

Tutorial on processing of single-crystal diffraction data collected at GSECARS 13-BM-C using APEX3 Crystallography Software Suite

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Reference data can be found at:

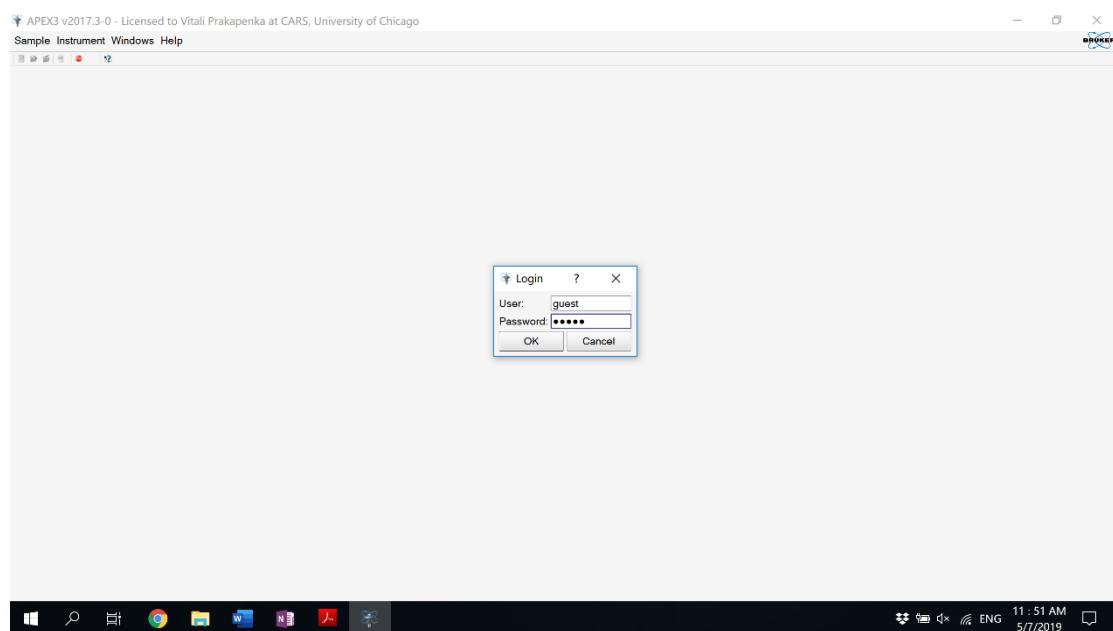
https://www.dropbox.com/s/l1nlakirwdafxnc/OEN_RefData.zip?dl=0

Ref data includes 2 runs. Run S013: 2theta/del = 0. Run S014: 2theta/del = -20.

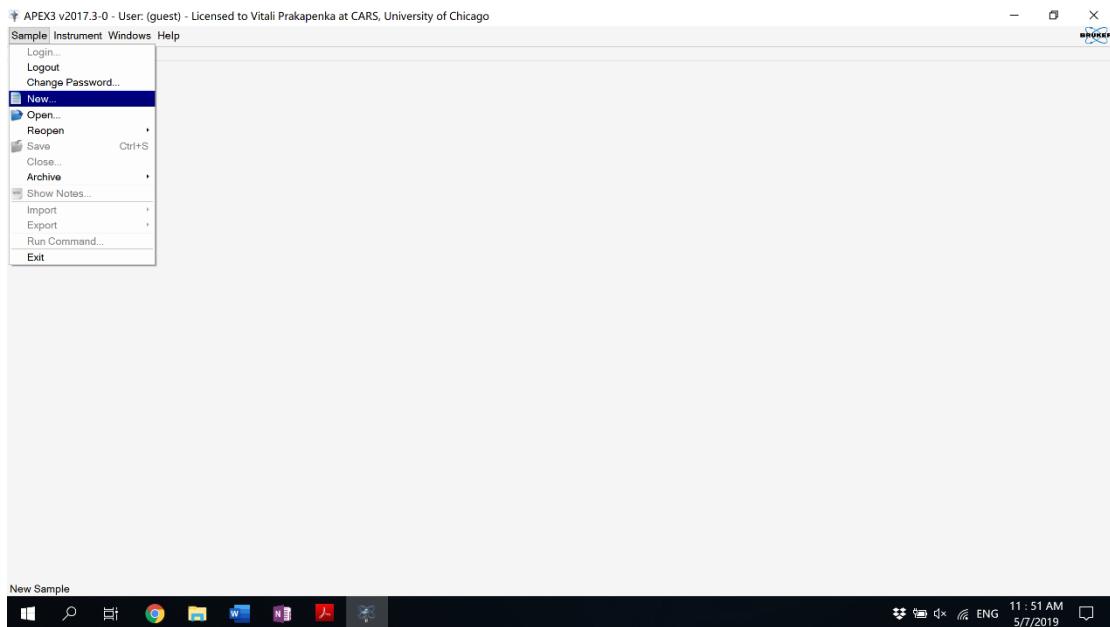
1. Start APEX3

Double clicking on the **APEX3** icon to start the program.

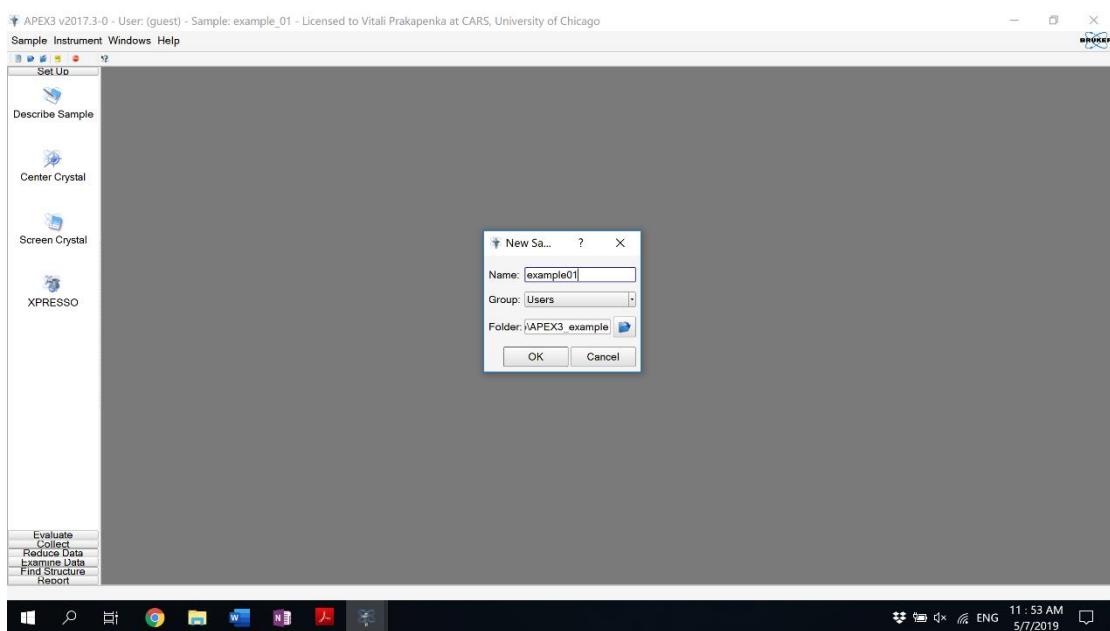
Input your user name and password to login.



Start a new project by clicking 'sample' --> 'New'



Choose a folder where your single-crystal diffraction data (cbf files) are located, and input a name of the new project.



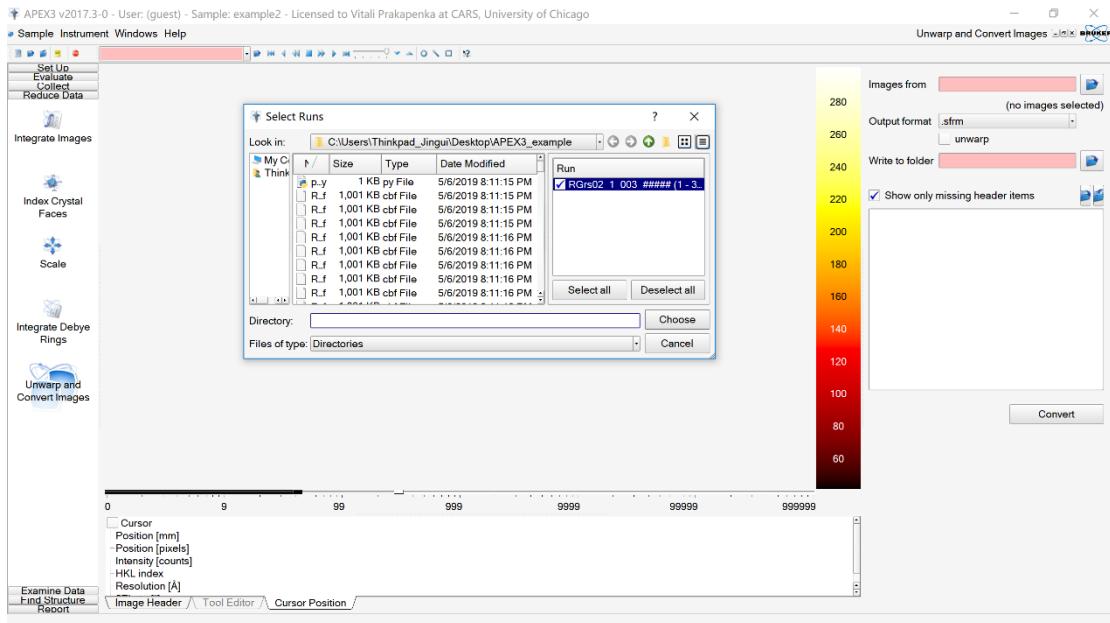
2. Converting images

At 13-BM-C the collected images are in '.cbf' format, we need to convert them into '.sfrm' format before the further processing.

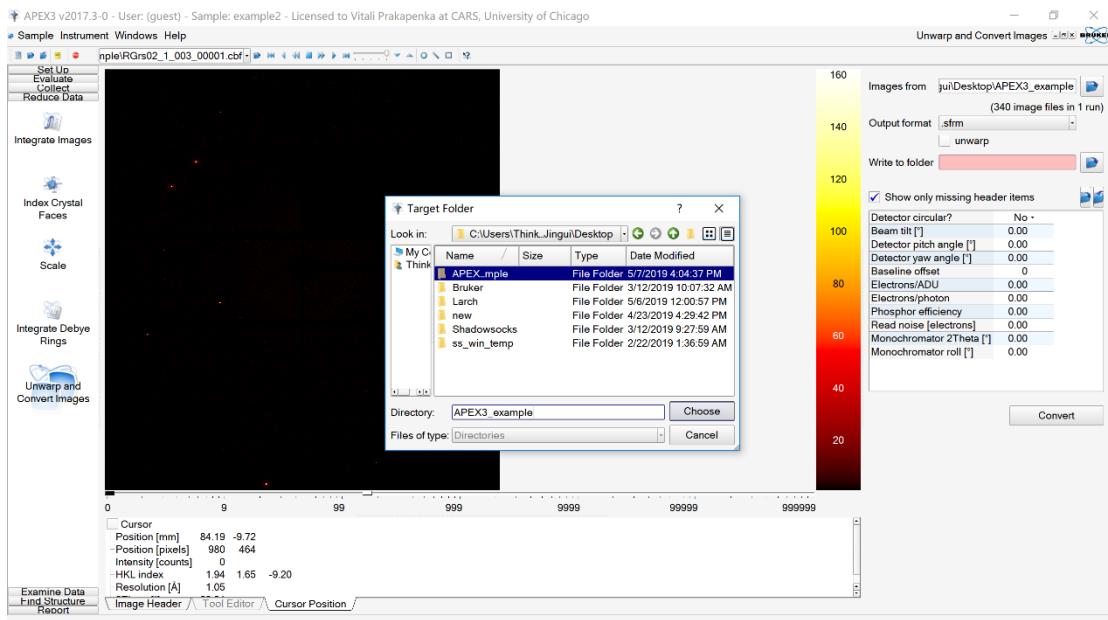
In the Task Bar's '**Reduce Data**' section, click the **Unwarp and Convert Images**

icon.

