RecipLatView
SCD Reciprocal Lattice Viewer

Introduction:

The reciprocal lattice viewer is designed to allow users to work with single crystal diffraction data in a three-dimensional environment by displaying voxels in reciprocal space that correspond exactly to time-of-flight histogram bins from the area detectors.

Getting Started:

Launching from ISAW

● To launch the reciprocal lattice viewer from ISAW, follow the menus:
Macros > Instrument Type > TOF_NSCD > SCDReciprocalLattice

● A new window will open containing several options.

Data Directory - Choose the directory where run data is stored.

Calibration File - Select a calibration file if you have one.

Orientation Matrix - Select an orientation matrix if you have one.

Run Numbers - This specifies the run numbers to be used. Multiple runs should be separated by commas; a set of runs can be represented by separating the beginning and end value with a colon. For example: 8336:8339, 8341

Threshold Level - This is used to limit how many bins (counts) are displayed in the viewer. If the value is set too low then many low-count bins will be included causing the system to slow down. 60 is usually a safe starting value.

Show Contours - Selecting this option adds contour lines to the voxels; turning this option on requires extra computing power.

Contour Level - This is used to limit how many contour [somethings] are displayed in the viewer. If "Show Contours" is used then this should initially be set to a higher number.

Show Regions - This is used to show the volume covered by the detector.

Show HKL Markers - This option allows the user to turn show marks at integer HKL points. Selection of this option requires that an orientation matrix is loaded.

Calculate FFTs – This option allows for initial calculation of FFTs when the data is loaded.

- When you are finished, press **Apply** and the Reciprocal Lattice Viewer will open.

Launching from a command line

- To launch the reciprocal lattice viewer from a command line type the following:

java -mx1000M DataSetTools.trial.GL_RecipPlaneView -D/usr2/ARGONNE_DATA/SCD_MOSIH_DUAL -T60 -Cinstprm.dat -R8760:8771

-**D** specifies the data directory
-**T** specifies an initial threshold
-**C** specifies the name of a calibration file in the data directory
-**R** specifies a sequence of run numbers
-**O** specifies an orientation matrix in the data directory

Note:
If an orientation matrix is specified, the reciprocal axes a*,b*,c* will be displayed. If no orientation matrix is specified or found, the axes are the Qx,Qy,Qz axes in "laboratory" coordinates.

If no calibration file is specified, the engineering values for the detector size and position are used.

Available Controls:

View Controls

● The view controls are always at the top of the control panel.

Altitude – This controls the angle above or below the horizontal plane in the viewing window.

Azimuth – This controls rotation around the vertical axis in the viewing window.

Distance – This controls the distance from the origin displayed in the viewing window.

Projection – This option allows the user to choose between orthographic projection and perspective projection.

Tip: Use your keyboard's arrow keys for fine adjustment.

View Tab

Peaks Threshold - This controls the cutoff level above which bins are displayed and used in subsequent processing.

Iso-surface Threshold - This controls the single level at which iso-level contours are drawn. Lower values for this setting require more computing power.

Iso-surface - This option allows the user to turn on/off iso-surfaces in the viewing window.

Detector Coverage - This option allows the user to turn on/off a visual representation of the detector's coverage area.

Integer HKL points - This option allows the user to turn on/off marks at integer HKL points. This only works if an orientation matrix was loaded.

Calculate FFTs of Projections

This operation is used to approximate the direction of normals for planes in a crystalline lattice within reciprocal space. The direction of three linear independent plane normals can then be used to generate an orientation matrix for the sample.

FFT projects the Q values in reciprocal space into one-dimensional lines pointing in various directions. These lines are Fourier transformed to identify directions that represent normals to families of planes in reciprocal space. The Fourier transformed projected data is displayed as rows in an image view where peaks are arranged in order of increasing d-spacing.

The plane normals are displayed in the three-dimensional view as a sequence of light blue boxes pointing in the direction of the normal. The distance between these boxes corresponds to the reciprocal length of an edge of the unit cell in real space. The d-spacing can be found on the border of the graph part of the image view.

Planes Tab

Qxyz Readout - This readout displays the Qxyz coordinates of the currently selected peak.

Origin Control - This is used to change the view center of the viewer window by clicking on a peak and then pressing the **Select** button. Pressing the **Select** button will also choose the initial point for two user-specified vectors that are used to identify a family of planes. The **Reset** button will reset the view center to the Qxyz origin.

(+) Control - This is used to change the endpoint of one user-specified vector to the last peak that the user clicked on.

(*) Control - This is used to change the endpoint of a second user-specified vector to the last peak that the user clicked on.

Constant h Planes, Constant k Planes, Constant l planes

- These allow the user to specify a family of planes with a constant h, k, or l value in reciprocal space.

    User - Clicking on User-> will create the constant h, k, or l plane information based on the normal to the plane

determined by the two user-specified vectors **+** and *****.

FFT - Clicking FFT-> will create the constant h, k, or l plane information based on the normal direction from the last selected row in the FFT image view window.

Filter On/Off – This option allows the user to restrict visible data to those which lie within 0.1 units of an integer value.

- The normal vector and d-spacing that correspond to the family of planes defined are displayed in the control windows. The values for d-spacing can be edited if better values are known.

