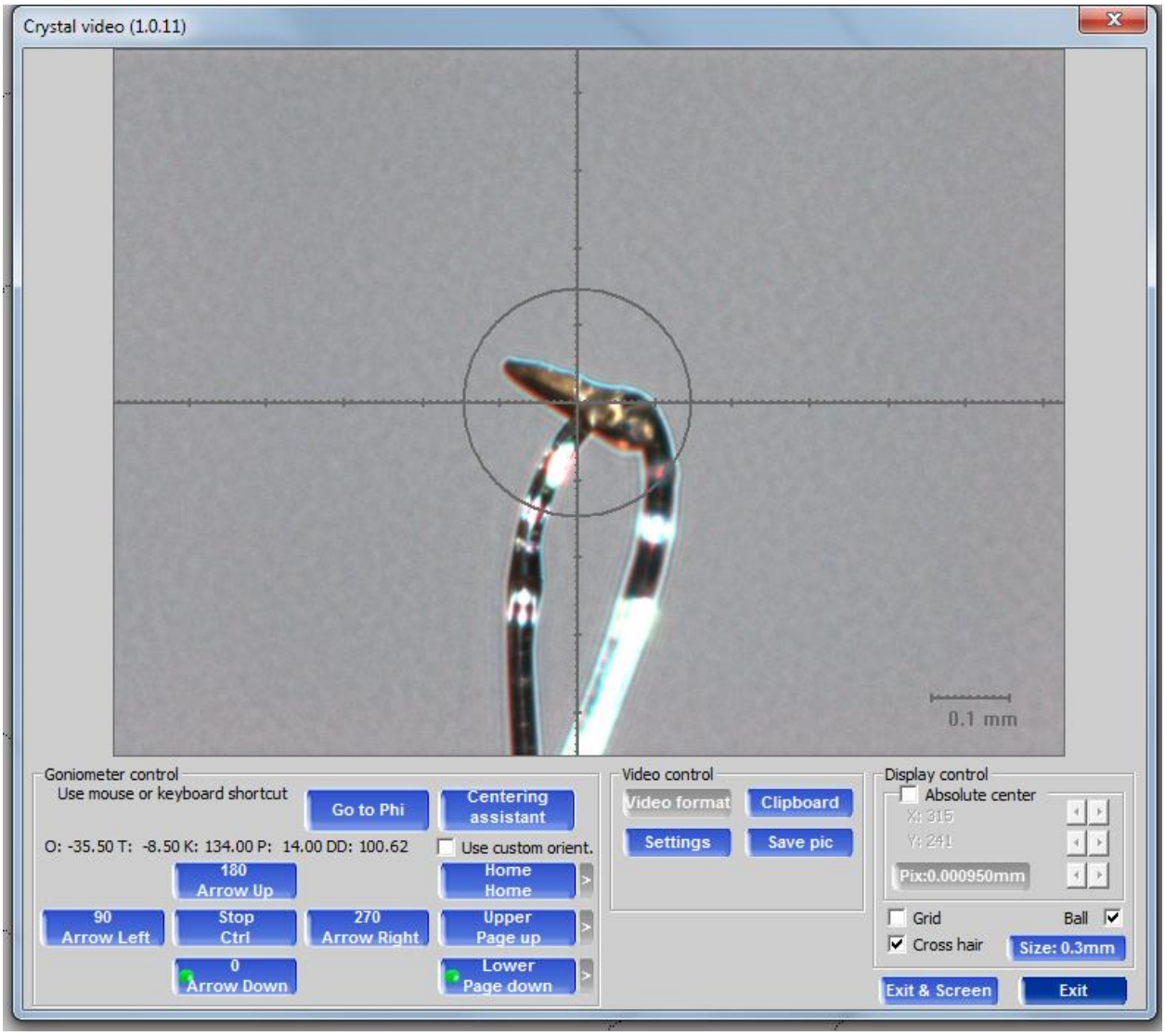


There is no password required for activation. The startup screen will appear as below.