Select the images from your project folder.

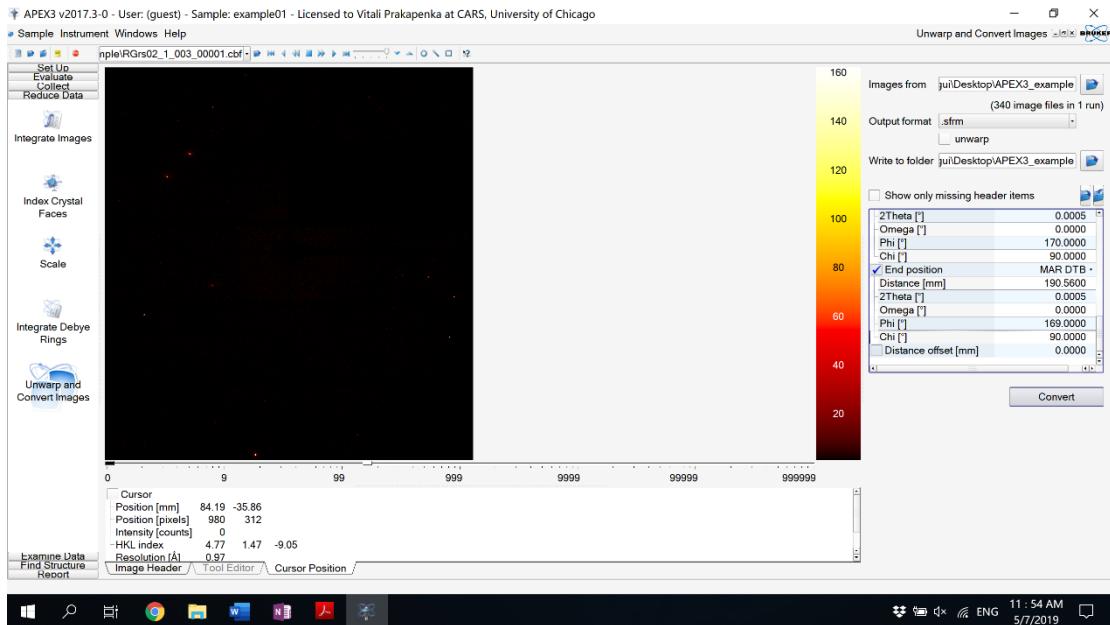


Chose a folder for the converted images.

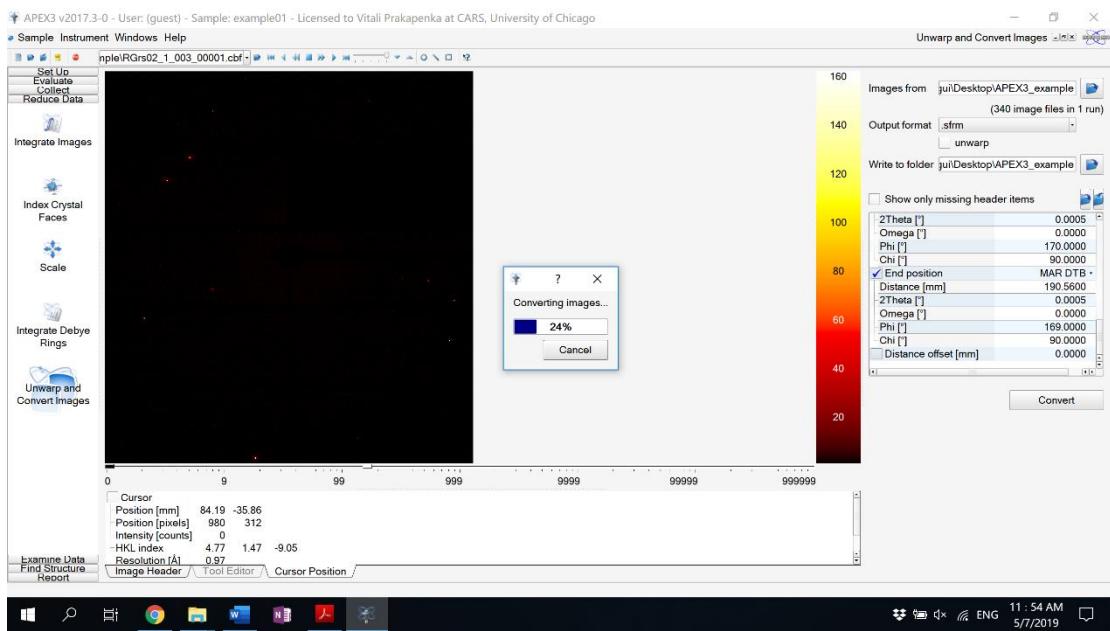


Uncheck the ‘Show only missing header items’ box. We need to modify some items for the images collected at 13-BM-C.

1. Set the value of ‘Detector roll angle’ to be 0;
2. Set the type of both the ‘Start position’ and ‘End position’ to be ‘MAR DTB’;
3. Set the ‘Chi(°)’ values from both the ‘Start position’ and ‘End position’ to be 90.
4. E/ADU = 1, E/Photon = 1, Phosphor efficiency = 0.5
5. If nu !=0, detector pitch = -nu.



Start converting images by clicking the ‘Convert’ button.

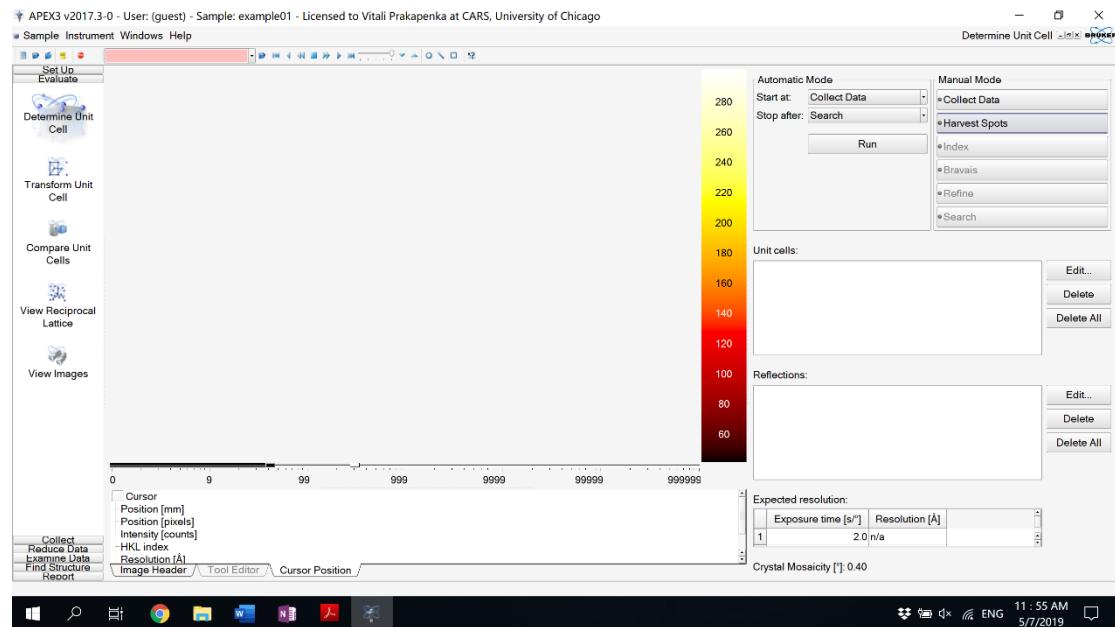


3. Harvest Spots

Go to the Task Bar’s ‘Evaluate’ section, click the ‘Determine Unit Cell’ icon



From the ‘Manual Mode’, click ‘Harvest spots’.

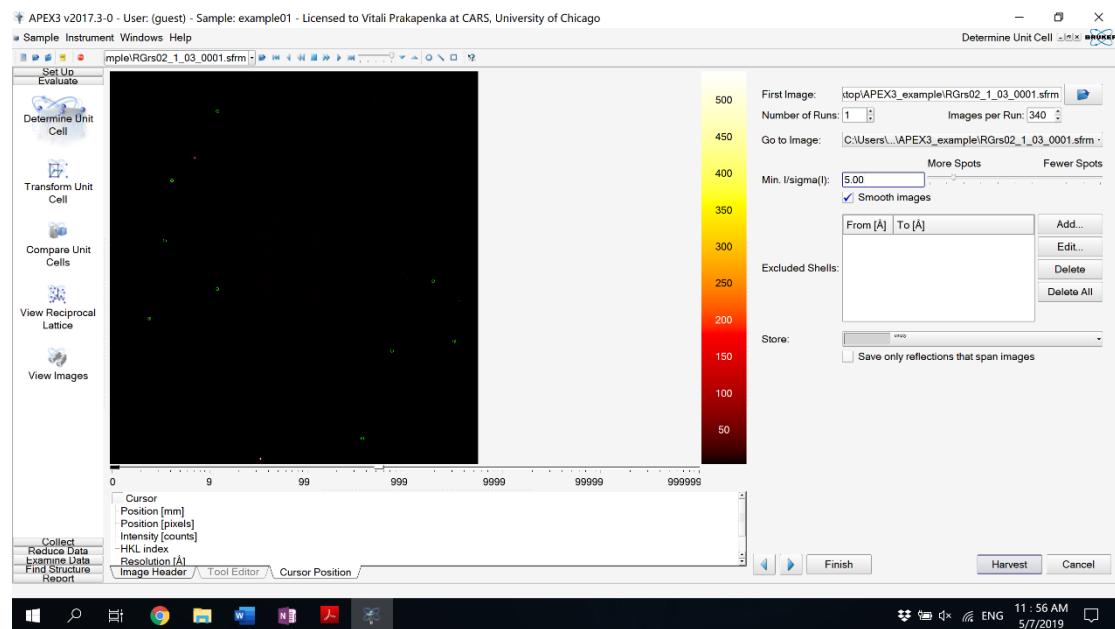


Go to ‘First image’, select the first image of the converted ‘.sfrm’ images.