Write Orientation Matrix

- When constant h, k, and l planes have been defined, the corresponding orientation matrix can be written to a file.

Write Peak Data File

- A list of the bins above the specified threshold can be written to a file.

Exercise: Determining the Orientation Matrix for Oxalic Acid Using the Reciprocal Lattice Viewer

The SCD Reciprocal Lattice Viewer in ISAW has proven to be very useful in determining the orientation matrix of a sample when it is difficult to index the peaks using Blind using the Initial Wizard. This exercise will take you through the steps needed to load data into the viewer, determine the unit cell and write an orientation matrix.

The data for oxalic acid at 80 K are run files 9164:9183 and are included in the ISAW tutorial. You will not need all of these data files to determine the unit cell and write an orientation matrix.

1. Start the viewer by following the menus: **Macros > Instrument Type > TOF_NSCD > SCDReciprocalLattice**
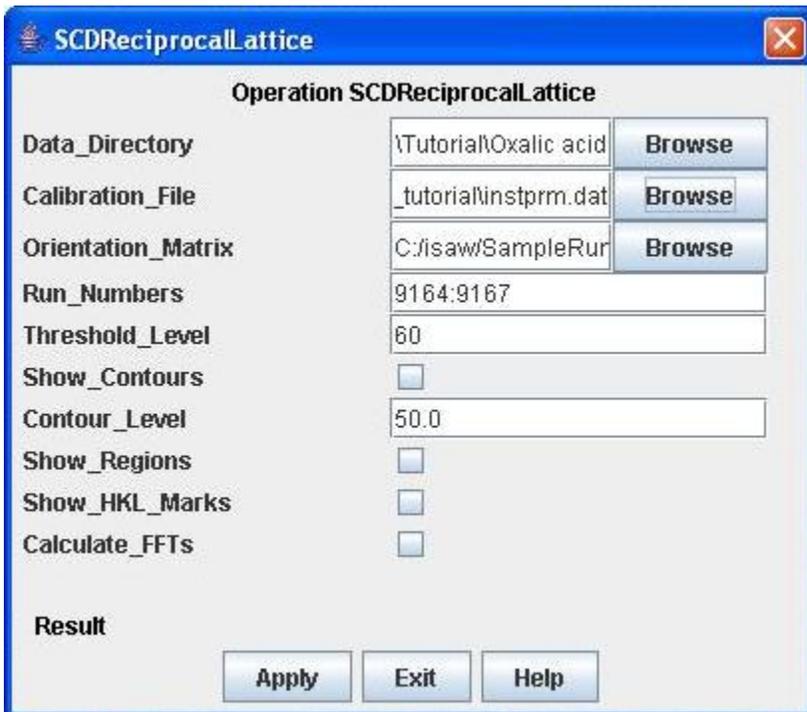

2. Load the run files 9164:9167 from the directory the data is in. Use the calibration file *instprm.dat* from the ISAW tutorial. You do not need an orientation matrix for this exercise – you will determine this from the raw data using the reciprocal space viewer.

Use the defaults for the threshold level and contour level. In the case that the data is weaker than the data for this exercise (orientation data taken on SCD is often a 30 minute histogram and therefore not as many counts may be collected at the detector), you can reduce the threshold level to increase the number of reflections included in the viewed data set. Do not

check the other boxes at this time.

Click **Apply** to view the data from the run files in the reciprocal space viewer.
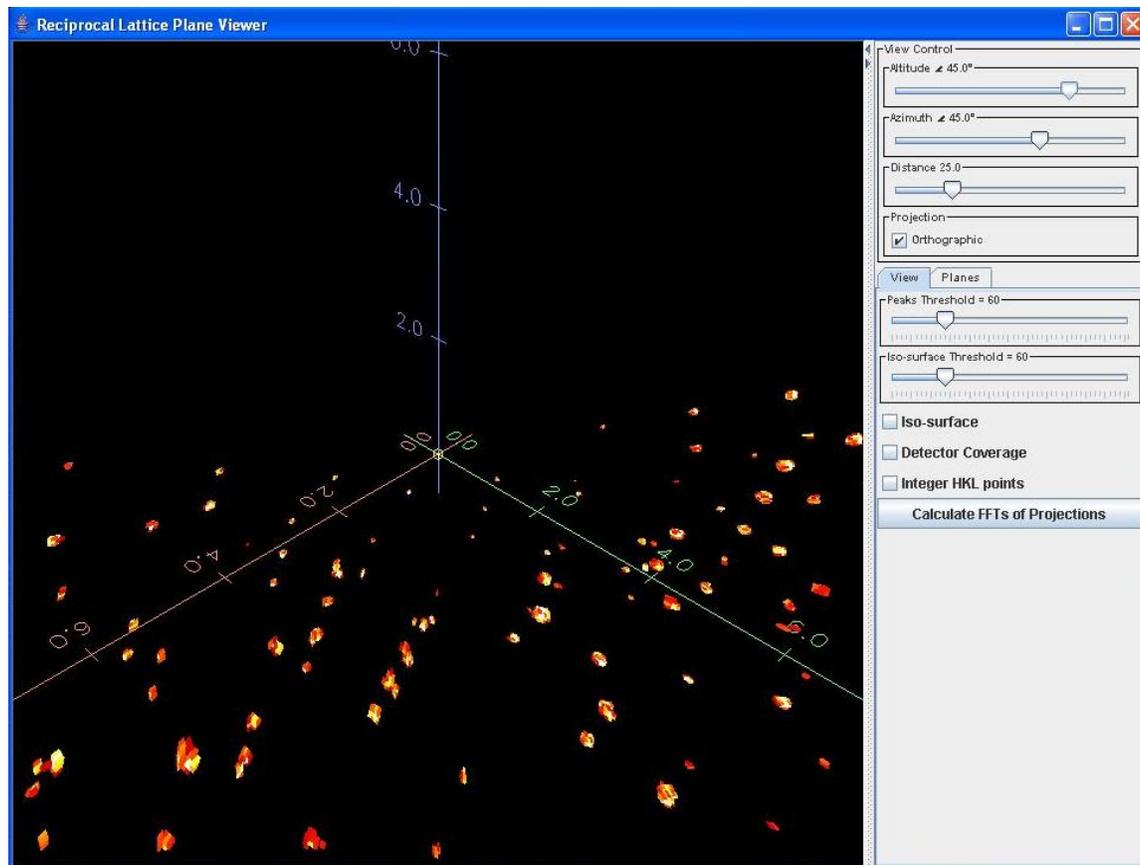
Your data screen should look similar to this. The red, green and blue axes are the x, y and z "laboratory" axes in Q-space (reciprocal space), respectively. The origin of the plot is clearly labeled where these axes meet. Each voxel is an individual reflection at its x, y and z position in reciprocal space. Clicking on the **Planes** tab and then clicking on a reflection will give you the Qxyz coordinates of that reflection.

