Using the R-Axis Spider to run powder samples

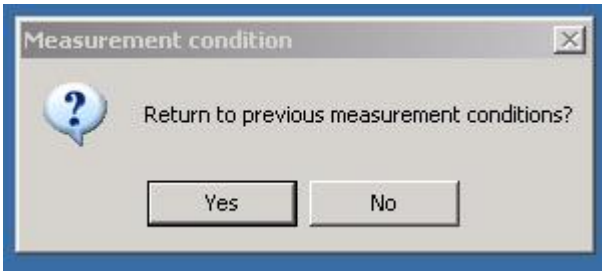
You can run loose powder samples on the R-Axis Spider. Prepare the sample as you would for the D8. You can use the agate mortar and pestle to grind the sample if the particles need to be ground to uniform size beforehand. Use the same kind of loop that you would use for a single crystal. A small amount of mineral oil mixed with the powder can help keep the sample together on the loop. The mineral oil can also protect air sensitive materials. Powder samples that contain solvent molecules can be protected from solvent loss by loading the samples in thin walled glass capillaries with some of the mother liquor. Allow the powder to settle with the solvent above protecting the sample. Align the powder region in the center of the beam.

Instead of using Crystal_Clear, use RINT_XRD XG for powder samples. Before starting the program, it is useful to create the data directory first. You will be asked for a data at the start.

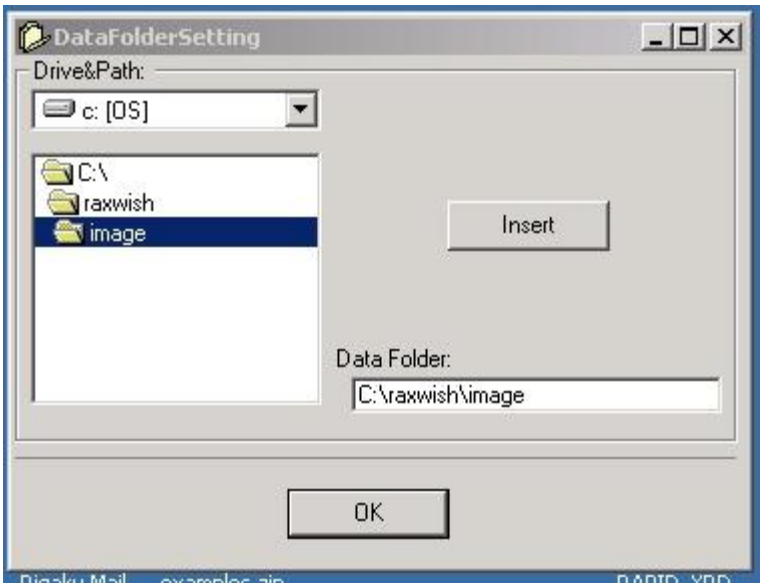
Double-click

to start.

You will be asked if you wish to return to the previous data collection parameters.