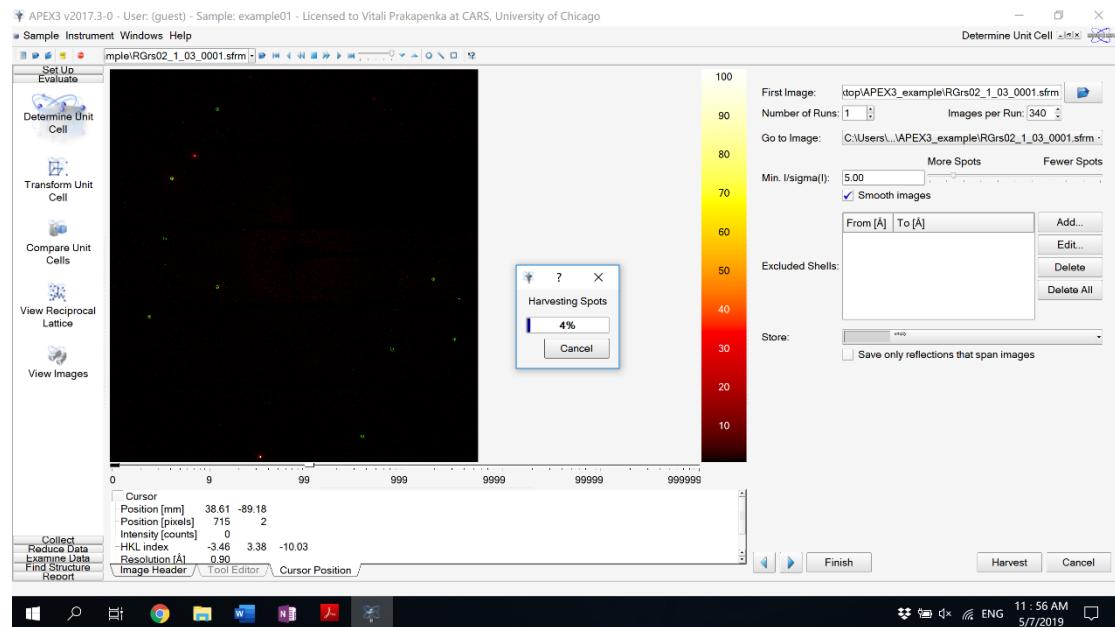
Number of Runs: in this case there is only one run to be examined.

Images per Run: in this case there are 340 images in each run.

Min. I/ $\sigma(I)$: set this value to harvest a reflection based on its intensity and standard deviation.



Start harvesting spots by clicking the ‘Harvest’ button.



4. Viewing harvested reflections in a reciprocal lattice

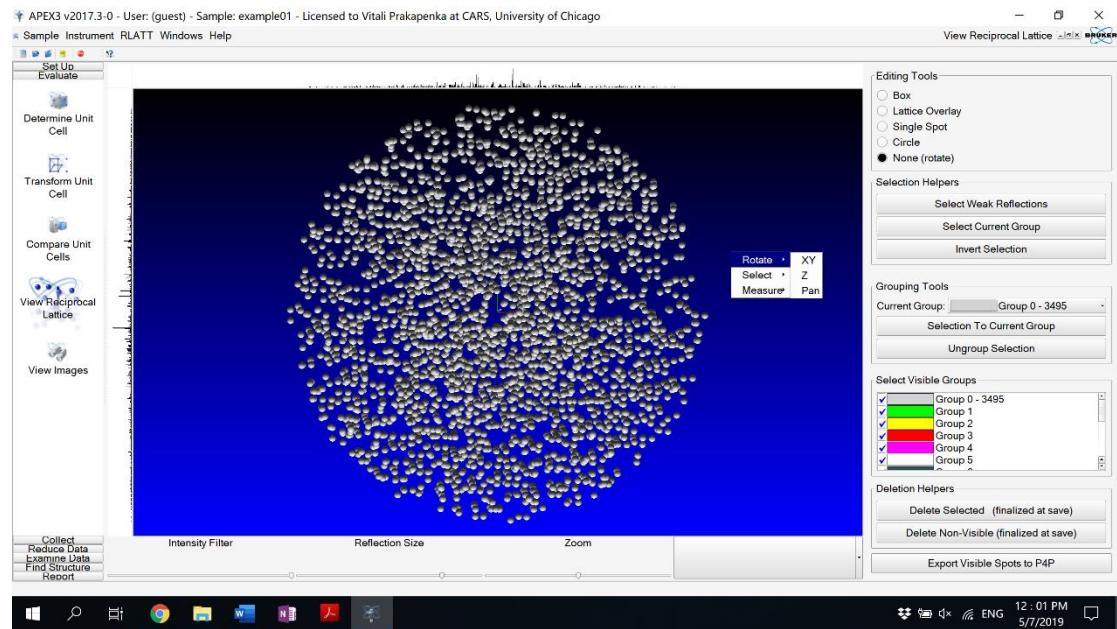
Go to the Task Bar's 'Evaluate' section, click the 'View Reciprocal Lattice' icon



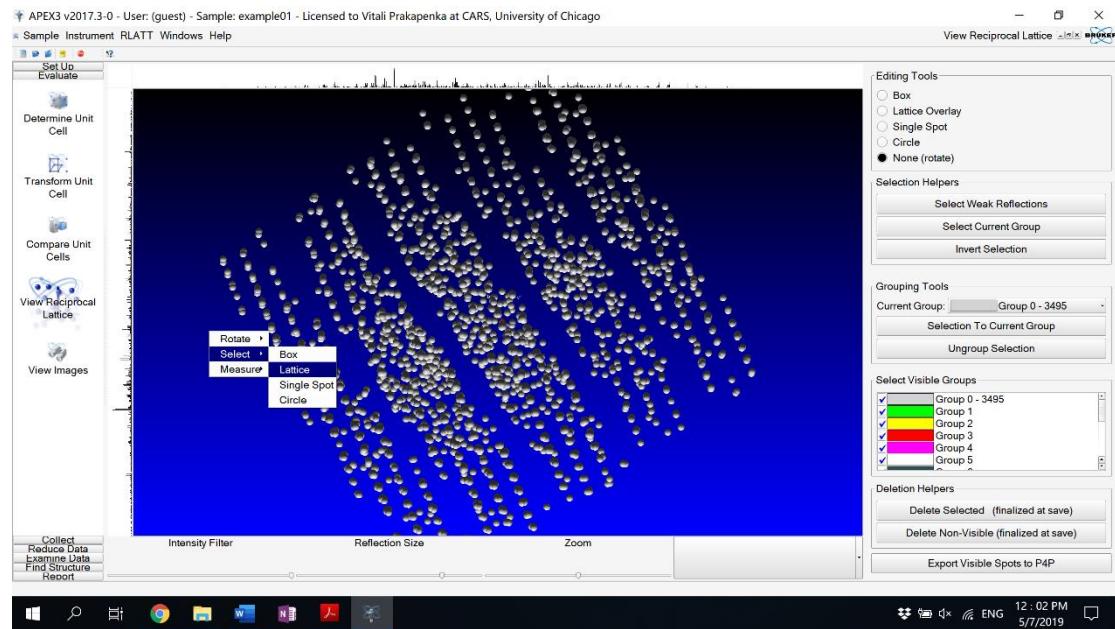
You would see an unoriented view of reflections.

Right-click and select the 'rotate' option, then drag the mouse to rotate the lattice display.

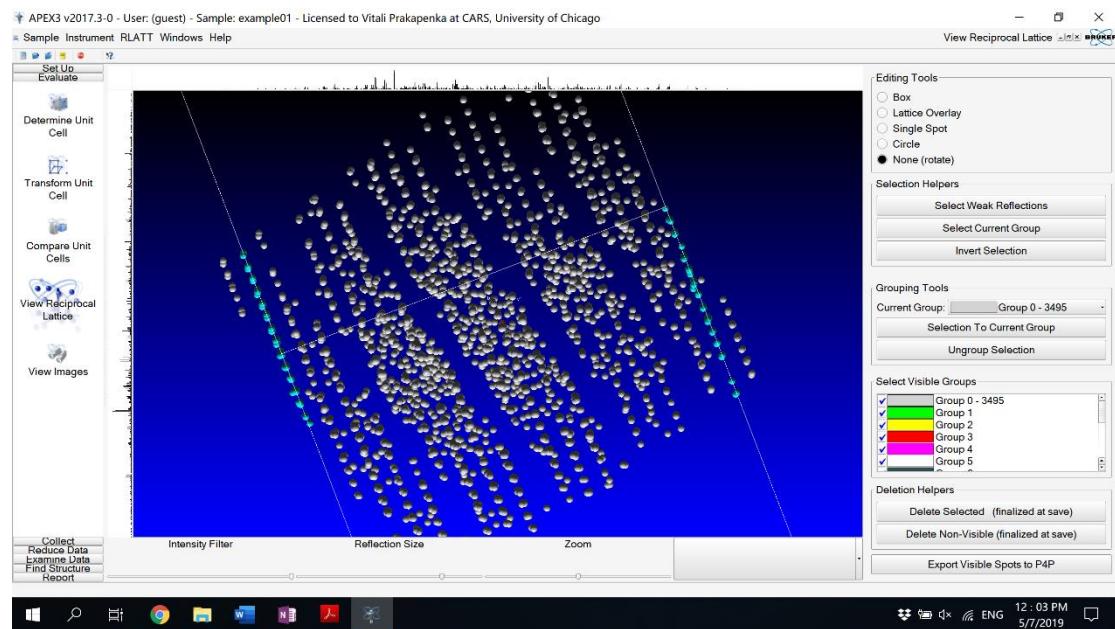
There are three sliders at the bottom of the view: '**Intensity Filter**', '**Reflection Size**', and '**Zoom**', you can move them to detect the weak reflections, change the reflection size in the view, and zoom in and out.



Use the reflection selecting tools to delete the non-fitting reflections.
First, rotate the lattice display to easily see rows and non-fitting reflections.
Right-click and choose the ‘Select’ -> ‘Lattice’ option.



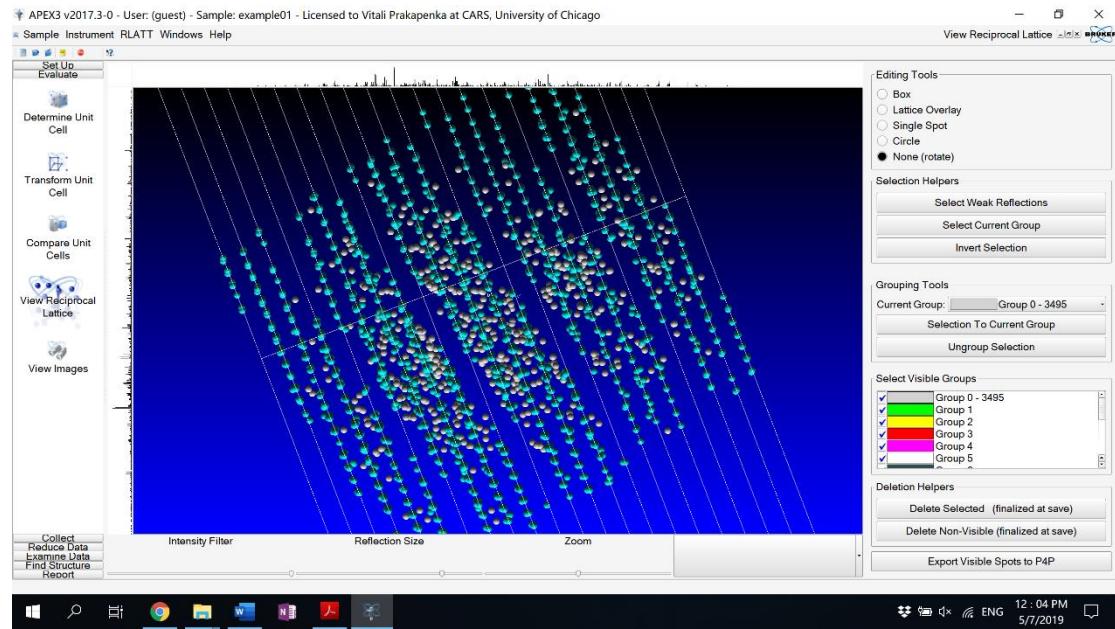
Move the mouse pointer to one reflection in a row, left-click and hold the button and move the mouse pointer to one reflection in another row. Once you release the mouse button, two rows are selected and marked, and indicated by two lines.



