

Panalytical MRD X-Ray Diffraction SOP

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1.0 Safety

- 1.1 **High Voltage** – This machine uses high voltages, especially to generate the X-ray beam. Do not touch any parts of the machine aside from those specifically mentioned in this SOP.
- 1.2 **X-rays** – X-rays are a very harmful form of radiation and care should be taken to minimize exposure. The radiation material **MUST** be reviewed prior to training or use of this machine. Additionally, all users **MUST** have obtained their X-ray badge prior to use or training on this machine. Your X-ray badge **MUST** be worn at all times during operation and **only those wearing X-ray badges should be permitted in the room.**
- 1.3 **Moving Components** – This machine has heavy moving parts. Although these parts only move inside the machine while the X-ray is on (and thus should be out of the users path, care should be taken to ensure you do not get in the way of a moving part. Do not attempt to open the doors while any parts inside are moving.

2.0 Training

2.0 Training for this machine is scheduled on demand. Please use the RSC website to request training if you would like to use the machine.

2.1 Prior to training, users **MUST** review safety information about X-rays and obtain an X-ray dosimetry badge. The training slides are available when you sign up for training and the dosimetry (X-ray) badge application is available in the Docs section of the RSC website.

3.0 Sample Preparation

3.1 Samples can be clamped or taped onto the sample stage. You may use a glass slide as a base if you prefer.

3.2 A glass slide will not introduce too much additional signal

3.3 Users are responsible for selecting masks and slits to ensure the X-Ray beam doesn't spill off their sample at low angles. Calculation references are found in the appendices of the training guide.

3.4 Samples may be loaded prior to TUMI login, **CHECK SHUTTER** prior to opening the instrument doors.

3.5 You will need to log into the TUMI system first if you need to adjust the Chi tilt to load your sample.

4.0 Pre-Operation

4.1 Ensure you have a reservation or that no one will have a reservation during your time.

4.2 Ensure that the system is at standby power by checking the voltage and current on the front of the tool

	Standby Power	Full Power
Voltage	45kV	45kV
Current	20mA	40mA

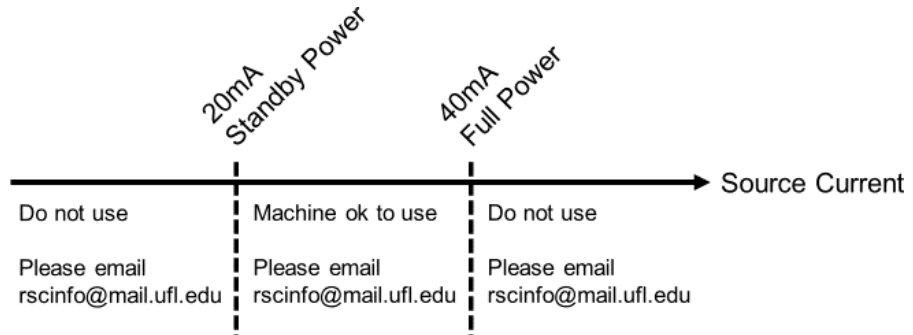
4.2.1 X-ray source voltage should be 45kV

4.2.1.1 **If the voltage is not 45kV**, do not use the tool. Please send an email to rscinfo@mail.ufl.edu

4.2.2 X-ray source current should be 20mA (standby current)

4.2.2.1 **If the current is higher than 40mA or lower than 20mA**, do not use the tool. Please send an email to rscinfo@mail.ufl.edu

4.2.2.2 **If the current is between 20mA and 40mA**, the machine can still be used, but please send an email to rscinfo@mail.ufl.edu with the time, date, and details



4.2.3 If you are using the machine IMMEDIATELY after another user, they may leave the system at full power for you. Otherwise, the machine must be brought to standby power after measurements are finished.



Indicator on the front of the machine showing the source voltage and current at standby power.

4.3 Log in to the TUMI system at the laptop computer

4.4 The Data Collector software should be left running. If it is not, follow these steps to get it running

4.5. Run the program “Data Collector”



Data Collector
Program

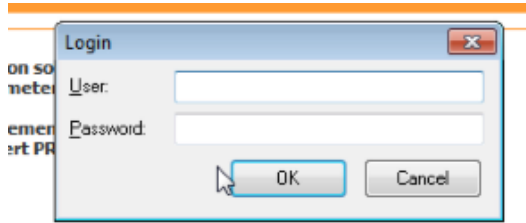
Data Viewer
Program

Data Collector and Viewer programs in the Windows bar.