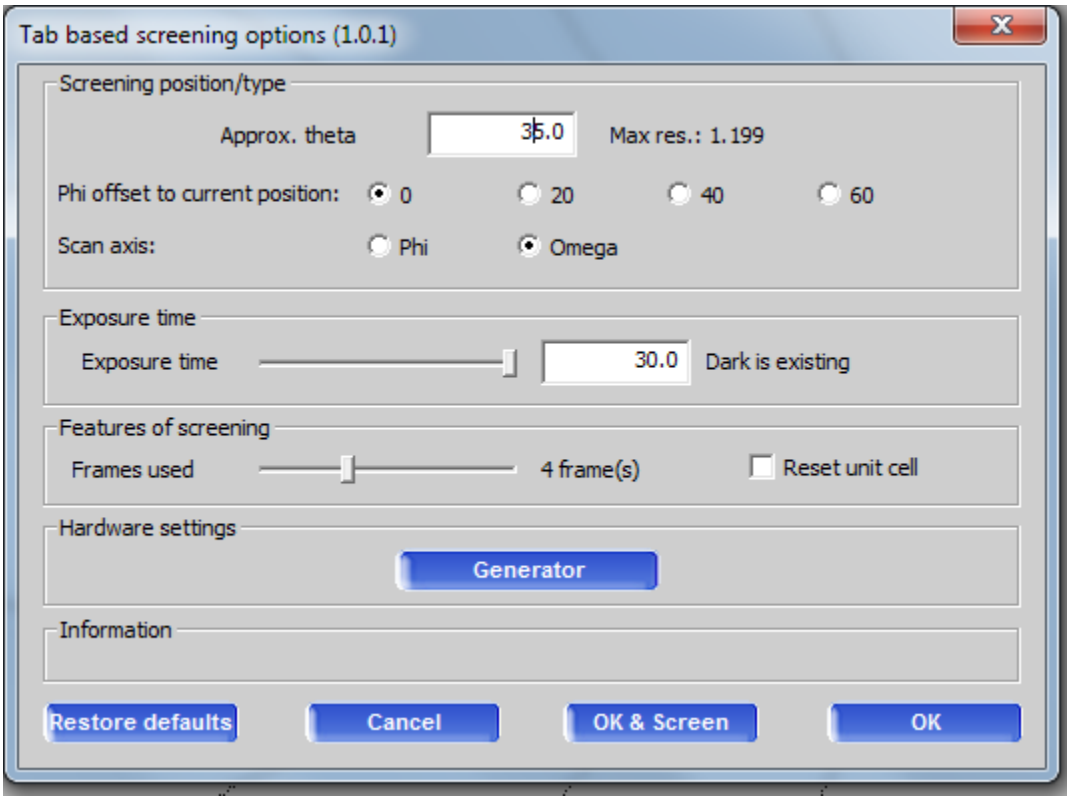


The Agilent has two X-ray sources available for use, Cu and Mo. The source selected will be listed in the orange box at the top right, which in the above figure is Cu. The Cu source will give the most intense beam and is generally preferred.

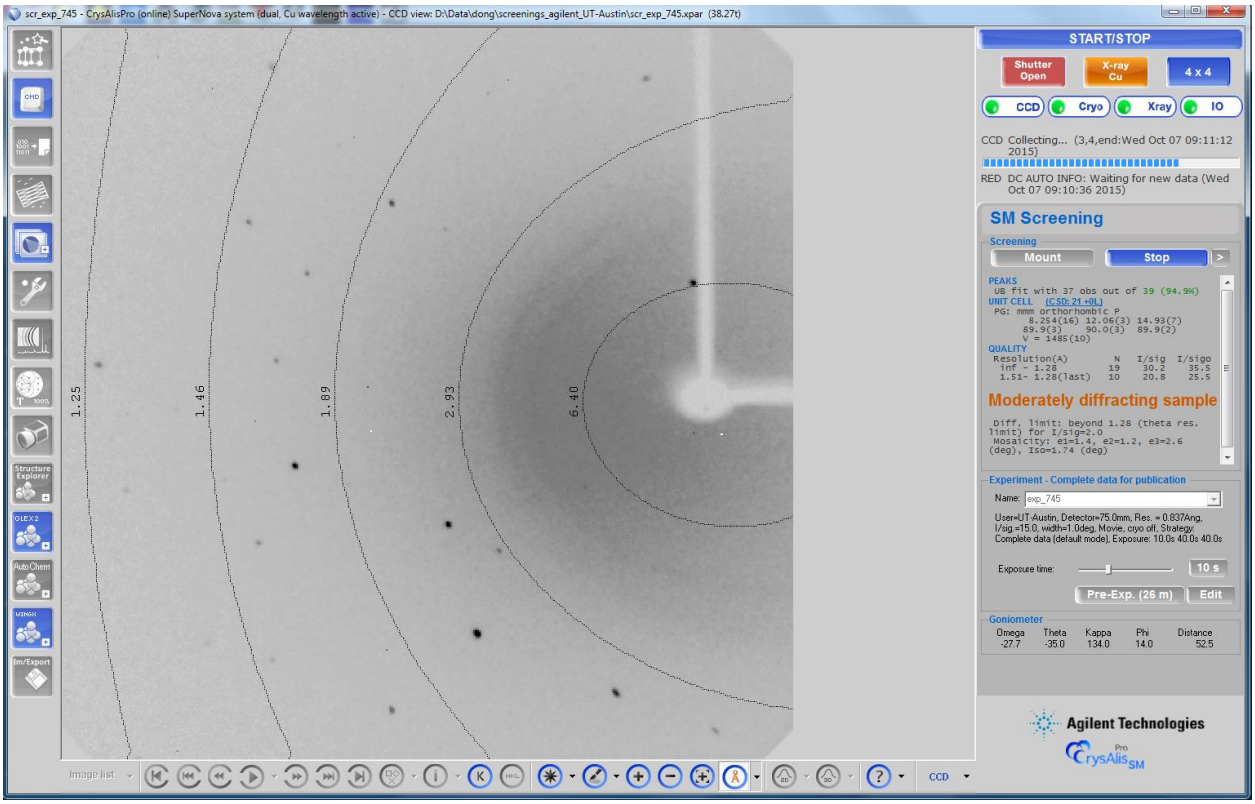
To begin, click on the Mount button. The instrument will drive to the crystal mounting start position. Unlock the door with the key and mount your crystal with the mounting loops provided.