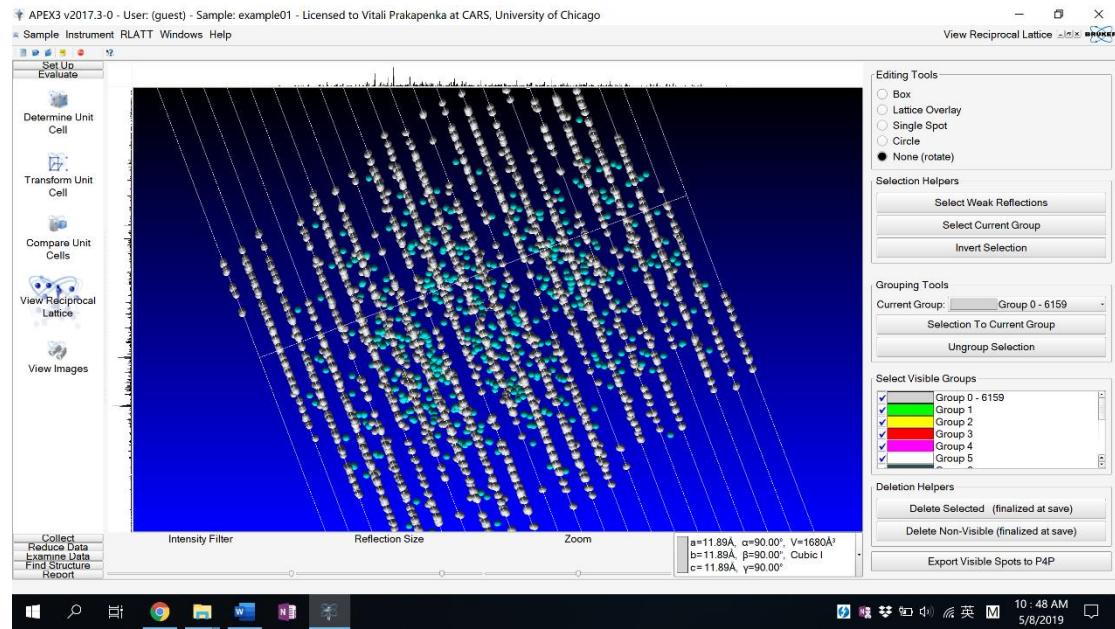
Press the '+' key to add one line between the two rows that have been marked, and press the '-' key to remove one line.

Press the '**Page UP**' key to add lines in the outside range of the previously selected

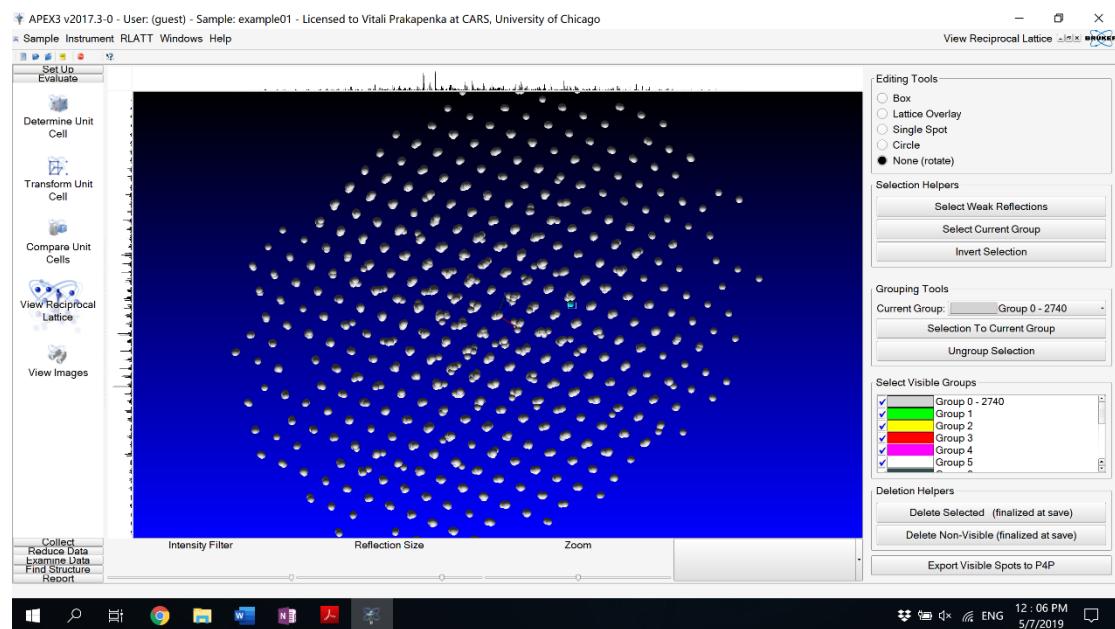
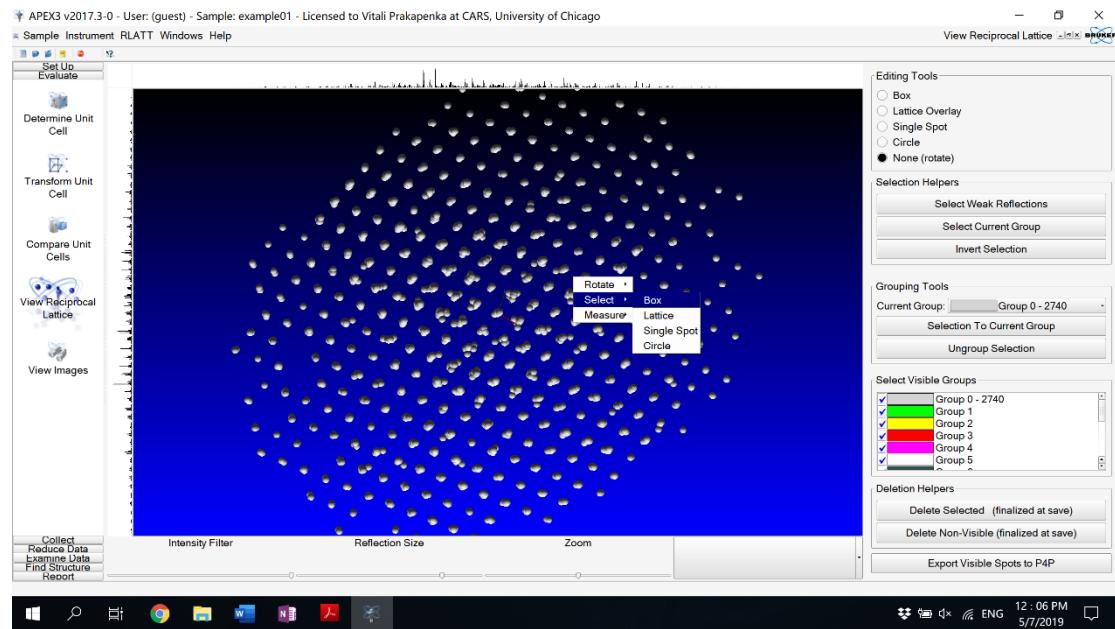
lines, and press the ‘**Page Down**’ key to remove lines outside.



Click the ‘**Invert Selection**’ under the ‘**Selection Helpers**’, then the non-fitting reflections are selected. You can delete these non-fitting reflections by clicking ‘**Delete Selected (finalized at save)**’ under the ‘**Delete Helpers**’. Then you can rotate the lattice display to other orientation and repeat the ‘**Selection**’ and ‘**Delete**’ operation to remove the non-fitting reflections.



You can also select reflections by drawing a box or circle, and select a single reflection by clicking on it.



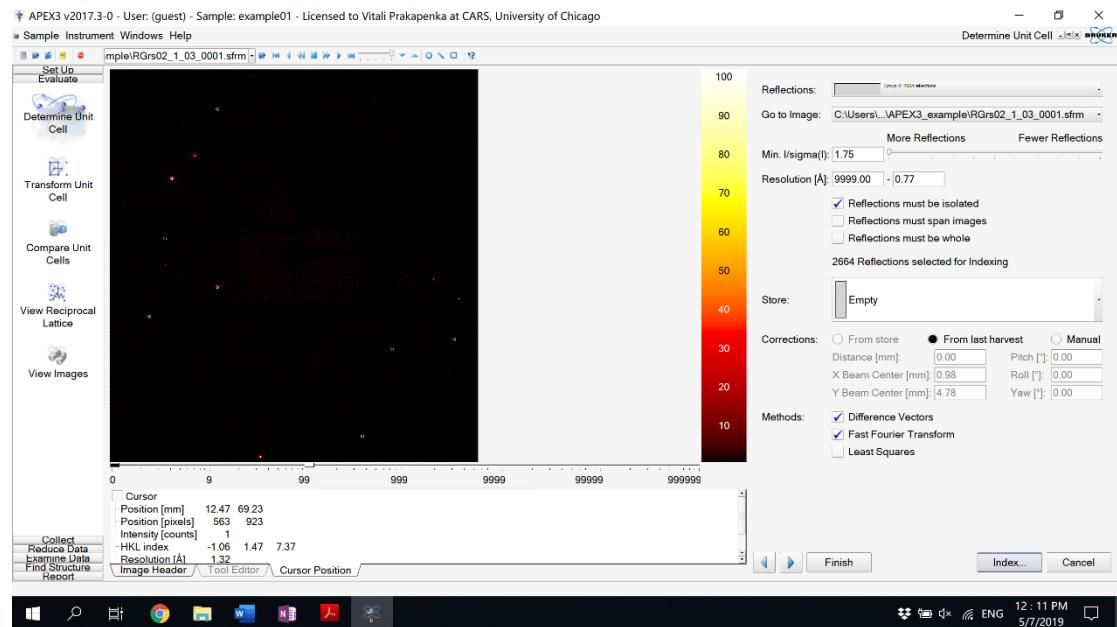
5. Index

Go to the Task Bar's '**Evaluate**' section, click the '**Determine Unit Cell**' icon

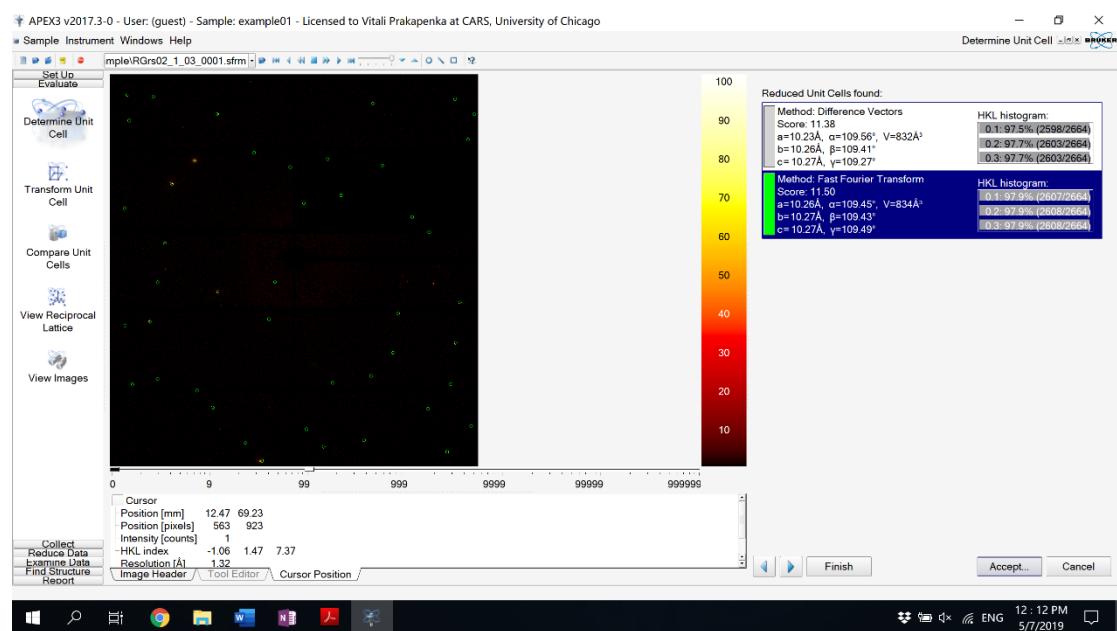


From the '**Manual Mode**', click '**Index**'.