4.5.2 Login with the username and password posted on the PC

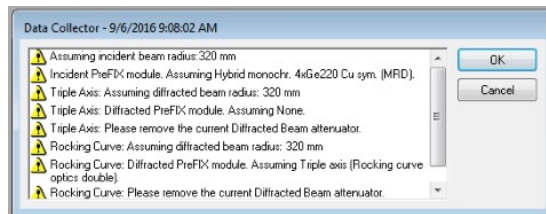
4.5.2.1 Username: class

4.5.2.2 Password: class1



4.5.3 Select Instrument -> Connect

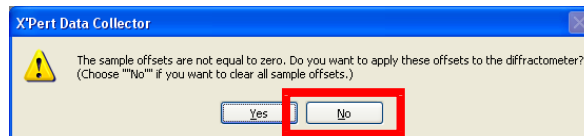
4.5.4 Select MRD Cradle (only configuration we have)



4.5.5 Select No to clear all coarse offsets

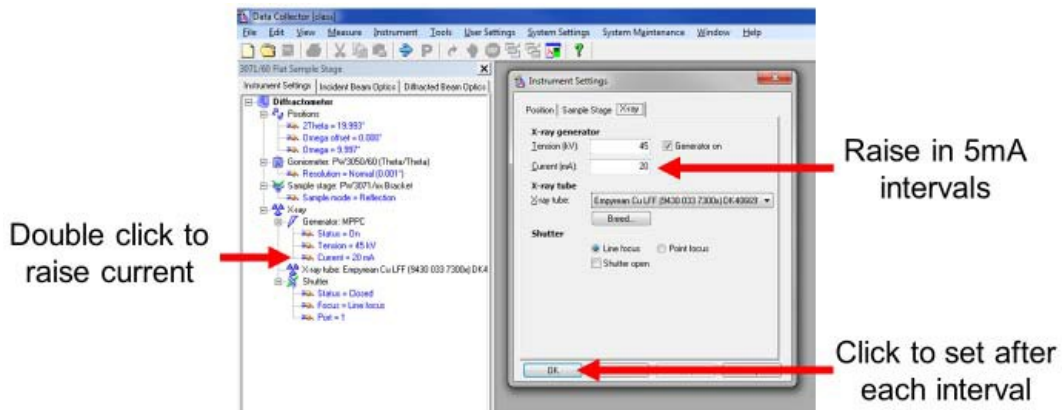
4.5.6 Select No to clear all fine offsets

****Failure to clear offsets could result in instrument collisions**



4.6 Once the software is running, raise the system power to full (40mA)

4.6.1 Double click the current in the left window 4.6.2 Increase the Current



by 5mA and select "Apply"

4.6.3 Double check on the front of the machine that the current was raised

4.6.4 Let the Machine saturate at this level for 30-45 seconds.

4.6.5 Repeat until you reach 40mA (full power) Select "OK" to close Instrument settings window.



4.6.6 Double check on the front of the machine that the current was raised to 40mA



The machine should now read full power.

5.0 Sample Height Adjustment

Clear all coarse and fine offsets in the User settings before performing any scans.

5.1 Sample height must be adjusted any time you place a new sample in the instrument. We use the direct beam method to ensure the sample is aligned with the X-Ray beam.

5.2 Insert the $1/32^\circ$ Slit into the Incident beam path and the Parallel Plate Collimator slit into the diffracted beam path (if using the PPC). Announce these changes in the data collector software.

5.3 We have a Batch Process (program) set up for automatic height adjustment, you may use the program designed for the current "default" optic configuration: Mirror + PPC.

5.4 If using the batch process, ensure all slits are in place, if prompted to add a slit, add it as indicated and select "OK". Please do not ignore prompts to insert slits, this may result in oversaturating the detector.

5.5 If you prefer to height align manually (OR if auto height align didn't work as expected):

5.6

5.6.1 Ensure automatic attenuator is set to ACTIVE anytime you are running manual scans, Automatic mode or always on both fine settings, but it must be ACTIVE.