The beam diameter for the Cu source is approximately 0.15 mm. You can check the alignment of your sample by clicking on the appropriate box shown in the lower left corner of the image. You can also use the up/down arrows on the keyboard. When you are satisfied that the crystal is centered, you are ready to begin the screening process. You must close and lock the door before collecting any data. Selecting Exit & Screen will begin the screening process using the starting values selected by the previous user. Selecting Exit will return you to the Start/Stop menu. Selecting the > symbol to the right of Screening will allow you to select the initial screening conditions as shown below.

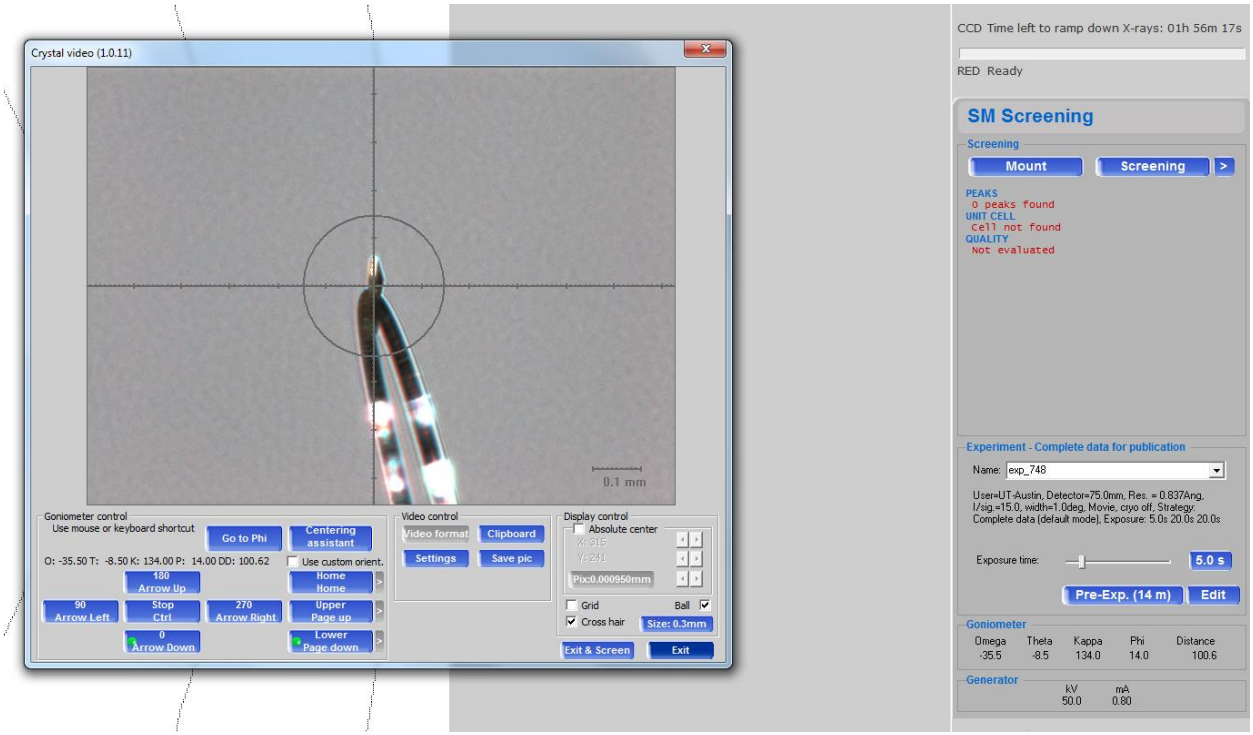


In the figure above, the screening images will have a detector offset of 35° for 4 frames at 30 seconds per frame. This is a reasonable starting point for an unknown sample. With some experience, you can adjust these settings as needed. Select OK & Screen to begin. OK takes you back to Start/Stop.