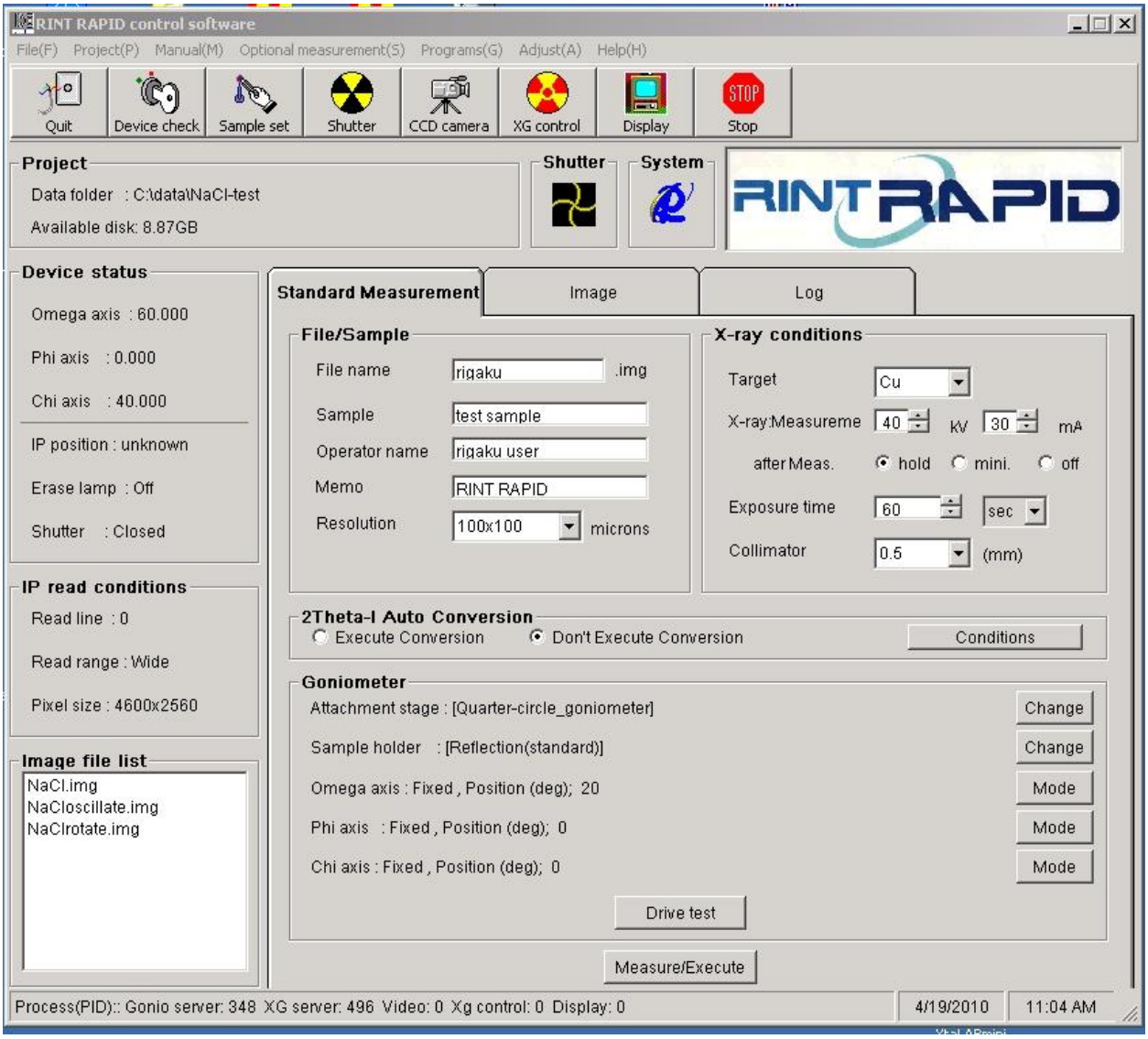


If you answer yes, you experimental setup will be the same as the one run just before you. If you answer no, the default experimental conditions will be set. The default setup uses an incorrect setting for KV and mA, which should be set to 40/40.



After selecting the proper directory, you must hit the Insert button. The selected directory will be displayed in the Data Folder entry. If you forget to hit Insert, you will go to the default directory, C:\raxwish\image.

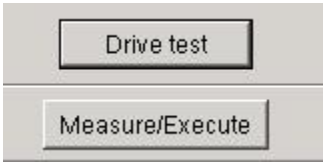
The program control panel will appear as below.



You should change the File name to something that makes sense to you. Under Sample, you can enter some additional useful information about the sample.

On the Spider, the target radiation is Cu. Change the X-ray measurement settings to 40 KV and 40 mA. A sample exposure time of 10 minutes is usually sufficient.

As with a single crystal, you have to align the sample. Set omega axis, fixed position to 70°, set chi axis, fixed position to 0° and the phi axis, fixed position to 0°. Hit the Drive test button