Adjust the value of '**Min. I/sigma(I)**', click the '**index**' button.

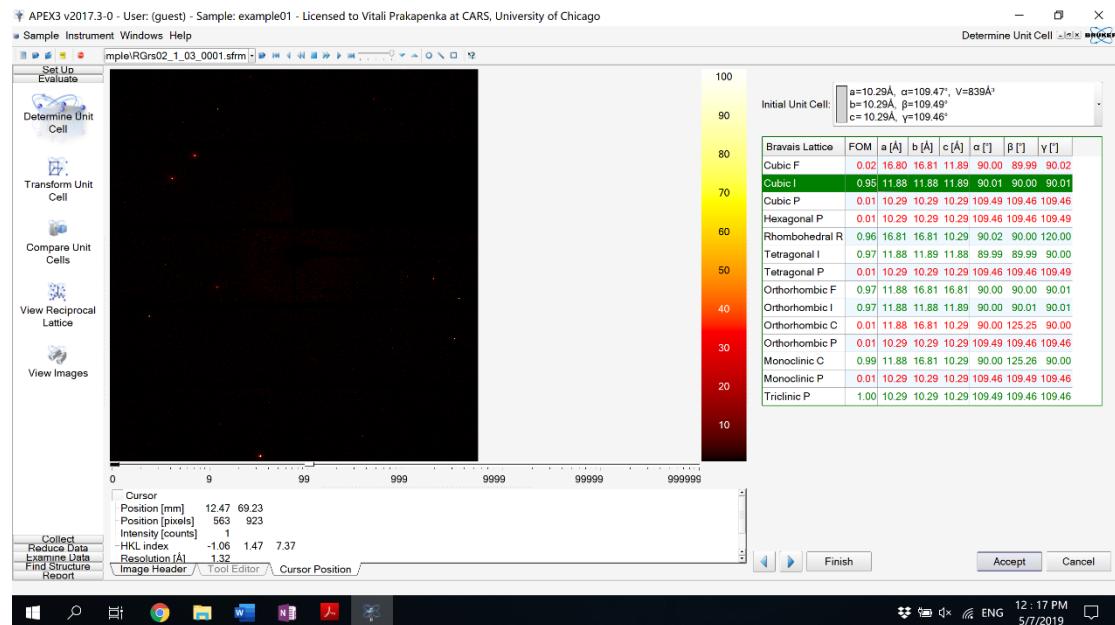


Select a Unit Cell from the ‘Reduced Unit Cells found’ field, and click the ‘Accept’ button.



Automatically go to the ‘Bravais’ section, which lists 14 Bravais lattice types for the selected unit cell parameters. Each type includes ‘FOM(the figure of merit)’, and the six unconstrained unit cell parameters ‘ a , b , c , α , β , and γ ’. Bravais lattices that are in agreement with the unit cell are marked in green. The most likely lattice type is selected automatically.

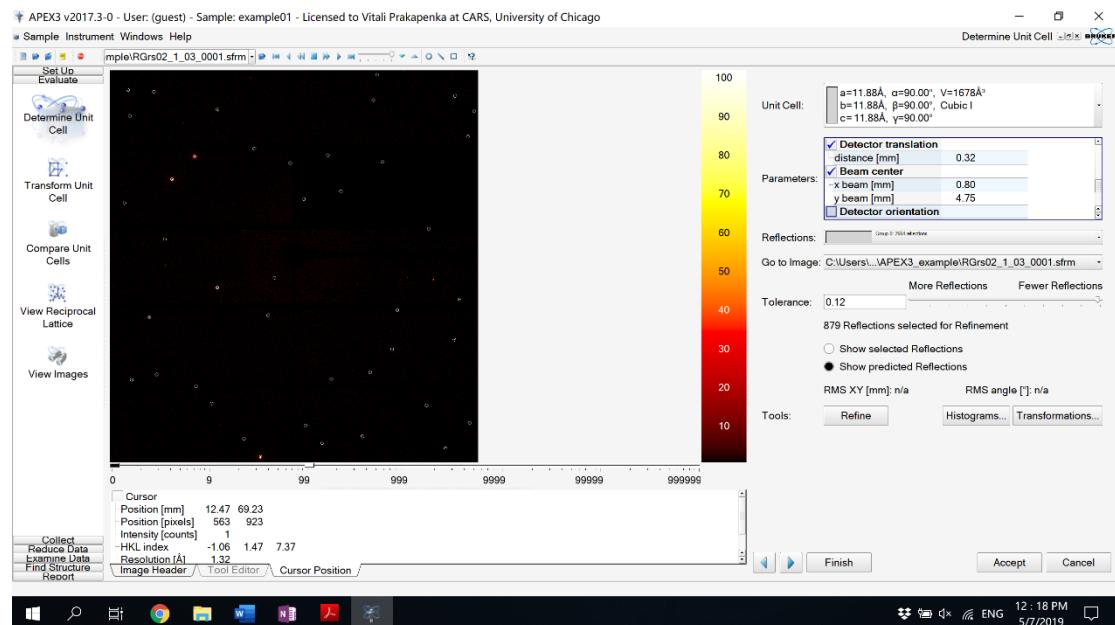
If the selected lattice type is acceptable, click the ‘Accept’ button automatically go to the ‘Refine’ section.



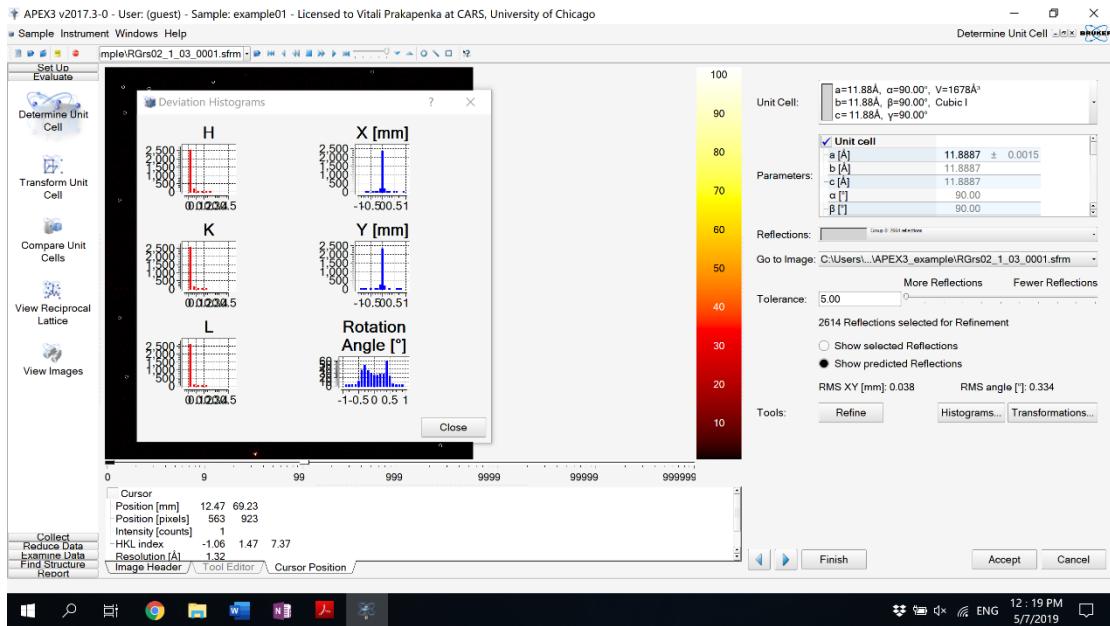
6. Refine

Under the ‘Parameters’ section, check the ‘Detector orientation’, uncheck the “detector translation” (keep as 0). Set a useful value for the ‘Tolerance’ (usually drag the slide all the way to the left).

Click the ‘Refine’ button, and the ‘Refine’ process may take a few seconds.



After the ‘Refine’, click the ‘Histograms’ button, a window will be opened and show the reflections’ distribution of deviations from the calculated orientation matrix. These include reflection indices **H**, **K**, and **L**, **X** and **Y**, and **Rotation Angle**. **X** and **Y** indicate the reflection positions in the **XY** image plane. The rotation angle indicates the reflection position along the trajectory of the reflection while it passes through the Ewald sphere.