5.6.2 Set X, Y, Z, Omega, 2Theta, Psi, Chi to 0

5.6.3 Measure -> Manual Scan

5.6.4 2Theta scan axis

5.6.5 Range ~0.4

5.6.6 Step size (range/100)

5.6.7 Scan time: low enough for a ~1min scan

5.6.8 On resulting graph, right click, peak mode, move to.

5.6.9 Save the offset as the new zero position. (User settings)

5.6.10 Move Z position to 5.5mm

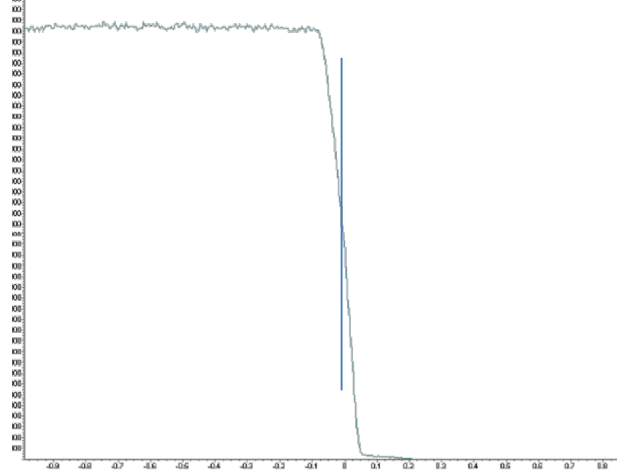
5.6.11 Manual Scan, Z axis scan

5.6.12 Range 10mm

5.6.13 Step size 0.01

5.6.14 Time per step can be low (2-5) aim for a 30 second to 1-minute scan time, or less.

5.6.15 In resulting graph, right click and select move mode. Drag line to the midpoint of the dropoff, this places the sample half way between out of the beam, and blocking the beam positions.



5.6.16 Measure -> Manual Scan

5.6.17 Omega Scan axis

5.6.18 Range can be ~0.4

5.6.19 Step size should be (Range/100)

5.6.20 Time per step can be small enough for a ~1 minute scan.

5.6.21 On resulting graph, right click, peak mode, select move to.

5.6.22 Repeat Z scan with smaller (~4) range

5.6.23 Repeat omega scan

5.6.24 Alternate Z and omega until change is insignificant (2-3x each)

5.6.25 Set the offsets in User Controls, select Fine Offsets, set offsets in user settings, fine offsets, "set new=0"

5.7 If running XRR scans in this session, execute the batch program for XRR alignment

- 5.7.1 This can also be done manually by:
- 5.7.2 Ensure automatic Attenuator is set to ACTIVE when running manual scans.
- 5.7.3 Manual scan
- 5.7.4 Omega axis, range 0.4, step size 0.004, time to make scan time ~1 minute
- 5.7.5 In graph, right click, peak mode, move to
- 5.7.6 Manual scan
- 5.7.7 Chi axis, range 4, step size 0.04, time to make scan time ~1min
- 5.7.8 In graph, right click, peak mode, move to
- 5.7.9 Repeat Omega and Chi scans 2-3x each until not changing significantly
- 5.7.10 Save the offsets in user settings.

6.0 Running an XRR Scan

- 6.0.1 You will need to insert the PPC Collimator slit from the diffracted beam path if it is not already there. Please start with the $1/32^\circ$ receiving slit in to ensure your lowest angles aren't oversaturating the detector. Announce changes to data collector.
- 6.0.2 You may use the example XRR program, however please don't modify anything other than the 4 scan parameter boxes.

Parameter	Value
Start angle (°)	19.993
End angle (°)	60.000
Step size (°)	0.0167113
Time per step (s)	10.160
Net time per step (s)	10.160
Scan speed (°/s)	0.208891
Pre-set counts (counts)	10000
Number of steps	2394
Total time (h:m:s)	00:03:26

Generally XRR scans have a start angle near zero (~ 0.1) and an end angle near 3-5 degrees. If only looking for critical angle, reduce scan to 0.1 to 2 for start and end angles.

- 6.0.3 You may use the example XRR program and change other settings (masks, slits, etc) but BEFORE making changes, File-> save as... and give it a new name.
- 6.0.3.1 **Please check the program settings to ensure the automatic attenuator is set to "at preset intensity"**
- 6.0.3.2 Close the program window and save changes.
- 6.0.4 Measure -> Program, select your XRR program. Save to your user folder, and make any needed notes in the spots available (these save to the report).
- 6.0.5 Right click on the axis of the plot, and change it to logarithmic scale to see the data as typically reported.
- 6.0.6 Use data viewer to convert data to XY format, or we have Reflectivity software designed to process these types of files. Reflectivity software requires some learning; it is also available for use on the JCPDS machine. You will need to tell the software what you know about your sample, and then help it model your curve.
- 6.0.7 Skip to 6.2

6.1 Running a GIXRD Measurement

6.1.1 You will need to remove the PPC Collimator slit from the diffracted beam path. It is recommended you use a slit and mask appropriate to the size of your sample. Announce these changes to data collector. Example programs are set to take any slit/mask combination announced in data collector.