on the lower middle portion of the control stream. The angles should

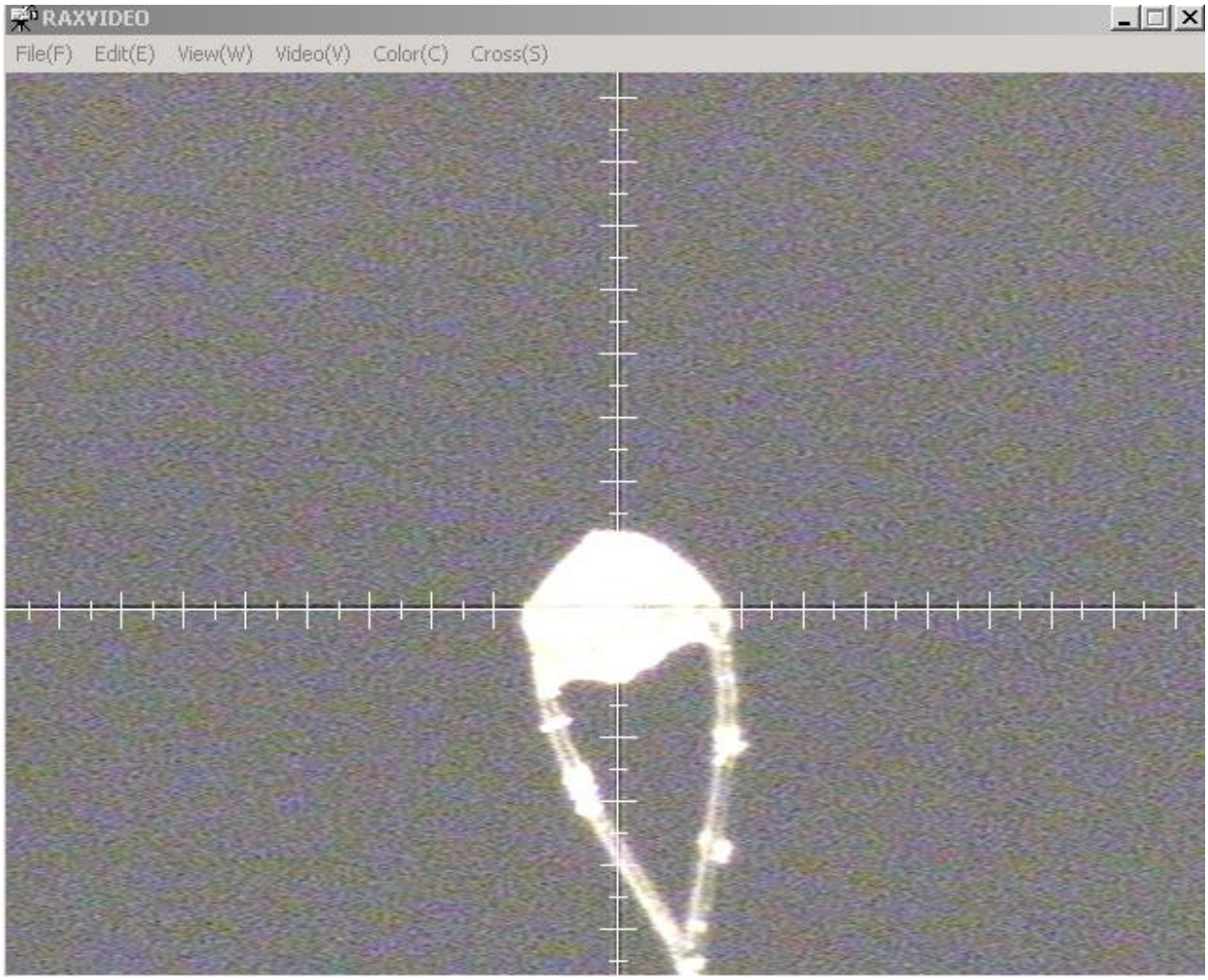
drive to the assigned location. Hit the CCD-camera icon,

to view the sample using the RAXVideo routine as in Crystal_Clear. Align the sample by selecting various settings of phi of 0, 90, 180 and 270° until the sample stays in the cross-hairs while rotating. After each drive instruction is issued, you must hit the stop button

before you can enter another drive instruction. Note the top portion of the control menu.

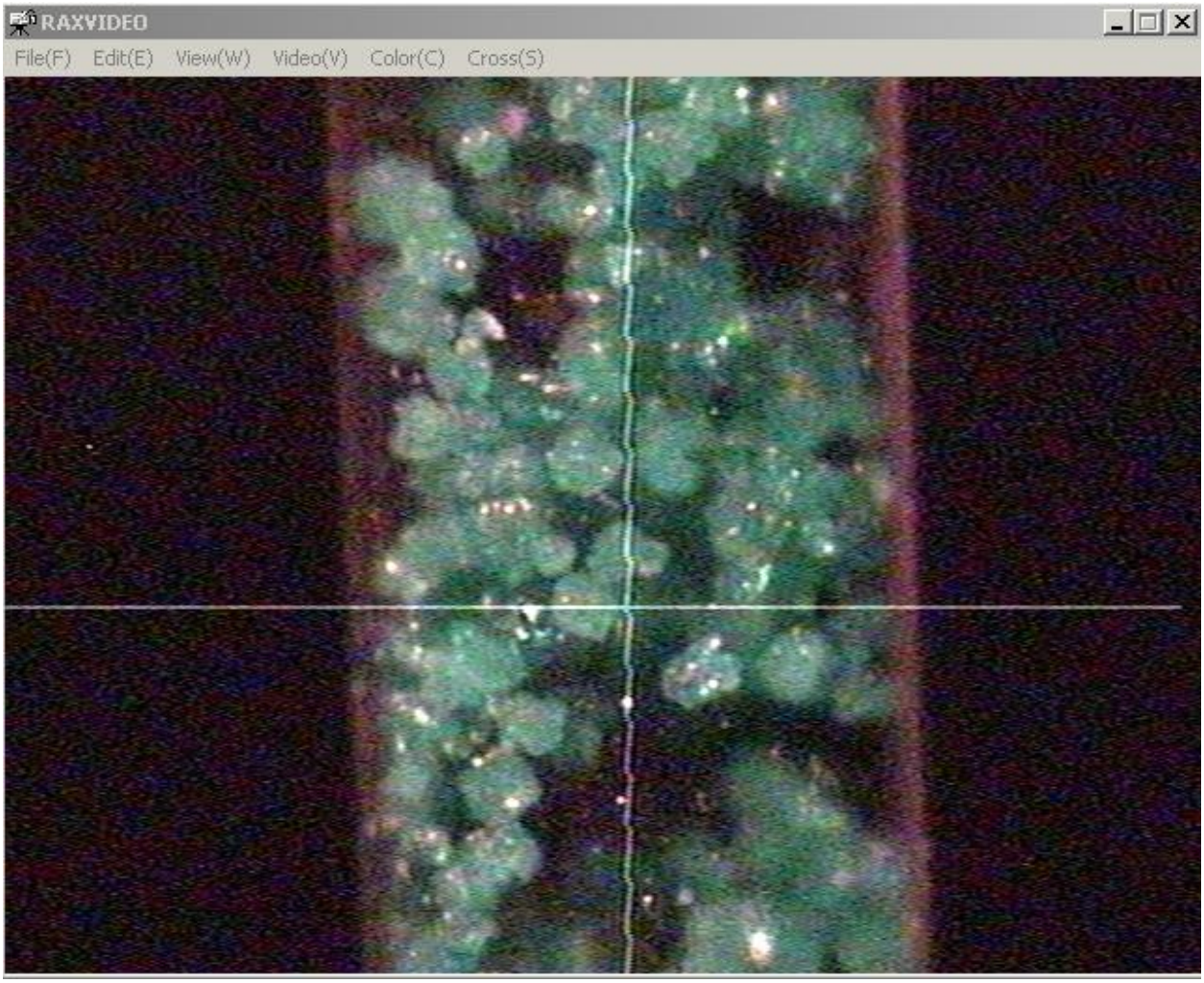
means that the program is ready to accept instructions.