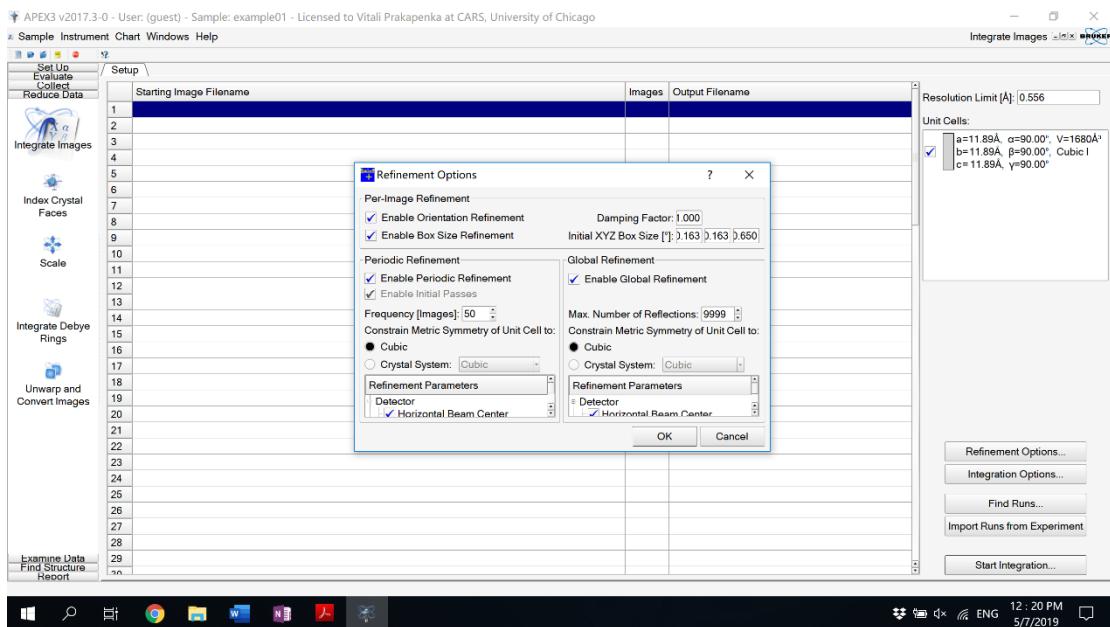


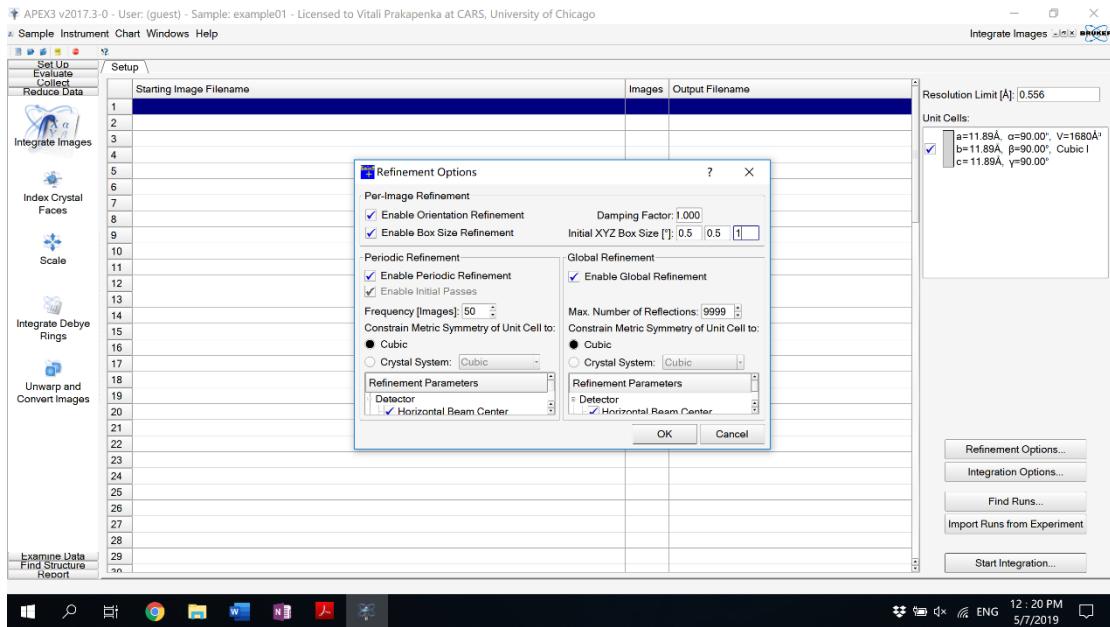
7. Data Integration and Scaling

Go back to the Task Bar’s ‘Reduce Data’, click the ‘Integrate Images’ icon

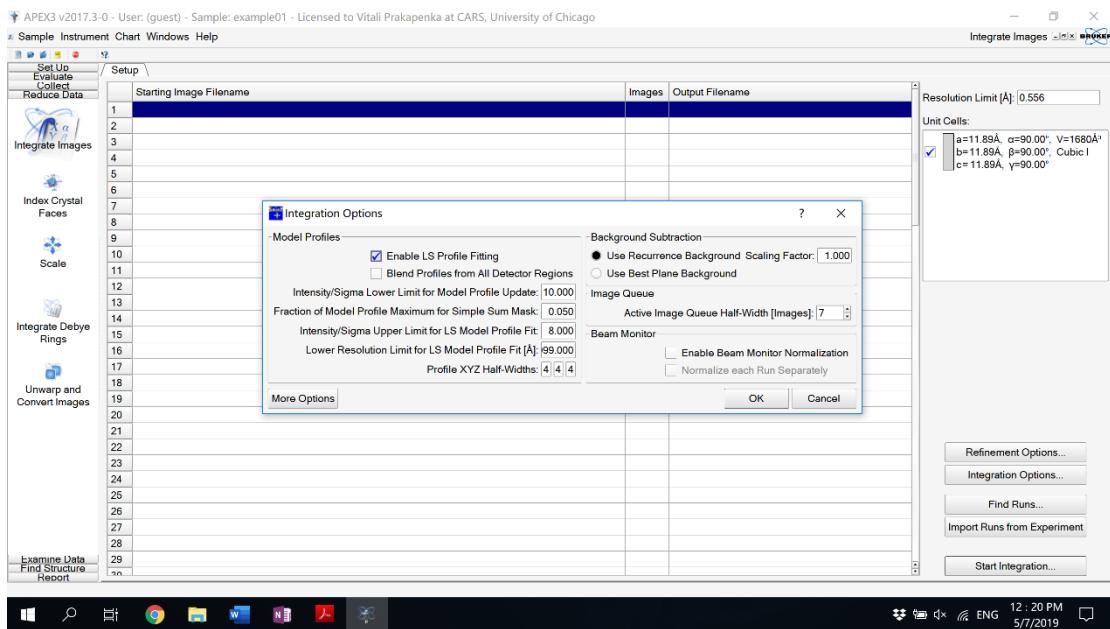


Click the ‘Refinement options’ button, change the values of ‘Initial XYZ Box Size[°]’ to ‘0.5, 0.5 and 1.0’.



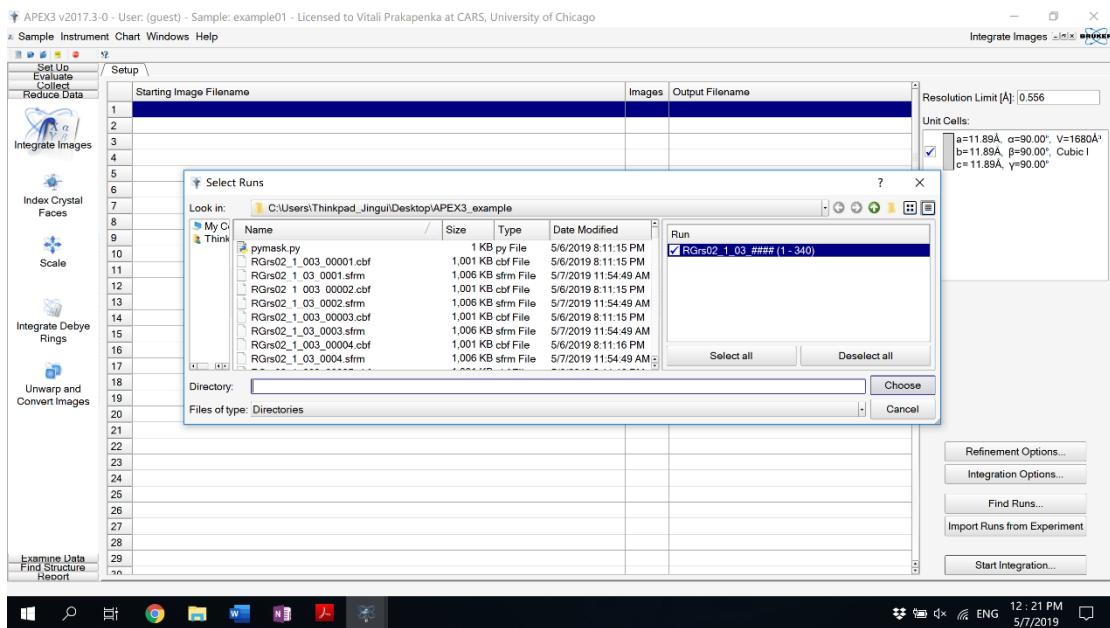


Click the ‘Integration Options’ button, uncheck the ‘Enable LS Profile Fitting’.

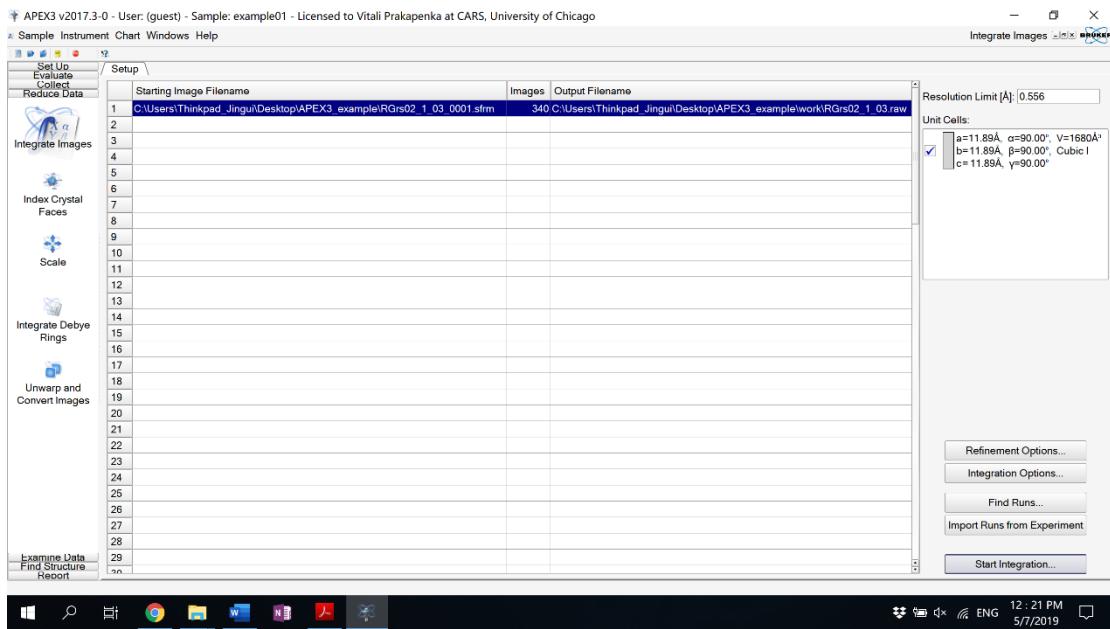


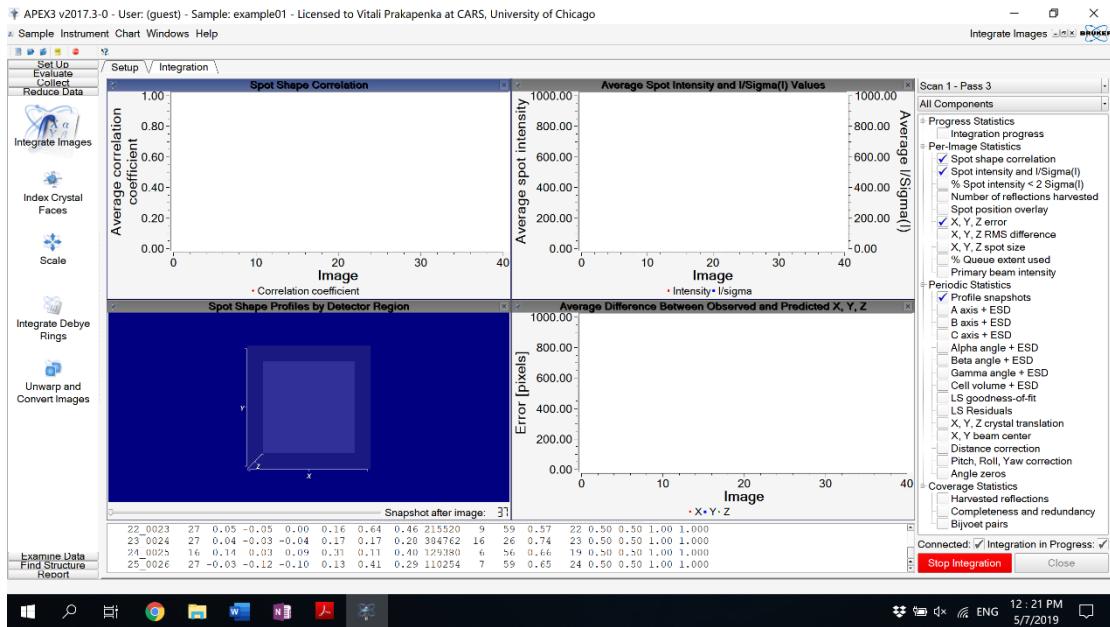
Click the ‘Find Runs’ button, browse to the runs correctly and check, click the

'Choose' button.