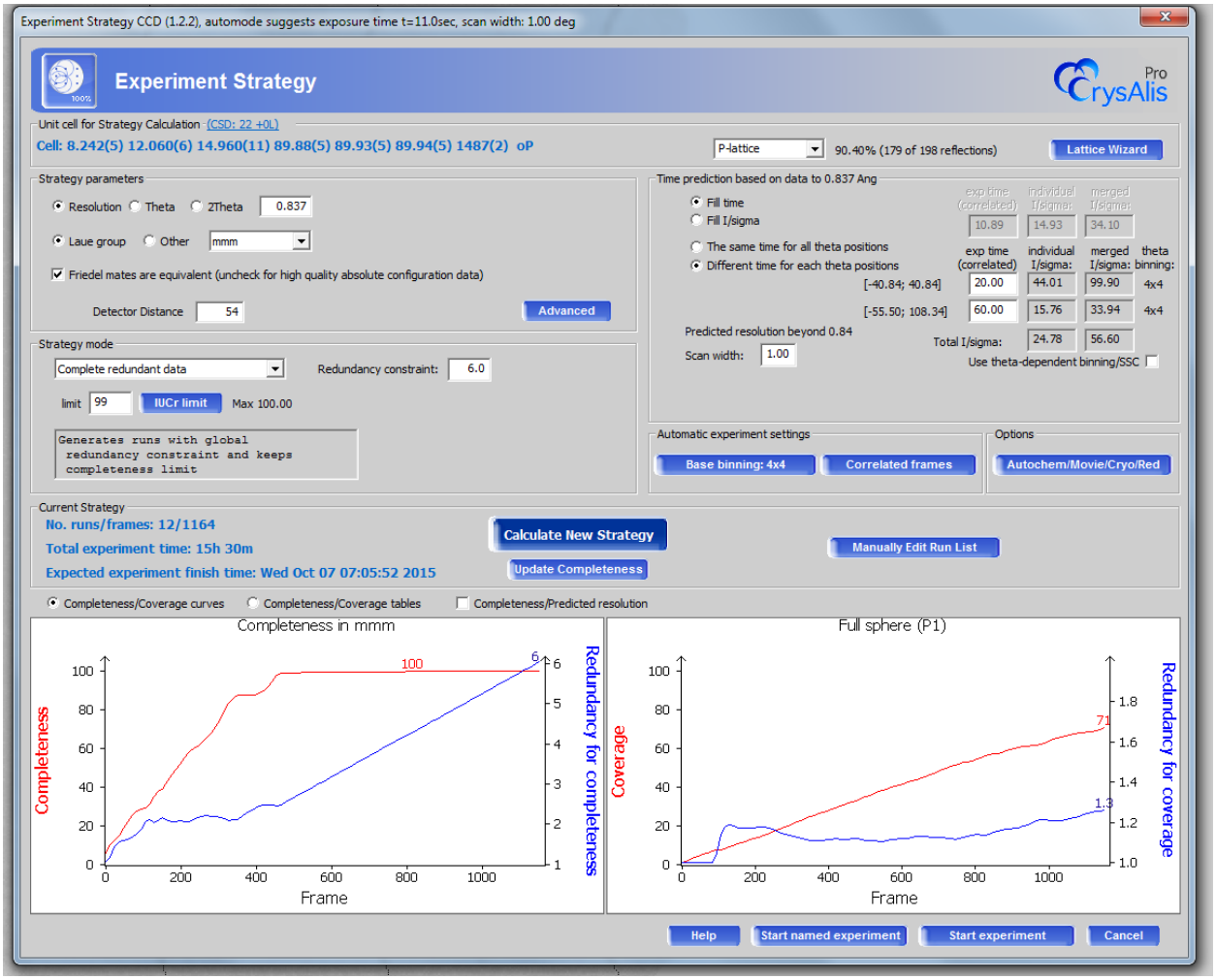


Depending on your sample, you should see something like the figure above. In the middle, far right portion of the image, the unit cell as determined using the screen images will be displayed. Under PEAKS, you will see the number of reflections found and the number that fit the displayed unit cell with a percentage of reflections that fit that cell. To the right of UNIT CELL, the number of entries in the Cambridge Crystallographic Database whose unit cell matches that determined for your sample will be displayed (CSD +xx). Double clicking CSD will display these entries. The entries list only those structures with a unit cell similar to the one determined for your sample. Given that there are over 700,000 entries in the database, there are likely to be a few matches to the cell. But it is possible that the crystal under study is not what you think it is and you can check the selected entries for possible matches.