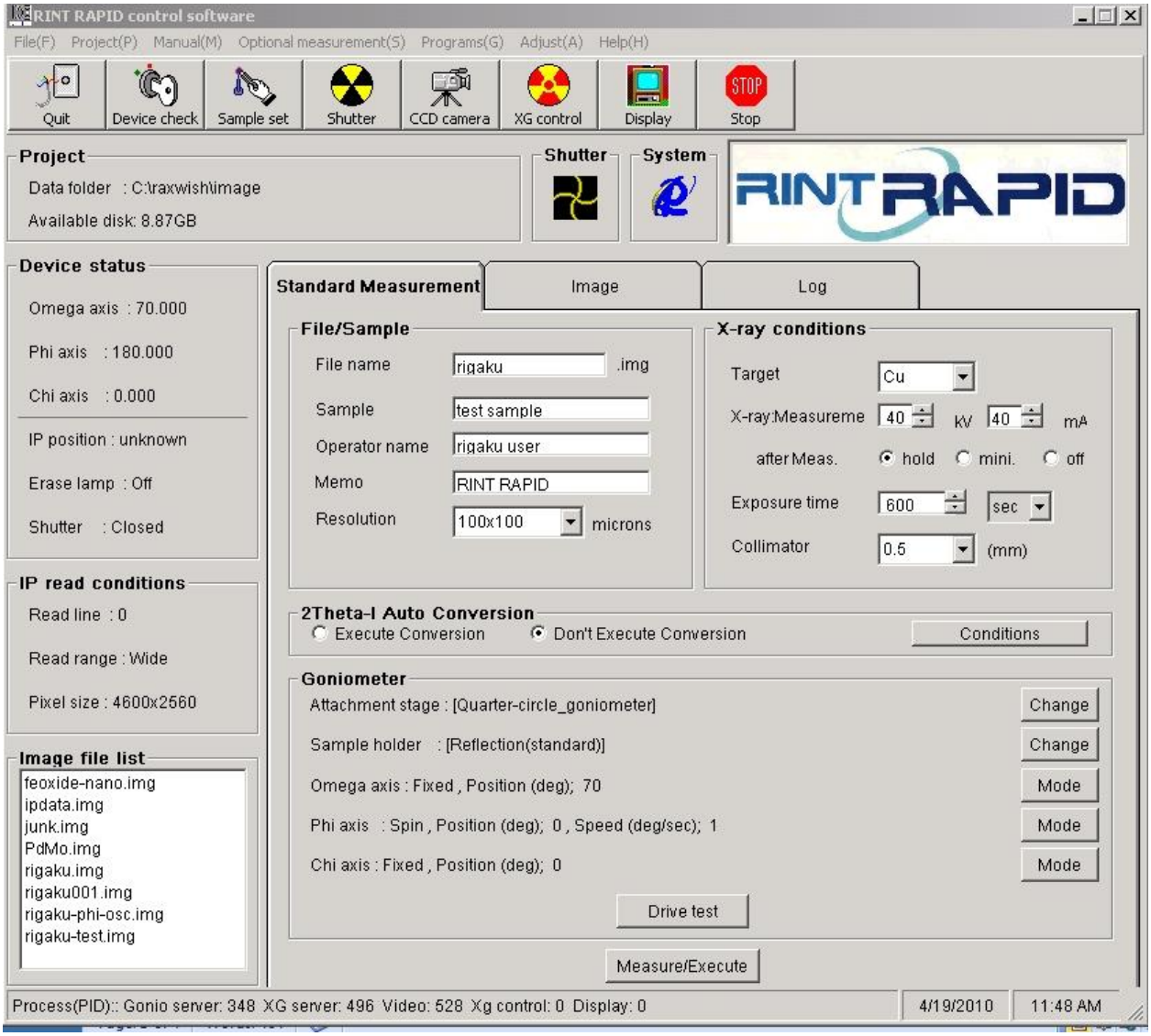
means the program is busy. The program can be slow to respond depending on what it is doing so be patient.