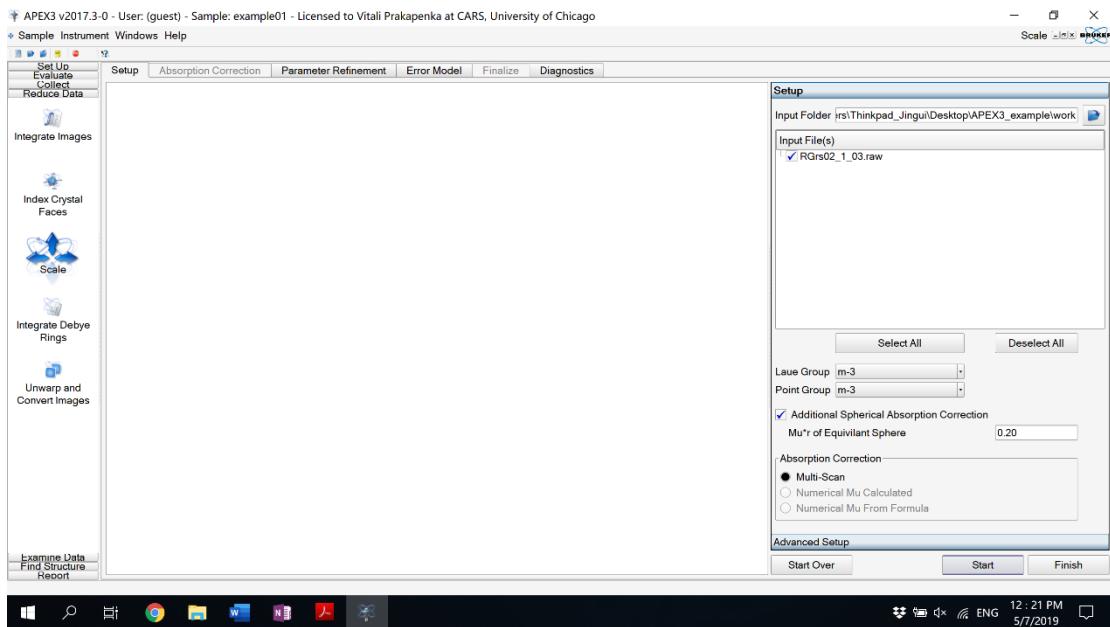
Start the integration by clicking the 'Start Integration' button.



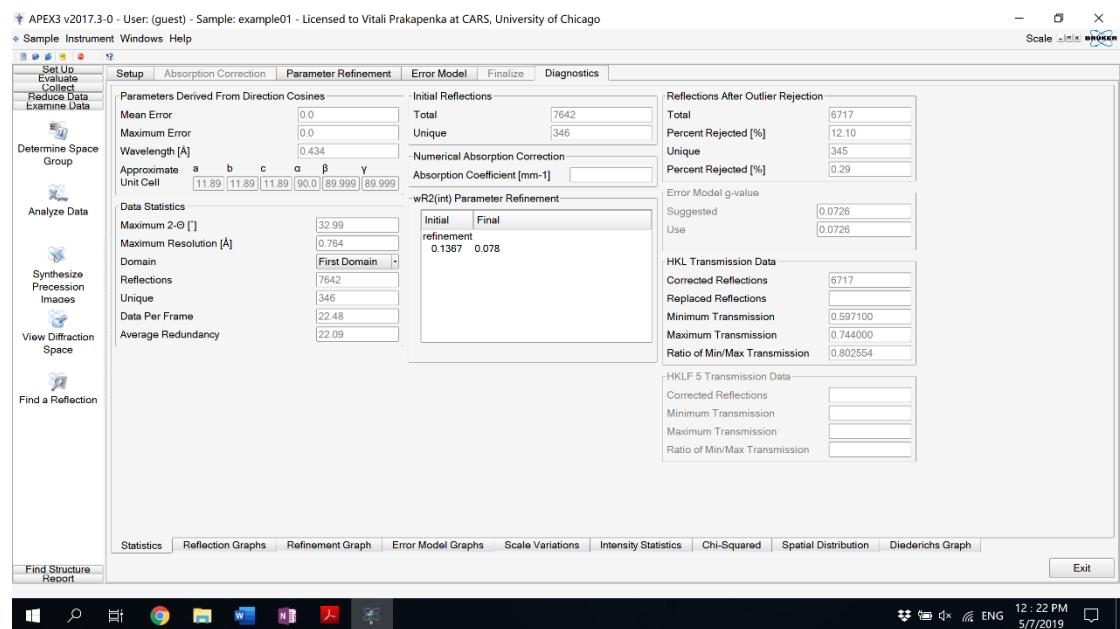
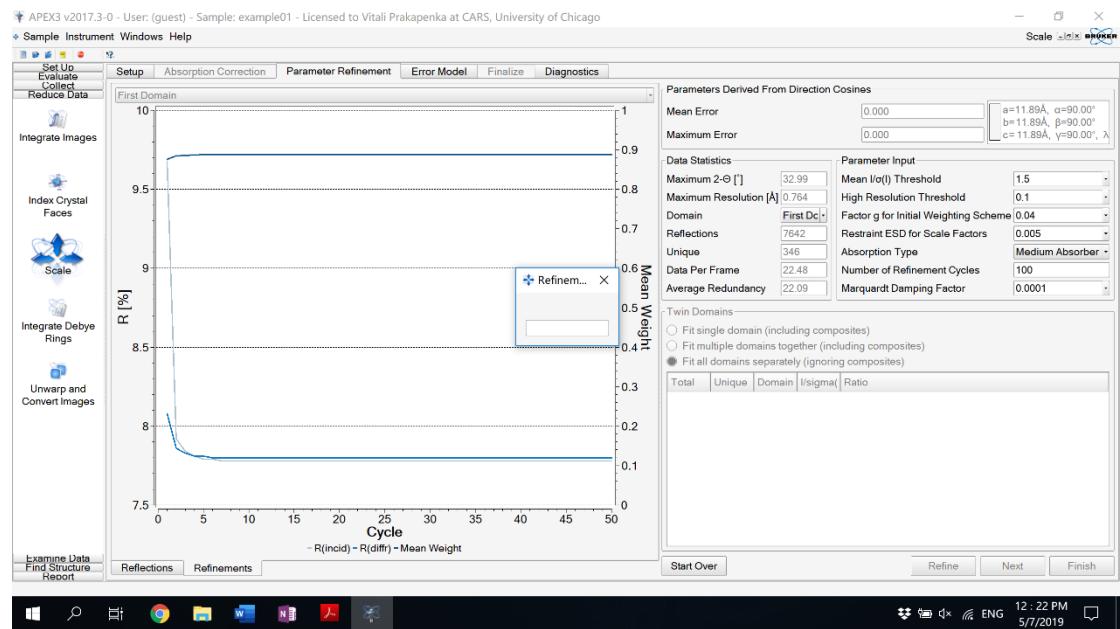


Go back to the Task Bar's 'Reduce Data', click the 'Scale' icon

Click the 'Start' button.



Choose an appropriate value of the ‘Number of Refinement Cycles’. Start Refine by clicking the ‘Refine’ button. Click the ‘next’ and ‘Finish’ button to finalize the process.

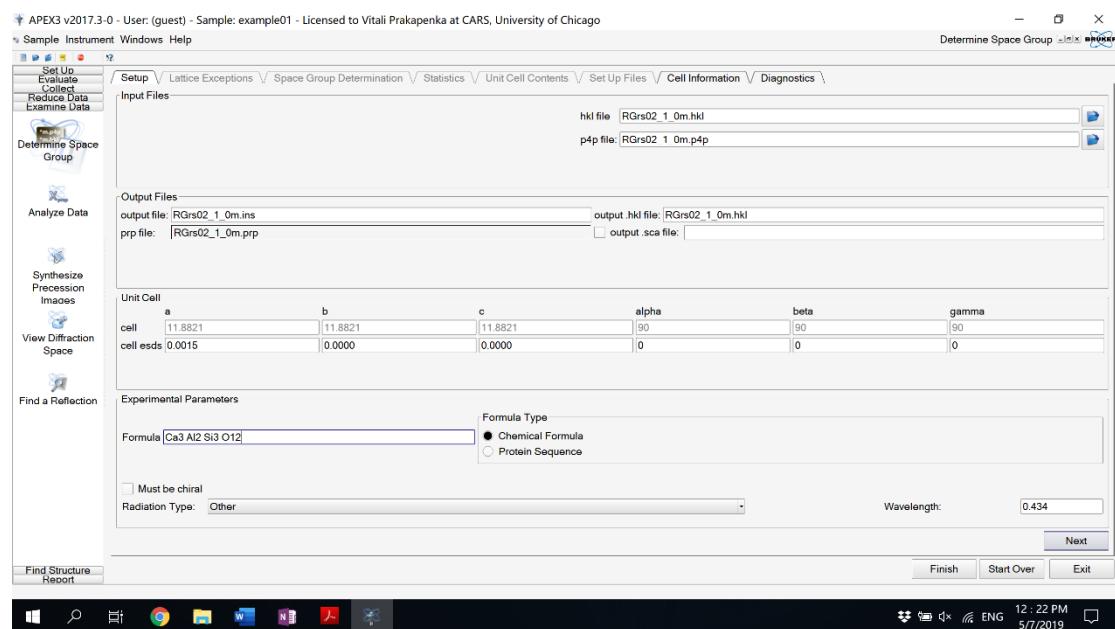


8. Examining Data

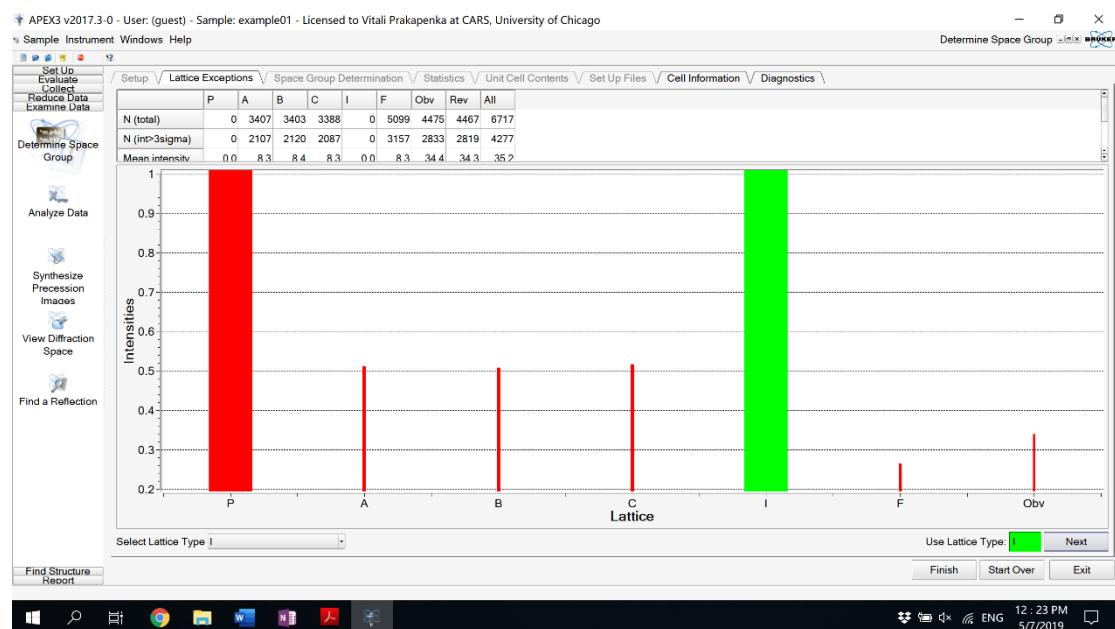
Go to the Task Bar's 'Examining Data', click the 'Determine Space Group'

icon .