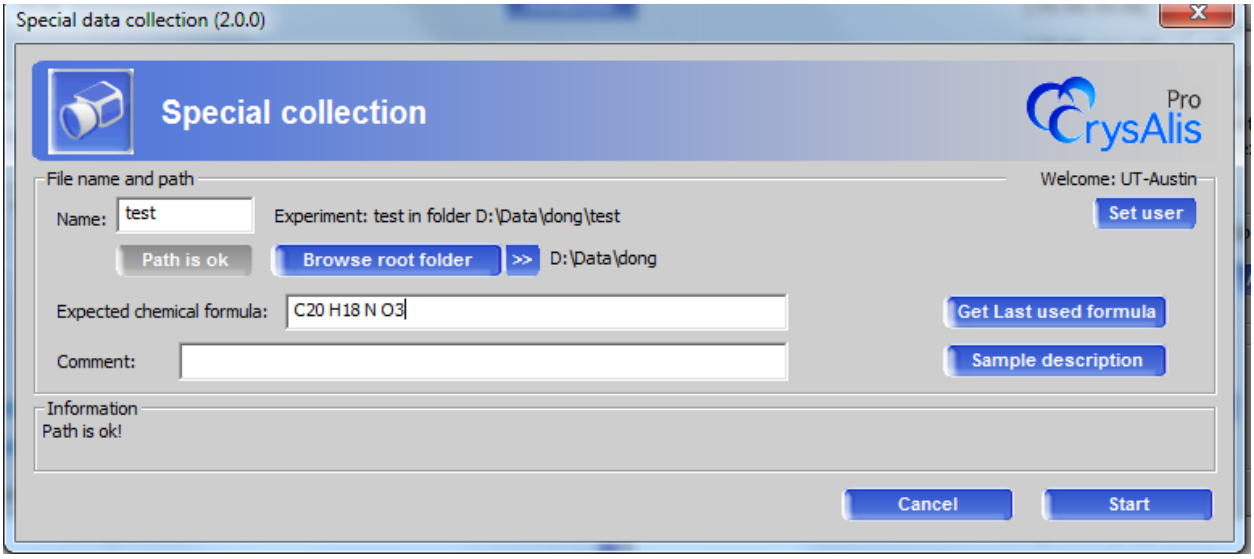
The initial screening is just that. It allows you to determine if the crystal is worth the effort of a full data collection. The next step before data collection is the pre-experiment. The pre-experiment uses the screening results to estimate a reasonable data collection time.



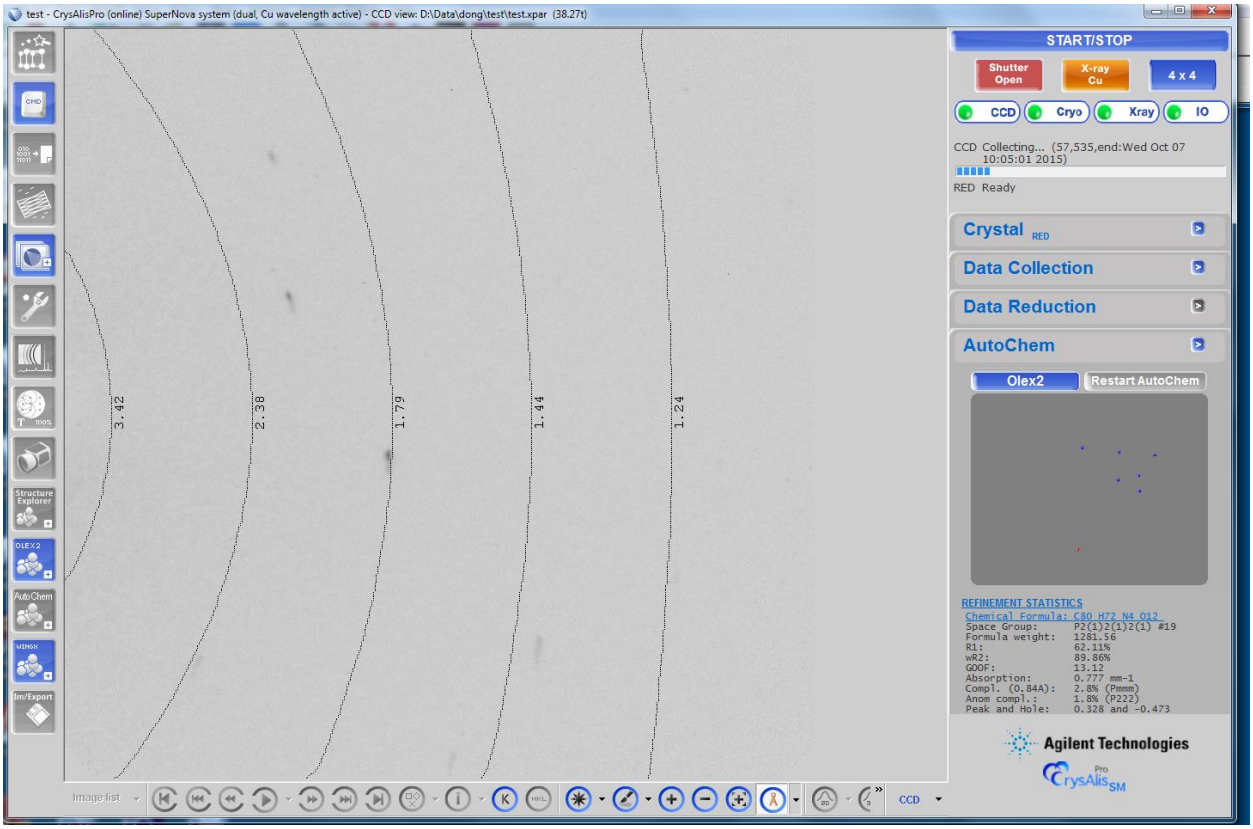
The Pre-Experiment performs a more rigorous search of diffraction space and should provide a more reliable unit cell and crystal system for data collection. After the Pre-Experiment has collected a suitable number of frames, CrysalisPro will display the new unit cell as determined from the newly collected frames. This new cell should match the original cell. At this point, you can continue with the Pre-Experiment or begin data collection. A menu item on the right will appear that will enable you to go to the data collection screen. The data collection menu is shown below.