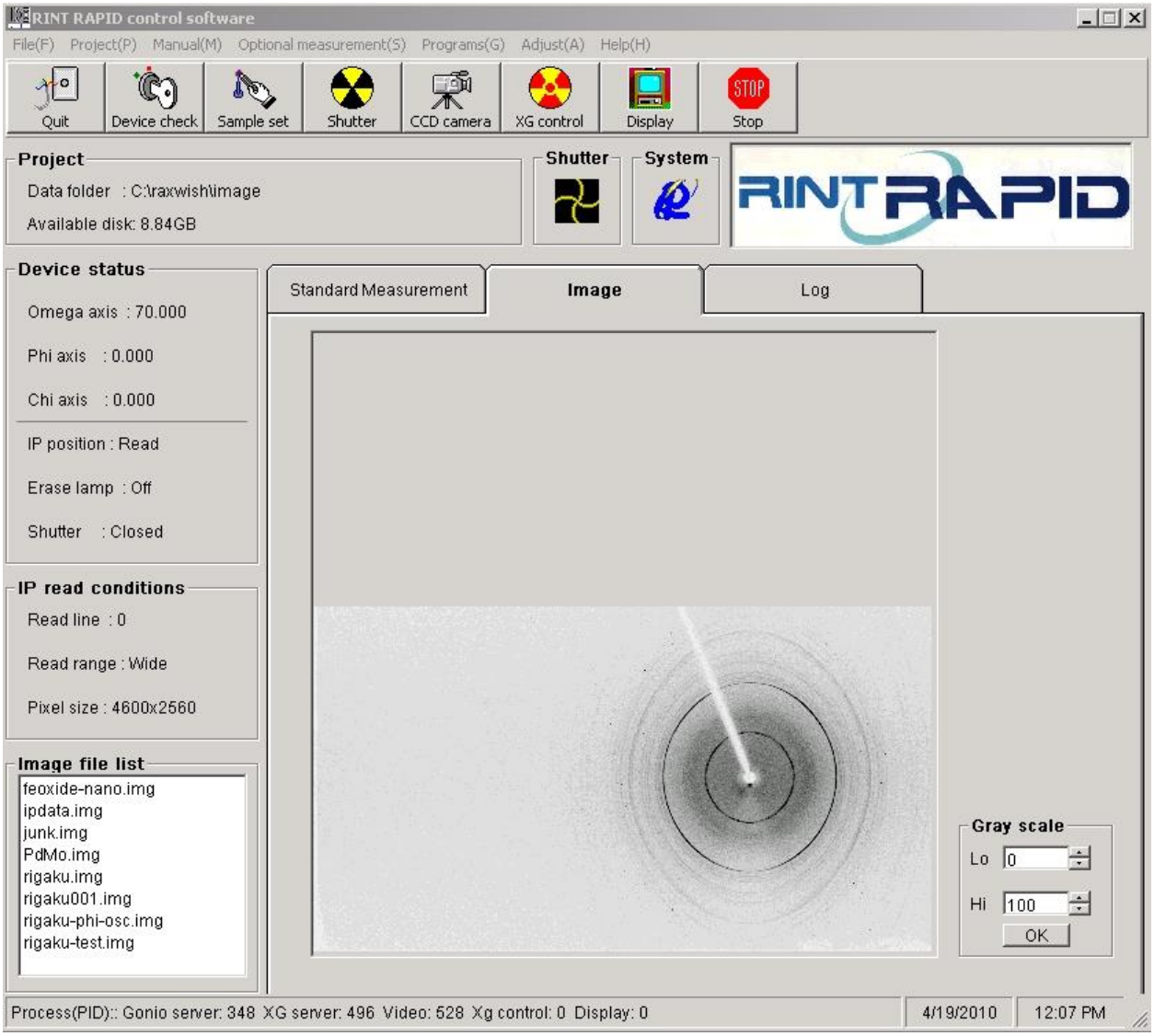
When you have aligned the sample, you can setup the data collection parameters. Typically, for a loose powder, you will leave omega at 70° and chi at 0° and you will rotate phi continuously. A good trial setup would be to collect data for 600 seconds while continuously rotating phi with the generator set to 40 KV and 40 mA.

For samples that may lose solvent during data collection, the powders can be loaded into a capillary and covered with the mother liquor. The powder will settle where it can be oriented on the diffractometer. The capillary can be held in place with a small amount of clay.