Use the **Altitude** and **Azimuth** adjusters to move the reflections around until you have aligned the reflections into a series of planes. You can use the arrow keys on the keyboard to fine-tune the movement.

3. You can use the constant h, k and l plane function to find the d-spacings and write an orientation matrix for the sample. First, orient the reflections so that you are viewing a family of planes: if you used runs 9164:9167, try moving the distance to approximately 31.8, the altitude to 43.1 and the azimuth to 44.1. You should obtain a screen like the following:
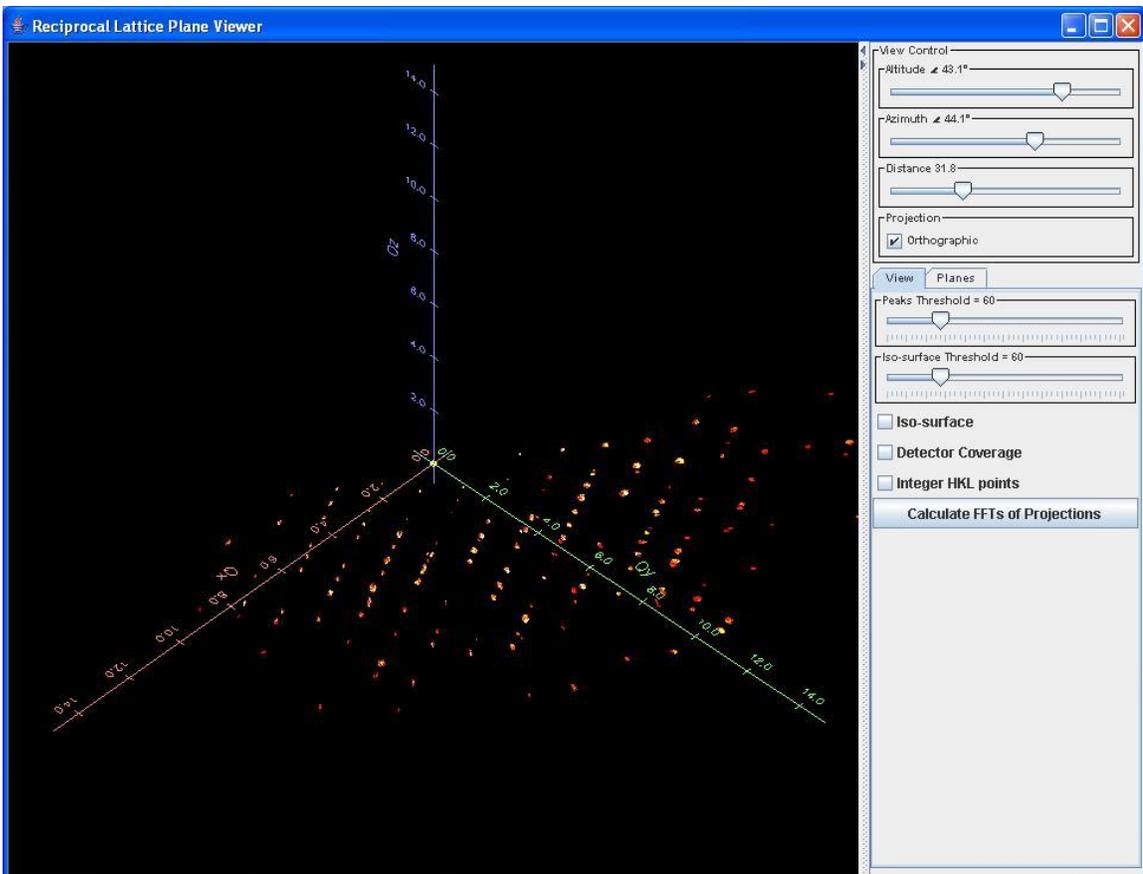
Using the **Planes** tab, we can now define a plane of constant h, k or l by clicking on three non-linear reflections within one of these planes. Clicking on the **User->** button will calculate the normal to our defined plane, and you should then have the interplanar d-spacing between the family of planes shown above. To step through the process, you can follow these steps:
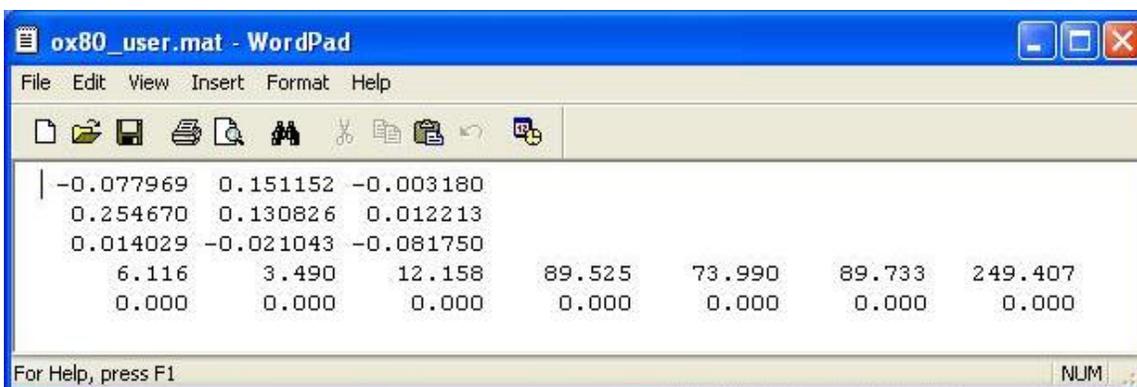
    a. Zoom back in to a distance where you can see the planes more closely, like 11.6.