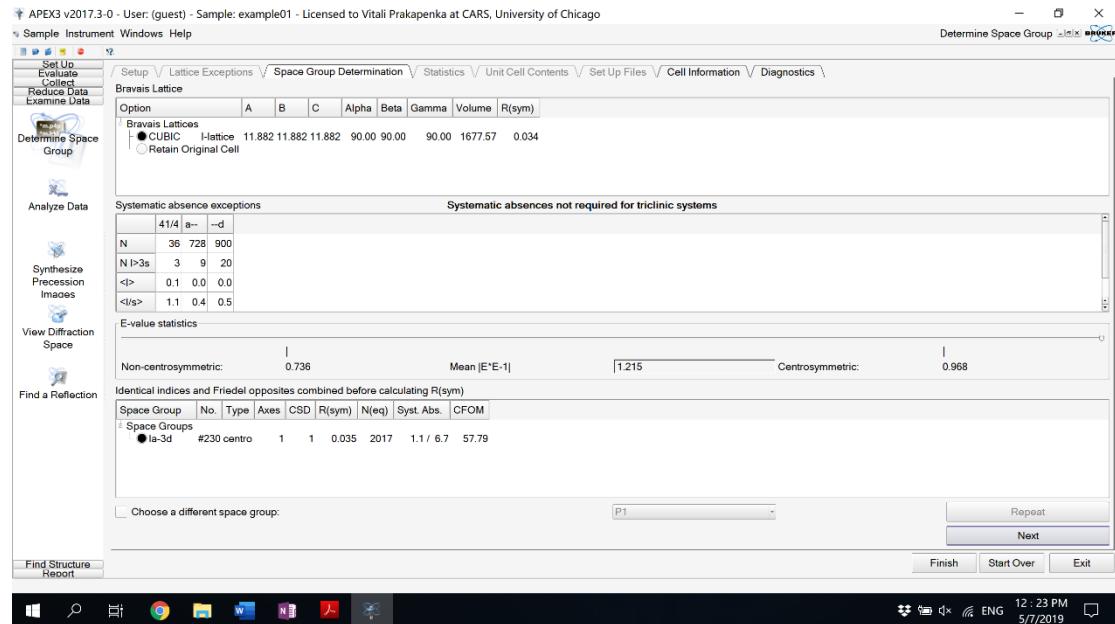
Under the 'Setup' tab, input your chemical formula in the box after the 'Formula'.



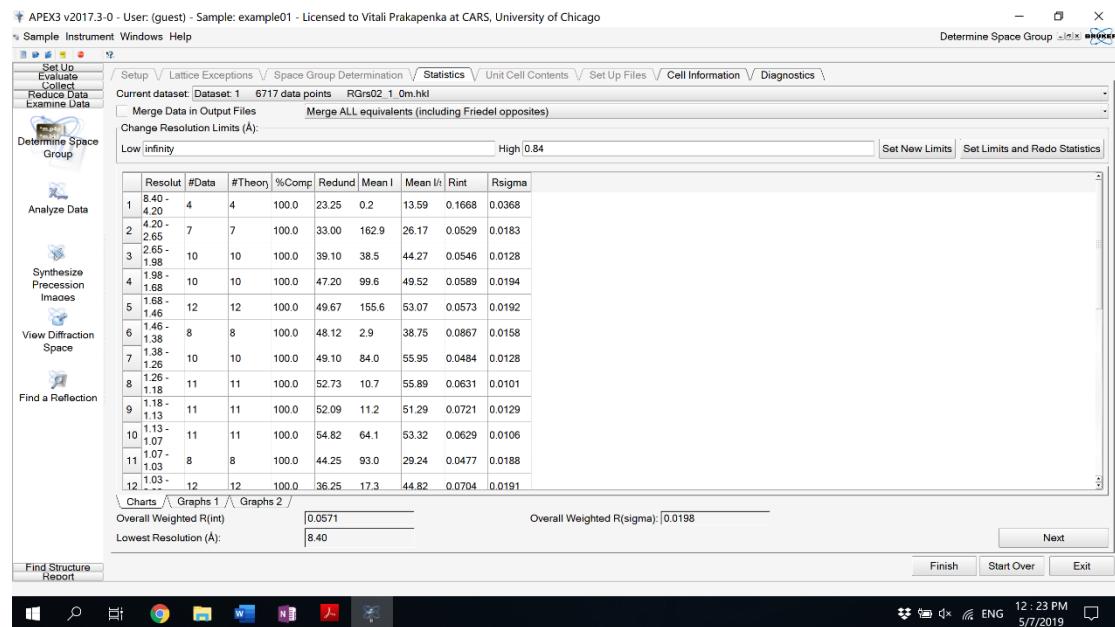
Click the 'Next' button, go to the 'Lattice Exceptions' tab, the green bar is the lattice type recommended by the software.



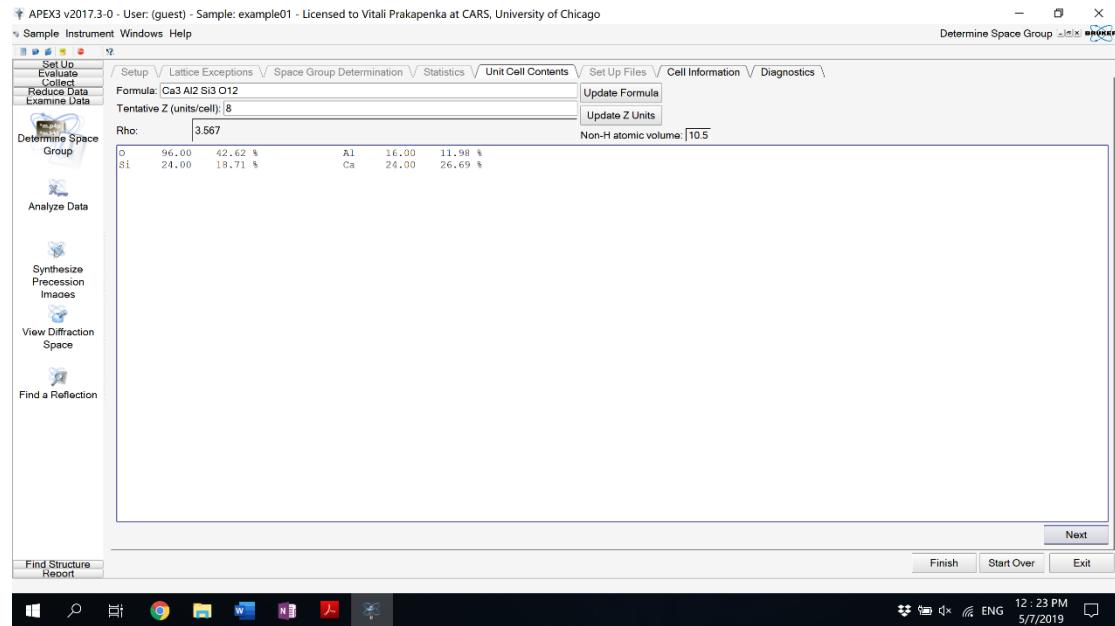
Click the ‘Next’ button, go to the ‘Space Group Determination’ tab. Under this tab, you can select the Bravais Lattice and Space group from the list.



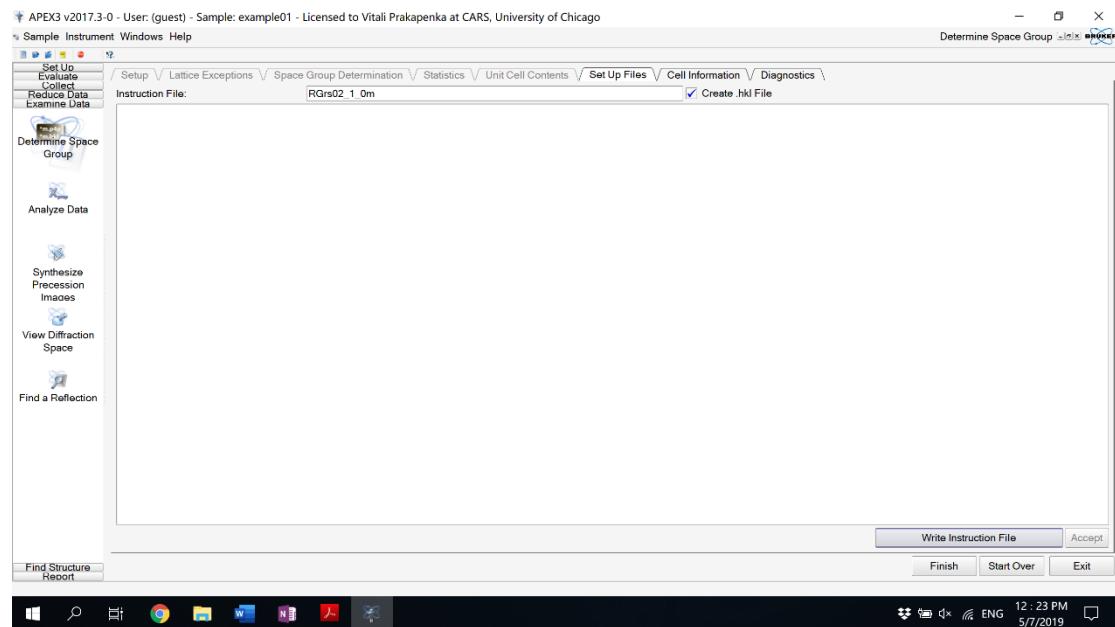
Click the ‘Next’ button, go to the ‘Statistic’ tab. Under this tab, you can adjust the statistic parameters if desired.

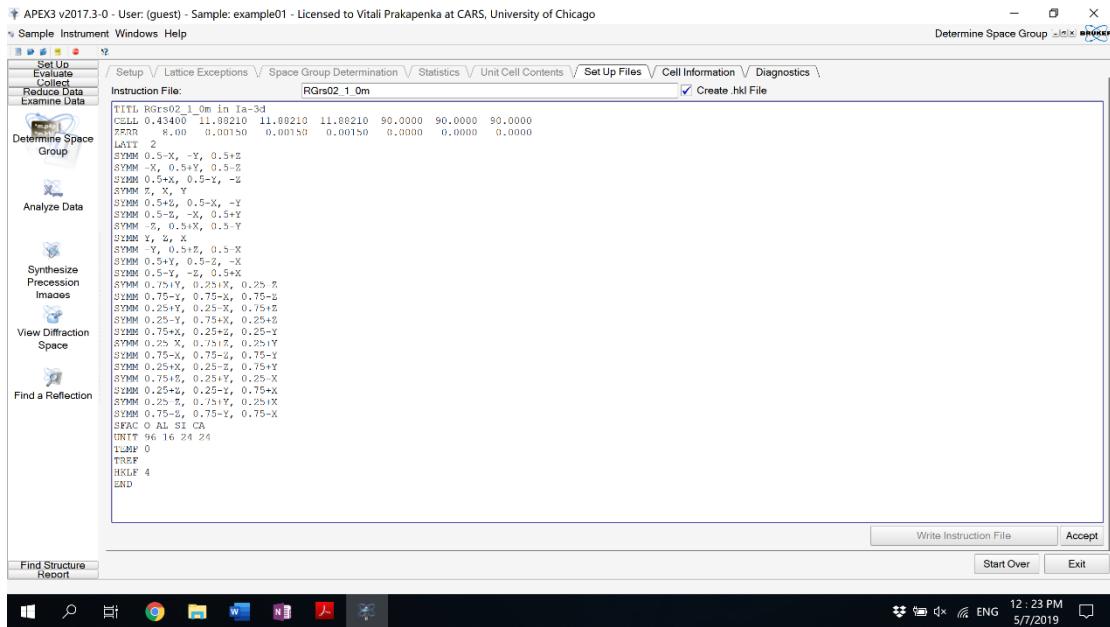


Click ‘Next’ button, go to the ‘Unit Cell Contents’ tab. This tab shows the formula, a tentative Z number, density and the atomic volume.



Click ‘Next’ button, go to the ‘Set Up Files’ tab. Under this tab, you can change the name of the current instruction file by typing a new name in the ‘Instruction File’ field. Check the ‘Create .hkl File’. Click the ‘Write Instruction File’ and ‘Accept’ button, to write the .ins file.





9. Move on to structure refinement

Now you have a folder named as ‘work’ in your data folder, which contains the .hkl and .ins file. Then you can proceed to the structure refinement process.