RAXVIDEO
File(F) Edit(E) View(W) Video(V) Color(C) Cross(S)

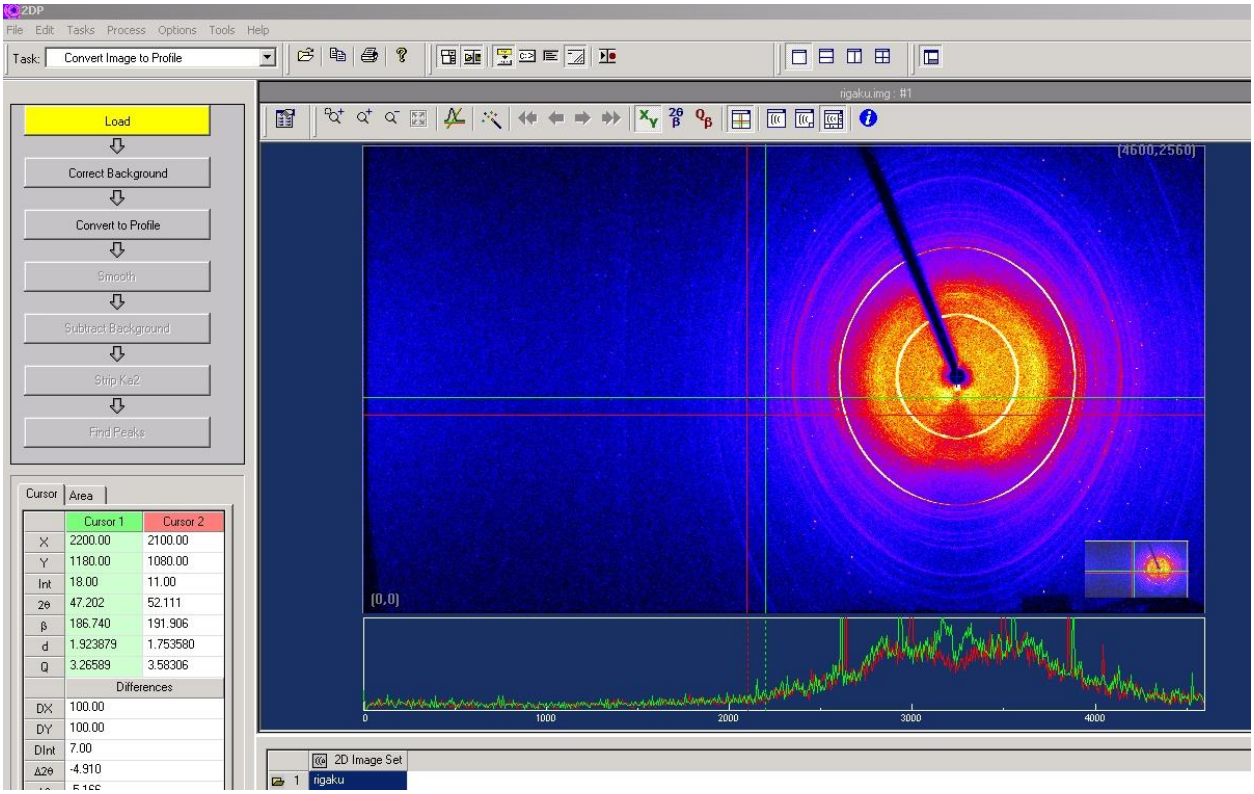


Hit Measure/Execute to start the data collection process. Be patient, the goniometer is slower than the others. In addition, the image plate detector may have to be erased before beginning. This process takes about 2 minutes as the detector is driven down to the read/erase heads in the bowels of the instrument. The yellow shutter open light will come on during data collection. When data is finished collecting, the image plate is driven to the read/erase heads and an image is created.