    b. Use the azimuth slider or your keyboard to move the planes to one side, so that instead of having all of the reflections lined up precisely, you can see more of the reflections that make up these families of planes. Try an azimuth angle of 49.6.

    c. Now you should redefine your origin and choose two other reflections that are non-collinear with your origin of choice. Click on a reflection in a plane and click on **Select** under the origin to define that reflection as the new origin. Do the same using two other reflections in that plane and clicking on **Select +** and **Select ***. The result should be a user defined plane:
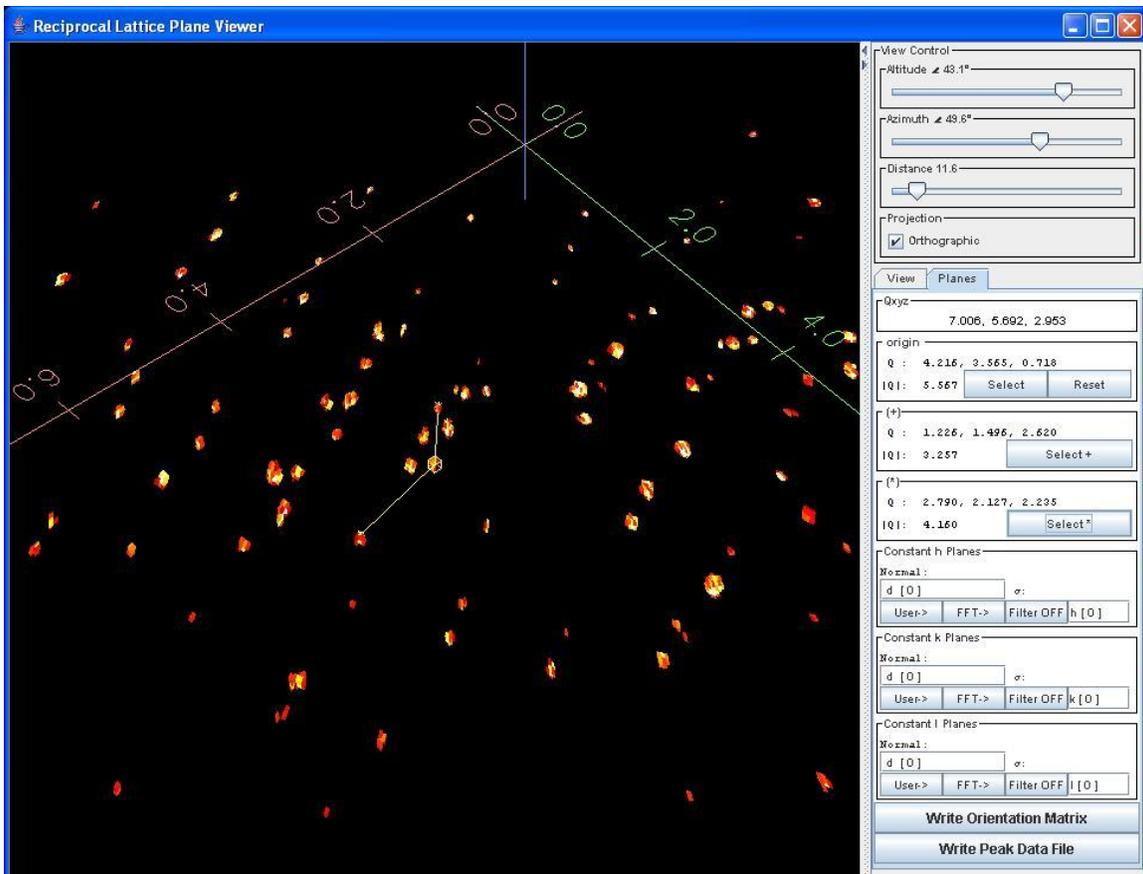
d. You can see now that the origin is no longer at 0, 0, 0; it is the central reflection surrounded by a box. (Don't worry; you can reset the origin to 0, 0, 0 by clicking on Reset under origin.) The two other reflections chosen by **Select +** and **Select *** have a vector drawn to them. The three reflections are non-collinear and define a plane.