6.1.2 You may use the example GIXRD program, however please don't modify anything other than the 4 scan parameter boxes.

The screenshot shows a software window with two tabs: 'Scan properties' and 'Repetition'. Under 'Scan properties', there are three radio buttons: 'Step', 'Continuous' (which is selected), and 'Pre-set counts'. Below these are several input fields for scan parameters. A red rectangular box highlights the following four fields: 'Start angle (°): 19.993', 'End angle (°): 60.000', 'Step size (°): 0.0167113', and 'Time per step (s): 10.160'. Other visible fields include 'Net time per step (s): 10.160', 'Scan speed (°/s): 0.208891', 'Pre-set counts (counts): 10000', 'Number of steps: 2394', and 'Total time (h:m:s): 00:03:26'. On the right side of the window, there are buttons for 'Description...', 'Comment...', and 'Settings...'.

The range should be similar to a powder scan, typically 20-100 is sufficient.

6.1.3 PANalytical indicated at their last visit, step sizes lower than 0.03 are overkill for GIXRD, and do nothing but increase scan time. Take that as you wish, this is by no means a rule, was their suggestion.

6.1.4 You may use the example GIXRD program and change other settings (masks, slits, etc) but BEFORE making changes, File-> save as... and give it a new name.

6.0.4.1 Please check the program settings to ensure the automatic attenuator is set to “at preset intensity” if using anything other than the example program.

6.0.4.2 Close the program window and save changes.

6.1.5 Before executing your GIXRD program, set omega in the instrument settings panel to desired incident angle. The example program is set to use the current setting as the incident angle. If you write your own program you can change this.

***You can use XRR to determine the critical angle (which is an ideal angle for GIXRD)**

6.1.6 Measure -> Program, select your GIXRD program. Save to your user folder, and make any needed notes in the spots available (these save to the report).

6.1.7 Use data viewer to convert data to XY format, or Highscore to process as you would a Theta-2Theta scan.

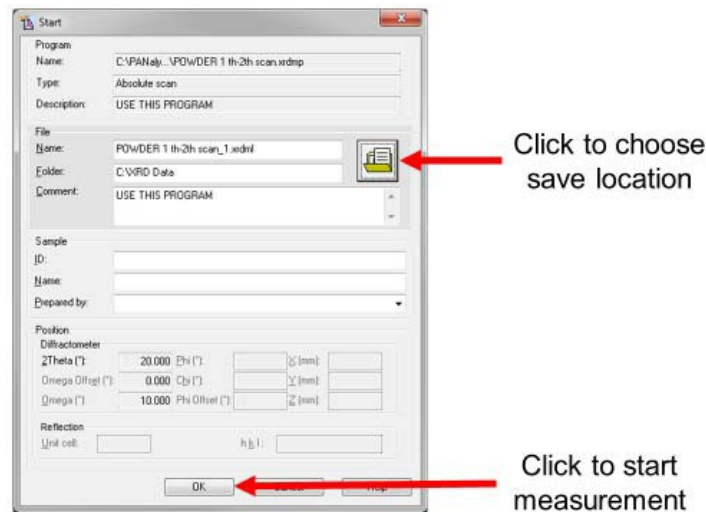
6.2 After you select to run a program:

This will open the measurement window

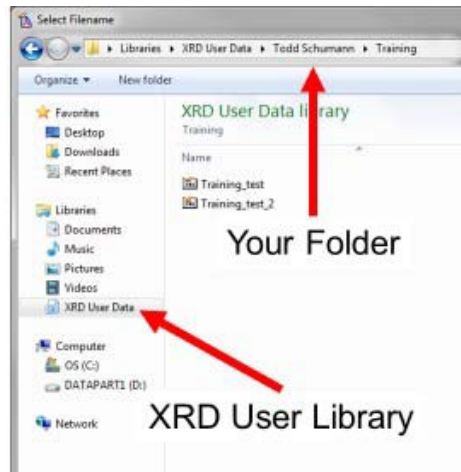
Select the large folder icon to choose where to save your scan data

Data should be stored in the XRD User Data library in a folder with your name

The folders and subfolders in your named directory can be whatever you like



Measurement window.