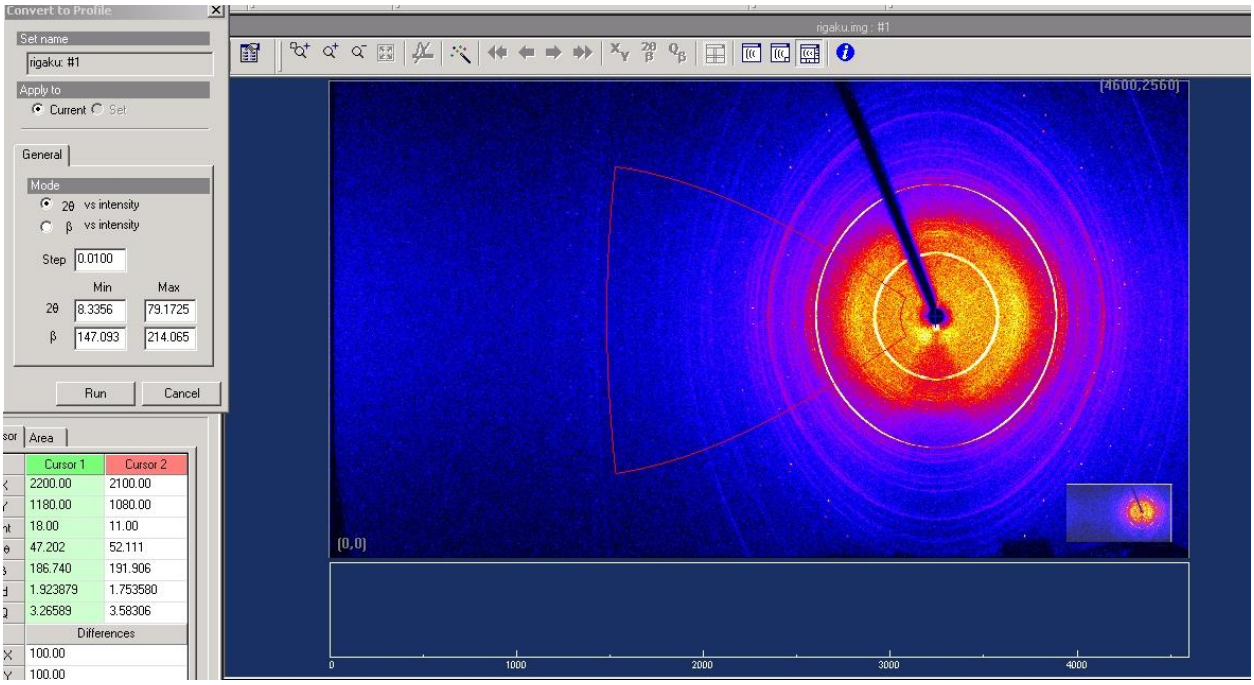


The 2D image can be converted into a 1D image suitable for data analysis in Jade or Bruker-AXS Eval using 2DP

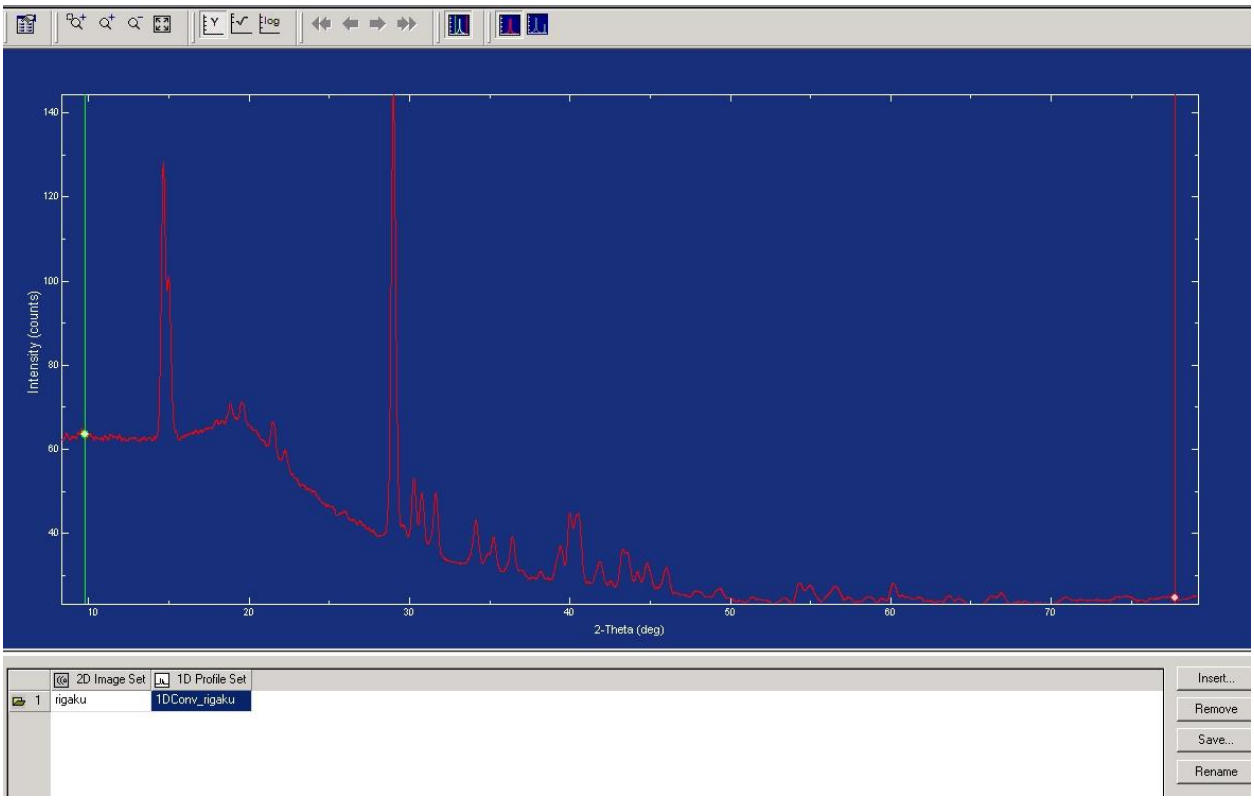
Load 2DP and select your image file for analysis. A 'feature' of 2DP is that if a binary file other than .img or .osc files are present in the working directory, 2DP will hang. There is no message output to explain why this is happening. The solution is to move the offending files to a different directory. So far, files of the type .raw (Bruker powder data files) and .jpg will cause 2DP to hang.