e. Click on **User->** in a Constant h Planes box to calculate the normal to the defined plane. The result in the d-spacing box should be in the neighborhood of 6.1 – exactly what we would expect for one of the unit cell lengths for oxalic acid. Notice that if you keep clicking on the **User->** button, the d-spacing may keep changing until it reaches a final value.

f. Reset the origin and locate a different family of planes. Try an altitude of 22.9 and an azimuth of -64.3. You will notice that the previously drawn vectors, now centered at the origin of 0, 0, 0, are pointing to two different planes. This is a good measure that you have located a different family of planes than you did previously at the beginning of step 3.
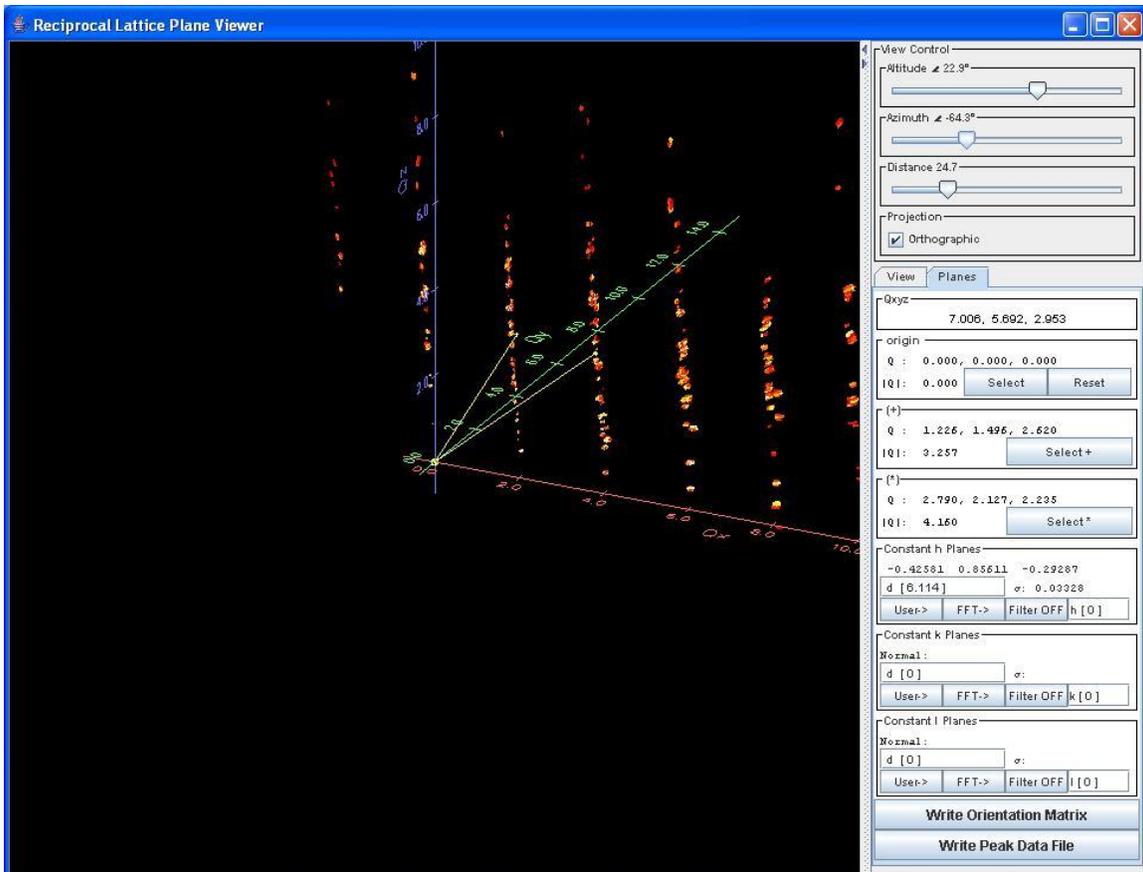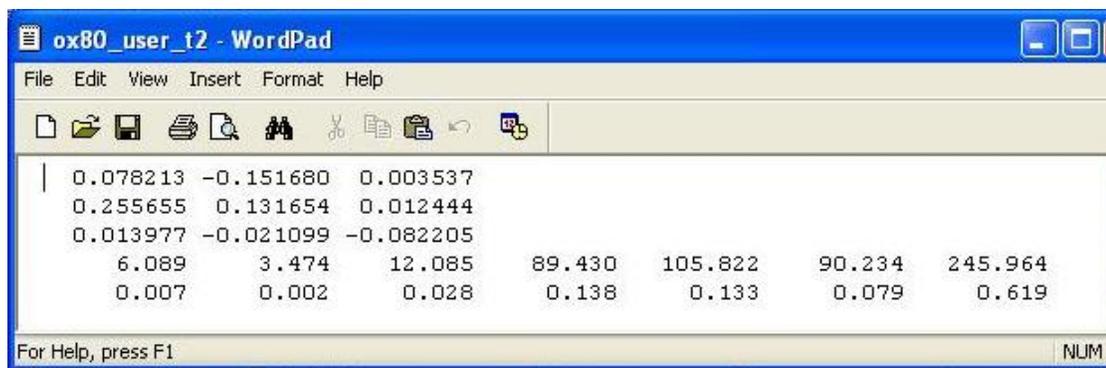
g. Repeat steps 3a-3e for this family of planes. Calculate the normal by clicking on **User->** in the Constant k Planes box. The resultant d-spacing should be approximately 3.4.