The screen above shows the parameters for data collection. The resolution is set to 0.837Å, which is the minimum resolution recommended by the IUCr. The Laue group is set by the results of the Pre-Experiment. Check that the detector distance is 54 mm. At this crystal to detector distance, there will be two entries for data collection time, one for each detector offset. The detector can be moved back for large unit cells as needed. The data collection limit is 100% by default. You should change this to 99%. The extra frames needed to increase the data completeness from 99 to 100% aren't worth the time needed. A better use of instrument time is to increase the data redundancy. The default value is 1, which will give a complete data set. The default value for scan range is 1° in ω. Whenever the completeness limits or the data redundancy are changed, you need to click on the Calculate New Strategy box for the new values to take effect. The expected finish time will be updated as this time. The finish time is updated whenever you change the data collection time. Click on the Start named experiment box to get to this menu.



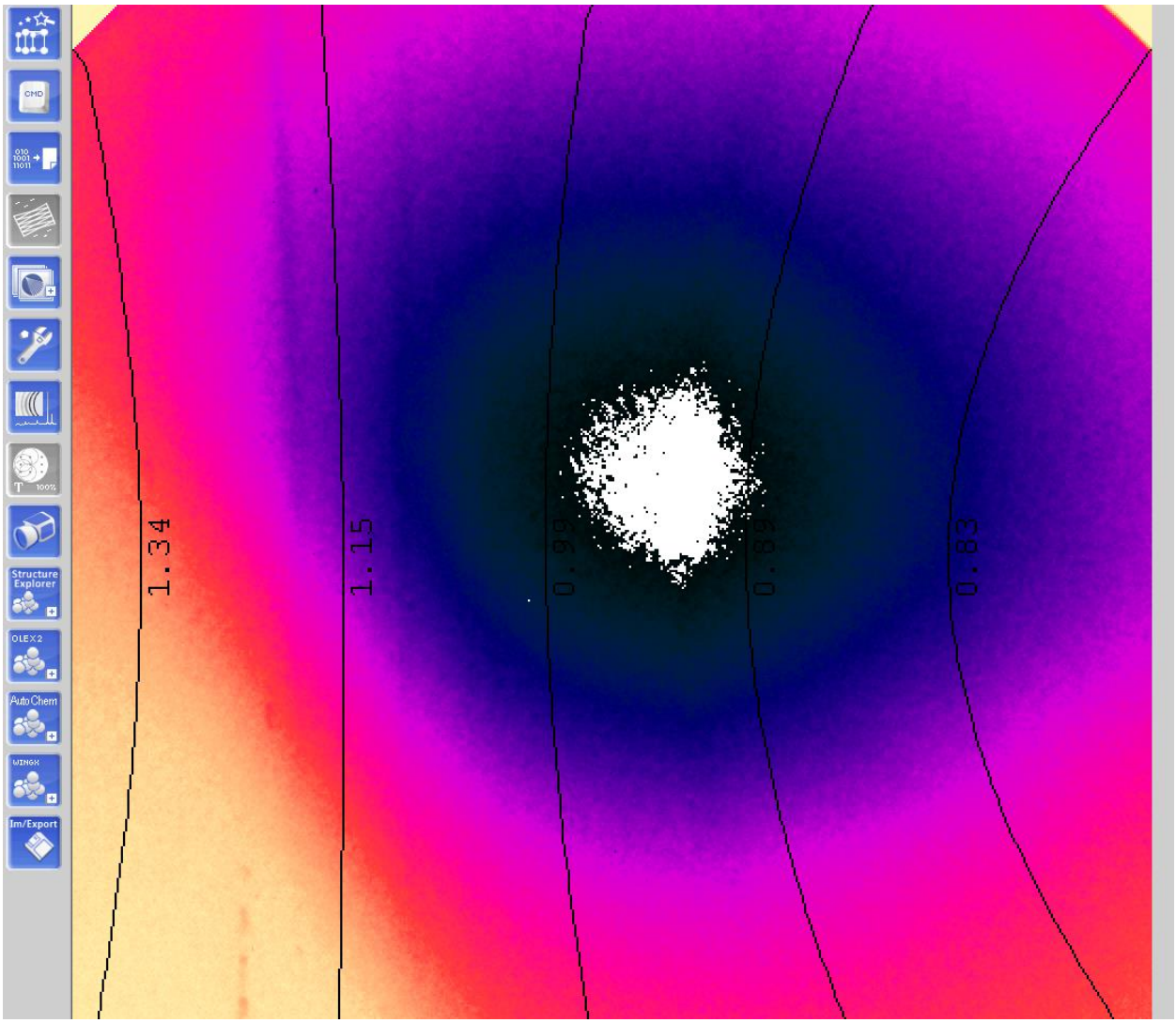
In the Special collection menu, you add the name of the experiment, where you want all the files to go and the expected chemical formula. CrysalisPro will process the data as it is being collected and attempt to solve and refine the structure using Olex2.