Store all measurements in your folder in the XRD User Data library.

Before beginning the run, ensure the machine doors are completely closed

Select OK to begin the scan

The tool will lock the doors. **DO NOT** attempt to open the doors.

The scan will run autonomously at this point. A graph will pop up and plot data as it comes in. There is no alert when the scan finishes. When all data is compiled, the machine will return the X-ray source back to the start angle and unlock the doors.

Once the doors are unlocked, you may proceed

Do not attempt to open the doors at any time while any pieces inside the machine are in motion. This will result in an error message and can damage the gears that move the parts.

7.0 Sample Unloading

- 7.1 It is recommended you set the Chi rotation to 90 before loading or unloading samples. You may leave the Chi setting at 90 upon logging out.
- 7.2 Once the doors are unlocked (there will be a loud click), you should be safe to open the door. However, ensure the X-ray is blocked with the indicator on the front of the machine.
- 7.3 If the X-ray is blocked (shutter is closed), open the doors slowly to retrieve your sample.
- 7.4 If you are running additional samples, you may load them at this point, close the doors, and run more measurements
- 7.5 If you are finished with all your scans, remove all samples and loading/buffer slides from the machine and close the machine doors

8.0 Post-Operation

- 8.1 When finished with the machine, you will need to return it to standby power
 - 8.1.1 If someone is going to use the machine **immediately** after you and you've arranged with them, you may leave the machine at full power
- 8.2 Double click the current in the left window
- 8.3 Decrease the current by 5mA and select "OK"
- 8.4 Repeat until you reach 20mA (standby power)
- 8.5 Ensure the 1/32° slit is inserted and announced in data collector
- 8.6 Ensure the PPC receiving slit is inserted in the diffracted beam path and announced to software.
- 8.7 Close all open windows in the Data Collector program, **BUT DO NOT CLOSE THE DATA COLLECTOR PROGRAM ITSELF**

9.0 Converting Data

9.1 The tool stores data in its own proprietary format, which will include the scan parameters, which can be opened in HighScore on the JCPDS computer. If you want an XY text file of data, use the following steps

9.2 In the Window's Start bar, select the Data Converter program



Data Collector and Viewer programs in the Windows bar.

9.3 In the window that opens, select File -> Open

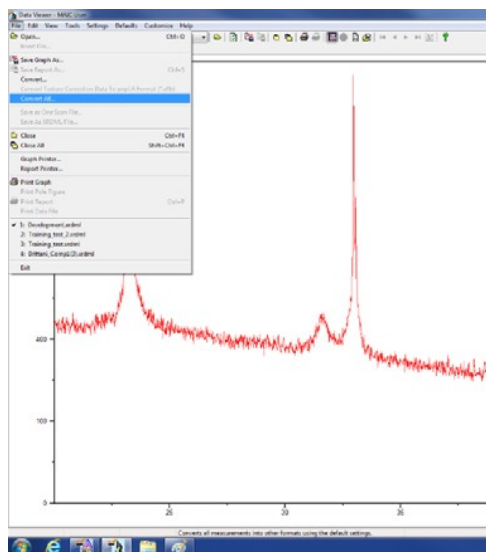
9.4 Navigate to Libraries -> XRD User Data -> [your name] and select all scans you'd like to convert (multiple scans may be selected)

9.4.1 Each scan will open its own window and an additional window will open with all scans plotted on top of each other

9.5 Select File -> Convert All

9.5.1 This process is quick, and the program does not acknowledge when it is finished

9.5.2 Your XY files will be in the same folder as the original scans



Data Collector and Viewer programs in the Windows bar.

9.6 Close the data viewer program

9.7 Transfer your data files to a flash drive, you may leave data on this machine, but it is periodically cleaned, and we are not responsible for lost data.

9.8 At this point, you can log off at the TUMI computer

10.0 Troubleshooting-

To stop a scan, use the stop sign in data collector.

Manual scans will not always close the shutter upon completion of the scan, you may need to press shutter close on the machine or in data collector before opening doors.

If you are not getting sufficient counts, you may need to increase the time per step or increase the slit size. You likely won't need to use anything larger than the $1/2^\circ$, remember larger slits means a larger X-Ray spot, you want to keep the beam size limited to the size of your sample.