h. Find the third set of planes by resetting the origin and moving to an altitude of 1.2 and an azimuth of -62.4. (In this orientation, the plane to define runs horizontally.) The resulting d-spacing should be around 12.1.

i. With three user defined unit cell parameters, you may now write an orientation matrix by clicking on **Write Orientation Matrix**. You will be prompted for a name for your matrix – give it a different one if you have already determined an orientation matrix by other means so you do not lose your previous work. You can view the resulting matrix file by opening it with a text editor.

```
ox80_user.mat - WordPad
File  Edit  View  Insert  Format  Help

 -0.077969   0.151152  -0.003180
  0.254670   0.130826   0.012213
  0.014029  -0.021043  -0.081750
      6.116      3.490     12.158      89.525     73.990     89.733    249.407
      0.000      0.000      0.000       0.000      0.000      0.000      0.000

For Help, press F1                                                    NUM
```

j. You will notice that this resulting unit cell should be transformed to the standard monoclinic setting to achieve the correct β angle of 106°, but the cell is correct for the material.

k. Click on **Write Data Peak File** to get a list of peaks for the reflections shown in the viewer. The peak file that is written out by the Reciprocal Space Viewer is **not** in the correct format to be indexed by the matrix determined in this exercise.

Using the menu items in **Macros > Instrument Type > TOF_NSCD**, it is possible to index the peaks, refine the least squares matrix, transform the cell and integrate them in a manual fashion, bypassing the Wizard Menu. You can follow these steps in the **Macros** as if you were going through the Wizard itself once you have a peaks file:

   i. Search for peaks on the run file(s) using **Macros > Instrument Type > TOF_NSCD > find_multiple_peaks** (Initial Wizard Form 1). A *filename.peaks* file will be written according to input under Experiment Name on the form. This peaks file, unlike the one generated by the Reciprocal Lattice Viewer, can be indexed using the determined matrix.

   ii. Use **Macros > Instrument Type > TOF_NSCD > JIndex** (Initial Wizard Form 3) to index the Peaks file using the matrix file written by the Reciprocal Lattice Viewer. If you view the peaks file again, the peaks should be indexed with hkl values.

   iii. Use **Macros > Instrument Type > TOF_NSCD > JScalar** (Initial Wizard Form 4) to search for alternate symmetries. This may or may not give a proper transformation matrix.

   iv. Use **Macros > Instrument Type > TOF_NSCD > JLsqrs** (Initial Wizard Form 5) and the correct transformation matrix of **[[-1, 0, 0][0, 1, 1][0, 0, 1]]** to transform the monoclinic matrix into the standard setting. Use a matrix with a different name if you do not wish to overwrite your matrix determined using the Reciprocal Lattice Viewer. Check your new transformed matrix to insure that your new matrix is correct:

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```
ox80_user_t2 - WordPad

File  Edit  View  Insert  Format  Help

  0.078213 -0.151680  0.003537
  0.255655  0.131654  0.012444
  0.013977 -0.021099 -0.082205
     6.089     3.474    12.085    89.430   105.822    90.234   245.964
     0.007     0.002     0.028     0.138     0.133     0.079     0.619