The data collection menu will appear as above. After several frames have been collected and the data processed, Olex2 is called upon to solve and refine the structure. If successful, the structure will appear in the window on the right hand side. Occasionally, Olex2 will be unsuccessful. Often this is due to a space group ambiguity or an incorrect cell. Often, these problems can be worked out after the data set is complete.

In general, all the files necessary for complete structure analysis will be in the Olex2 folder. Alternatively, you can begin the process using WinGX by selecting the WinGX icon on the left side of the menu.

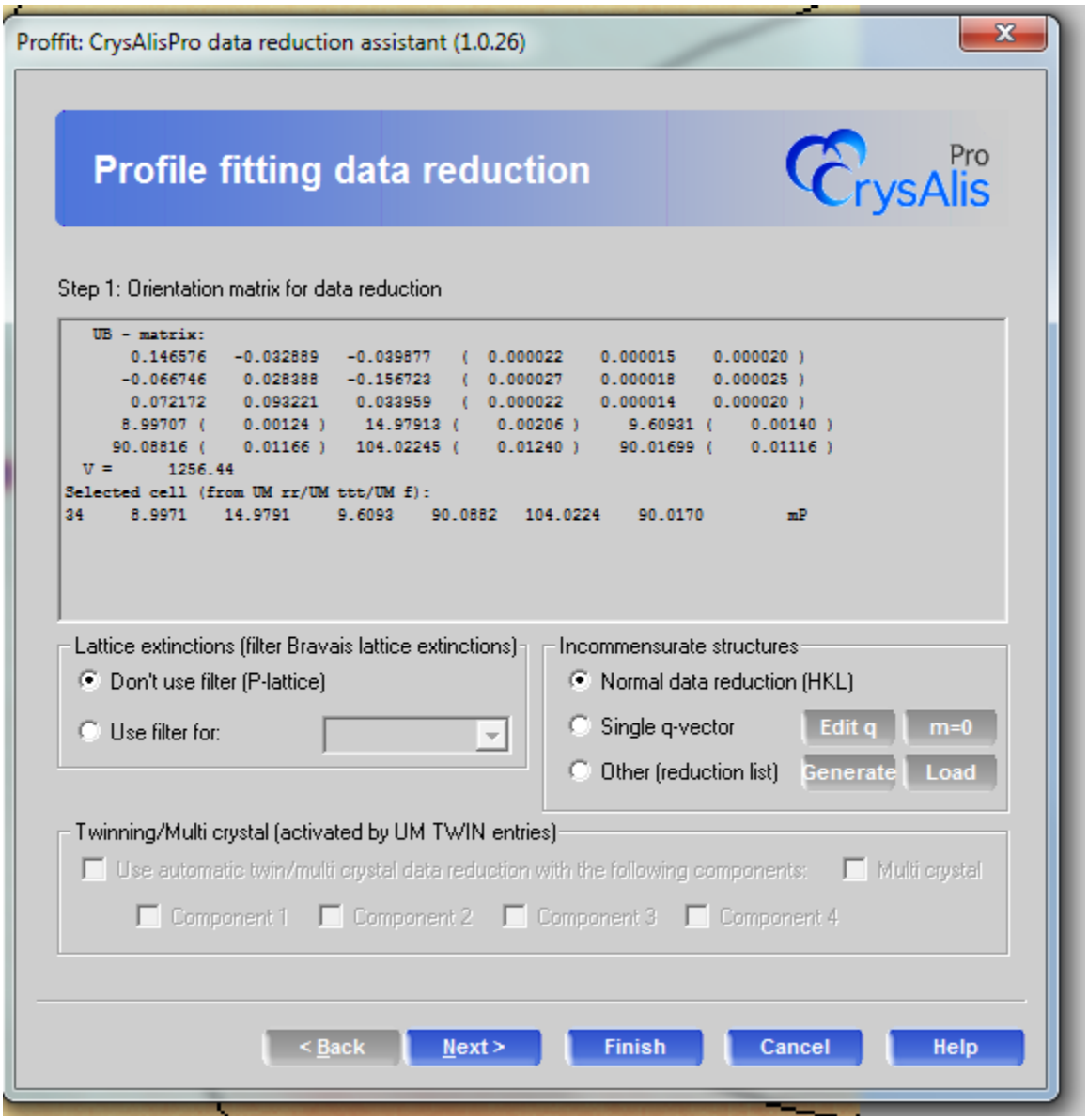
CrysalisPro will integrate the frames during data collection. Generally, a refined structure and a complete data set will await you when data collection is complete. However, it may happen that what appears to be a good data set gives very poor integration results. This can occur because of a few bad frames. One such frame is shown below. It occurs when the steel mounting pin holding the nylon loop in place is clipped by the X-ray beam. X-rays scattered off the pin generate a very intense 'background' that causes the integration to go off the rails. In such cases, you have to remove those bad frames from the data integration process and re-integrate.