Select the convert to profile menu item to generate a 1-dimensional pattern from the 2-dimensional image collected.



Use the mouse to control the size of the area to be converted. You will have to experiment to see what size area gives the best results. If you exceed the acceptable limits, the Run button will be grayed out. When you are satisfied with the area, hit run. You will see something like this:

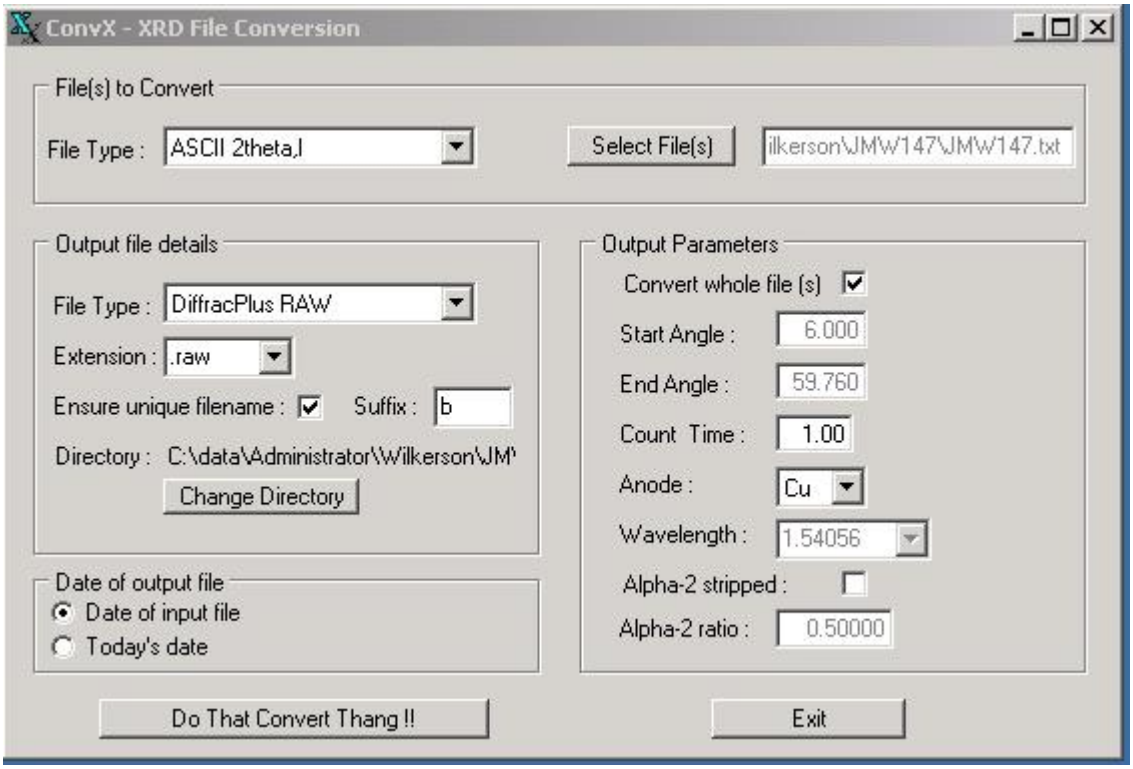


You can improve the 1-dimensional pattern by using a 'blank' for background correction. A blank is created by using an empty loop and running the same experimental conditions as the sample. You can correct background by using the 'Correct Background' feature listed under the Load instruction. You will be prompted for the name of the background file.

You can save this file for analysis in either Jade or Eval. If you wish to use Jade, save the file as type .asc. If you plan on using Eval, save as type .txt. You will have to use the free-ware program ConvX.exe,

to convert from the ascii format .txt to the Bruker Raw format.

The program menu for ConvX will appear as below.



The default file type for ASCII 2theta, I is .dat. You will have to select the appropriate .txt file. The output file type is DiffracPlus Raw. This is the Bruker format needed for Eval. Make sure the Anode type is set to Cu. The wavelength is set automatically. You can convert one or several files at once. The newly created .raw files will be stored in the same directory as the .txt files.

Note that there is an issue with 2DP when the directory containing the .img files also contains the Bruker .raw files that causes 2DP to hang. It is important to move the Bruker .raw files to a separate directory before using 2DP again.