For Help, press F1                                                      NUM
```

    v. You should then use **Macros > Instrument Type > TOF_NSCD > JIndex** (Initial Wizard Form 6) again with the new matrix file to reindex the peaks file so they are consistent with the transformed unit cell.

Still confused? There are future plans to include an option to index the peaks file with a pre-determined matrix in Form 2 (JBlind) of the Initial Wizard in ISAW, so that these steps through the Macros menu may be eliminated. These steps should guide you, however, in transforming an orientation matrix in a non-standard setting using the Macros menus in ISAW.

    4. You may also use the **Calculate FFTs of Projections** option under the **View** tab to write an orientation matrix and peaks file.

        a. Click on the **Calculate FFTs of Projections** button in the **View** pane. A new window will appear showing the Fourier transforms of the reflections shown. Choose **Options > Color Scale > Optimal** from the menu in the new window to have the FFTs appear as described in the preceding documentation.
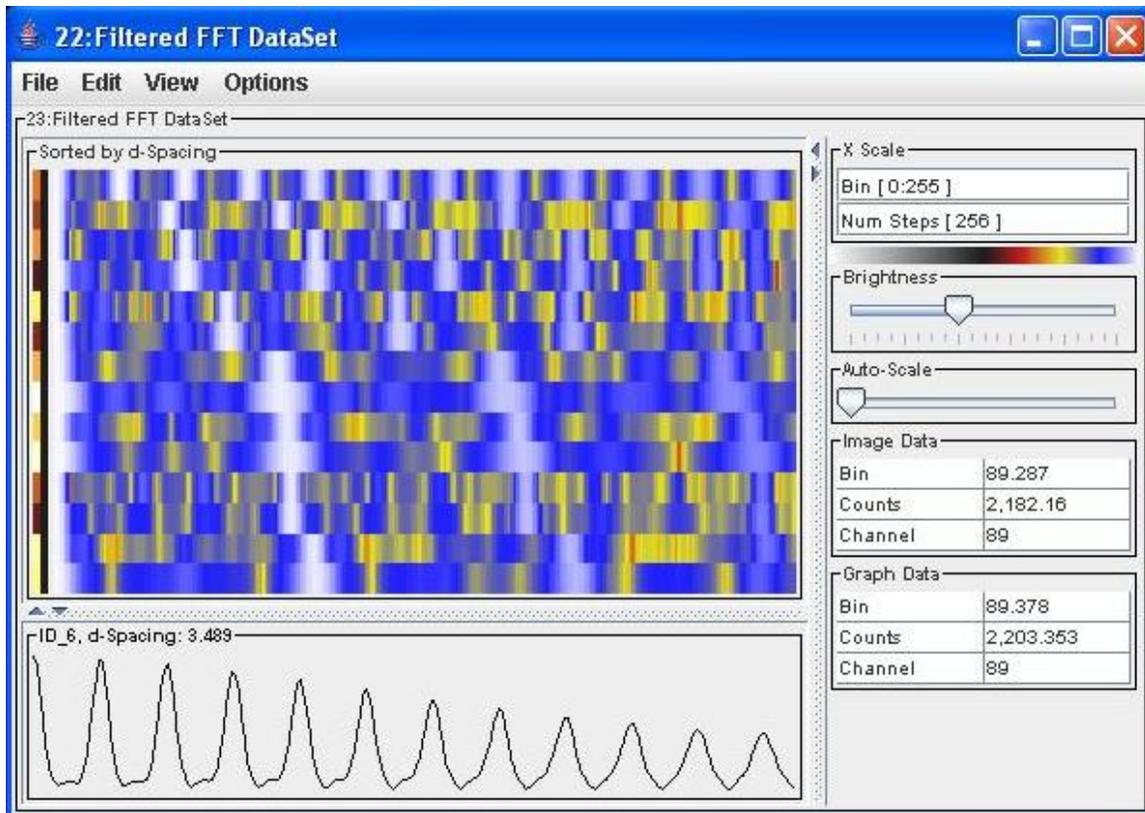