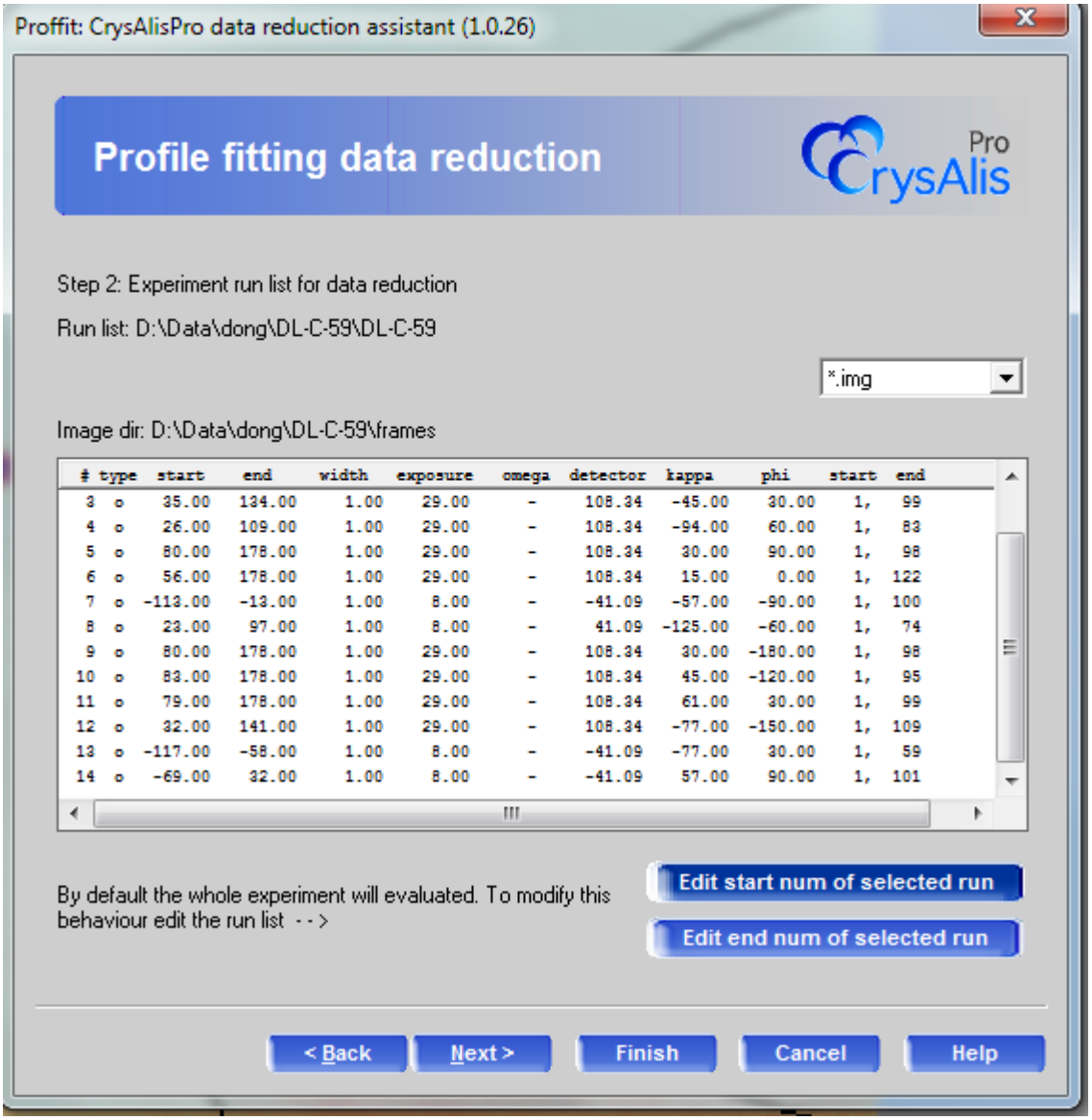
To determine which frames may be causing problems, you can replay the data collection process using the tools at the bottom of the screen.

The icon, ▶, will play back the images of the collected frames and display the frameset number. Not where the bad frames appear. You can use the other icons to go back and forth through the frames one frame at a time or 10 at a time. Hover the mouse over Data Reduction to bring up the data reduction with options menu.

Select data reduction with options to bring up the data reduction assistant.



You can toggle through the process by selecting Next> until you reach the menu that will allow you to sort through the frames to use.



If you wish to remove an entire frameset from the integration step, select “Edit start num of selected run” and enter 0 for the first frame. If the bogus frames occur at the beginning of the frameset, enter a number where the ‘good’ frames begin. If the bogus frames occur at the end of the frameset, enter a frame number of one of the last ‘good’ frames in the “Edit end num of selected run”.

At the end of the data reduction procedure, note the Rint for the integration. A good data set will have low values, less than 0.05 is pretty good.