You CAN run the GIXRD program with the $1/32^\circ$ divergence slit in place, but can achieve good results with lower count times if you use a larger slit. If you do not wish to change slits, you can always increase your count time.

If you are getting no GIXRD data, try realignment with manual technique.

It is better to error on the side of too many slits/masks, or too small slits/masks. It is easier to rescan a sample than to replace a burnt detector.

If you get a pop up telling you to add a slit, add it BEFORE selecting OK

If you need help, feel free to ask Kristy: **Office 203, or Ph. 352-273-2252.**

Each system has a "Useful Information" folder where I will store files that will help you determine slit sizes, settings for scans, software user guides, and other information XRD users will find helpful. Feel free to copy these folders for your reference later. In here are quick guides for the MRD techniques as I received from PANalytical, these do not replace the SOPs, but can be used as a supplement to the SOPs. These folders are updated on an as needed basis.

Additional MRD Techniques: Rocking Curves

Rocking curves are best collected using the hybrid and triple axis optics. You should request assistance to collect rocking curves if you have not received advanced training on the MRD. For rocking curves, the detector is placed on the front (nearest the door) position on the triple axis optic. **DO NOT TOUCH THE HOUSING FOR THE CRYSTAL ON THIS OPTIC.** A 1/2Q slit is recommended on the incident beam side, and masks if your sample is less than 25mm. A 1/2Q slit is recommended for the diffracted beam path as well. Announce all changes in data collector.

This technique describes rocking a sample on the omega axis to collect the rocking curve. Modify as needed :

- 1.0 There is no example program for rocking curves because they are sample dependent and require performing a manual scan. You need to manually center 2theta on a known peak, or select one in the Instrument settings to have the instrument move to the peak location for you.**
 - 1.1** Ensure your sample is appropriately height aligned and necessary offsets are applied and saved. (see Basic SOPs for this procedure).
 - 1.2** Double check the Attenuator is set to automatically activate at preset intensity (do not modify values) and ensure it is ACTIVE.
 - 1.3** Manually move to the 2theta position you are interested in observing... OR
 - 1.3.1 Under Instrument Settings, adjust the Unit Cell box to match your sample material.
 - 1.3.2 Enter the h k l peak you would like to observe (place a space between each value. Click Apply to move the detector to the correct position.
 - 1.4** Scan omega and move to maximum. (peak mode, move to)
 - 1.4.1 If the peak is very sharp, consider rescanning with smaller step size and range.
 - 1.5** Scan Chi and move to maximum. (peak mode, move to)
 - 1.6** Repeat the omega and chi scans until neither changes significantly
 - 1.7** Scan 2theta with a range of 1°, step size of 0.01, and 0.1 time per step.
 - 1.7.1 Only move 2theta position if necessary to center on the peak. The 2theta resolution is low in this configuration.
 - 1.7.2 Save sample offset omega=1/2(2theta), Chi=0
 - 1.8** Perform a manual omega scan with high resolution and small steps. This will be your rocking curve data.
 - 1.9** If the scan is sufficient for your needs **SAVE THE DATA.**
 - 1.9.1 Manual scans do not automatically save, you will need to click on the graph, then select file-> Save Asto save your rocking curve data.

Additional MRD Techniques: "Powder" Scans, Gonio (Theta-2Theta)

1.0 Running a Theta-2Theta Measurement- **This is what the Powder machine does, it is over 10x faster to run one there because of the detector type.*

- 1.1** You will need to have the fixed divergent slit optic installed on the incident beam side, and programmable receiving slit on the diffracted beam side. Run a sample height alignment **using the 1/32° slit and beam attenuator.**
 - 1.1.1** Remove attenuator after alignment, insert beta filter. Announce these changed in data collector incident beam settings.
- 1.2** *Select mask and slit combination necessary for your sample size and composition.* Request assistance if you need suggestions, or start small and use larger ones if necessary. Announce changes in data collector.
- 1.3** You may use the example Th-2Th program, however please don't modify anything other than the 4 scan parameter boxes: Start Angle, End angle, Step size, and Time per step.
- 1.4** You may use the example Th-2Th program and change other settings (masks, slits, etc) but **BEFORE** making changes, File-> save as... and give it a new name.
 - 1.4.1** Close the program window and save changes.
- 1.5** Measure -> Program, select your Th-2Th program. Save to your user folder, and make any needed notes in the spots available (these save to the report).
- 1.6** Use data viewer to convert data to XY format, or Highscore to process as desired.