b. Click on the first row containing the white boxes. The plane normals are displayed in the three-dimensional view as a sequence of light blue boxes pointing in the direction of the normal. The distance between these boxes corresponds to the reciprocal length of an edge of the unit cell in real space. The d-spacing can be found on the border of the graph part of the image view.
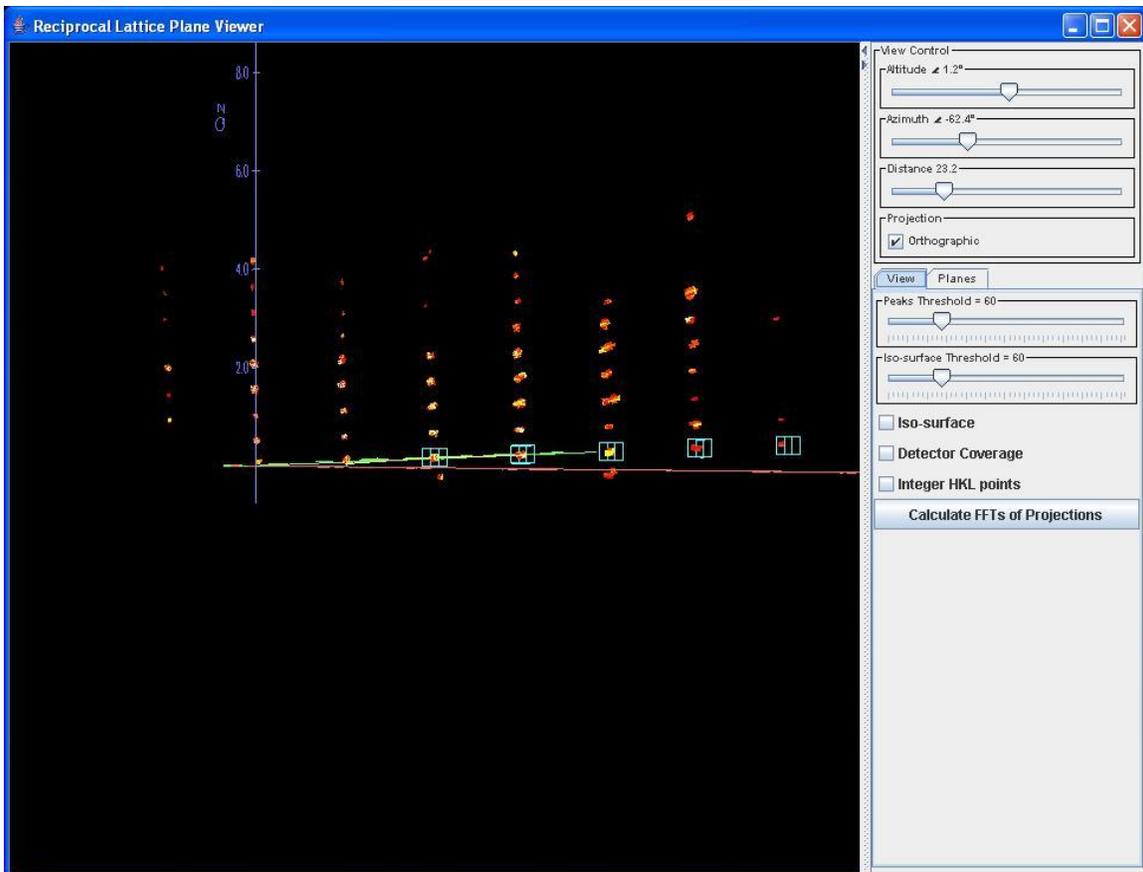
c. Here you can see how the 3.4 Å cell edge relates to the family of planes in the reciprocal space viewer as shown at the angles from step 3.h. above. You can scroll through the Fourier transforms by clicking through them or using the arrows on your keyboard. Click on the d-spacing that corresponds to the 12.4 Å cell edge to see how this relates to the same graph.
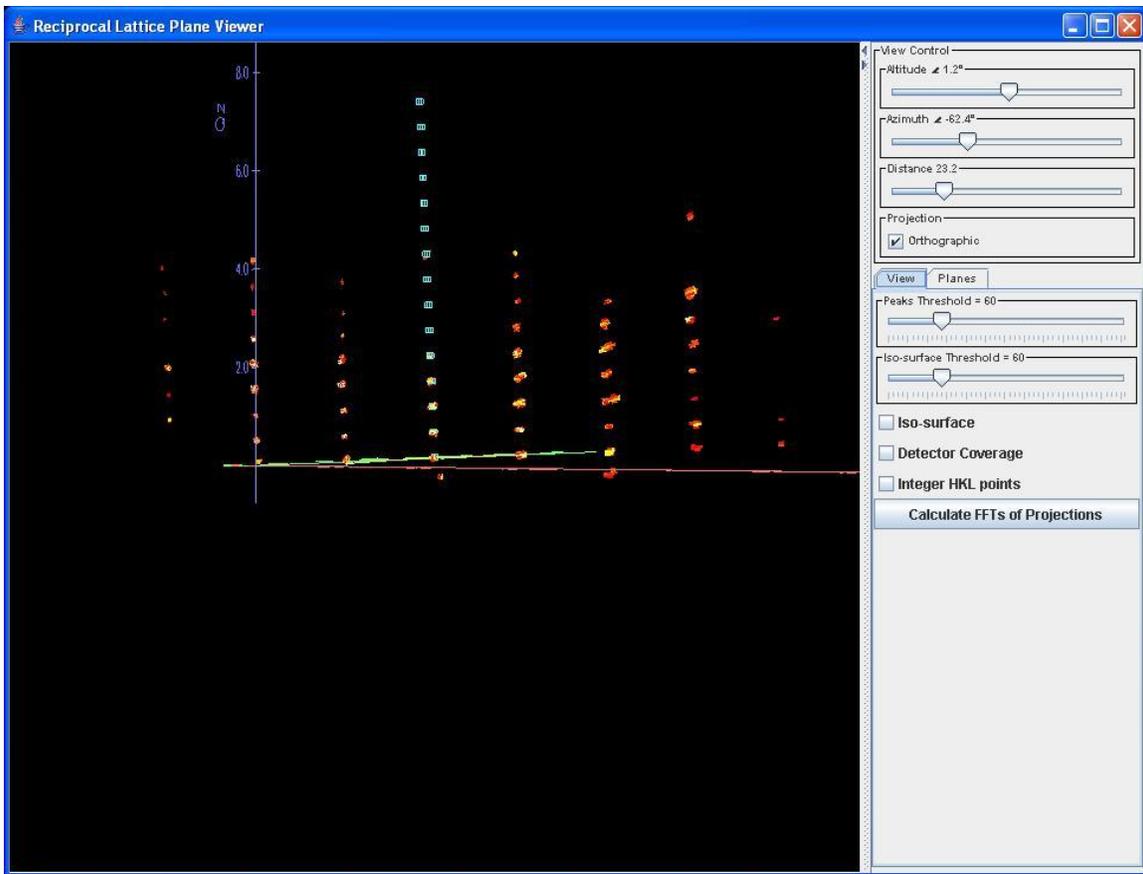
d. To use the Fourier transforms to define constant h, k or l planes: select the d-spacing that best defines the interplanar spacing, then click on **FFT->** in the Constant h, k or l Planes window under the **Planes** tab.

e. Follow the same procedure as for 3.1-k to write an orientation matrix and peaks file.