XRD Training Notebook

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Before you begin…

- ❑ Complete the required safety training modules on UC Learning
  - ❑ Laboratory Safety Orientation (Fundamentals) 2013
  - ❑ Hazardous Waste Management
  - ❑ X-Ray Safety
  - ❑ Compressed Gas Safety
- ❑ Submit a copy of your Training Transcript to Lab Manager
- ❑ Review the MSE XRD Policies and Regulations
- ❑ Fill out the XRD FAU Authorization Form with PI signature
- ❑ Receive a user name and temporary password for Faces scheduling
- ❑ Arrange a time for XRD training with Lab Manager
- ❑ Schedule a 2 hour block on Faces for your training
- ❑ Receive a Data Collector password

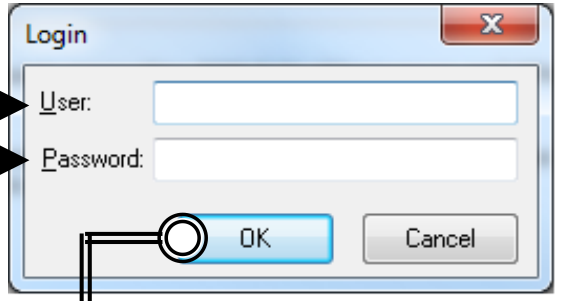
XRD Operation

I. Initiate Software
II. Sample Preparation
III. Membrane Holders
IV. Irregular Holders
V. Round Holders
VI. Sample Loading
VII. XRD Cabinet
VIII. X-Ray Settings
IX. New Measurement Program
X. Editing Measurement Program
XI. Start Measurement
XII. Data Viewing and Exporting
XIII. Data Analysis
XIV. Sample Unloading
XV. Cleanup
XVI. Troubleshoot

I. Initiate Software – 1/1

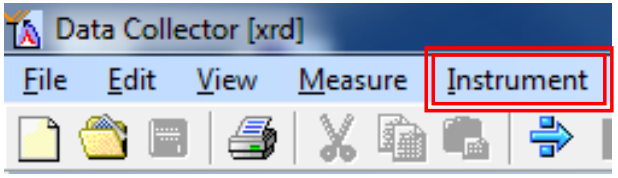
1. Record your time-in on the sign-in sheet

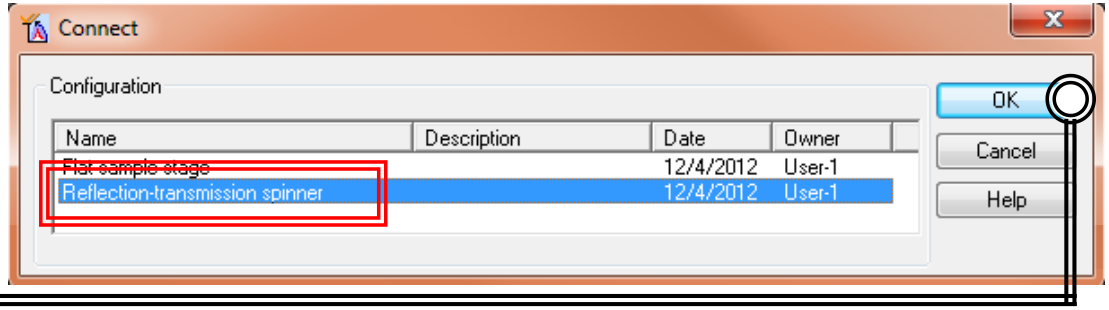
2. Double left-click on the ***Data Collector icon***



3. Enter User Login = <***Faces Username***> and Data Collector Password = <***Given by Lab Manager***> and click on ***OK***

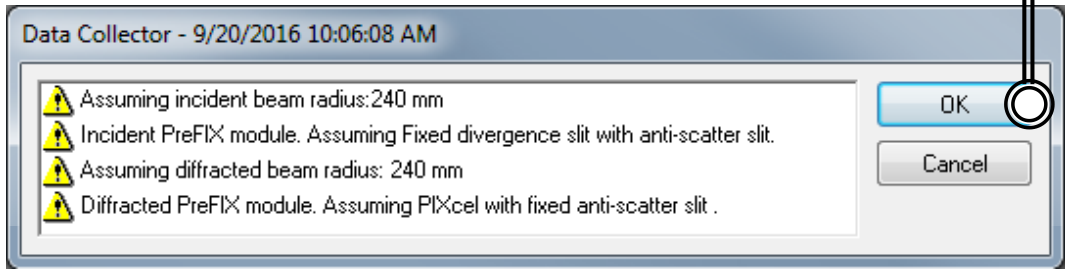
4. Select ***Instrument –> Connect***





5. Select ***Reflection-Transmission Spinner*** and click ***OK***

6. A dialogue box will appear, just click ***OK***



II. Sample Preparation – 1/2

1. The sample holder and preparation will vary depending on your sample
2. Three types of sample holders are available for use are located in the storage container
3. CLEAN UP AFTER EACH USE AND WIPE DOWN!

Membrane A

Membrane B

Membrane C

Irregular

Miscellaneous Tools

Tweezers
Scissors

Press

Round A

Round B

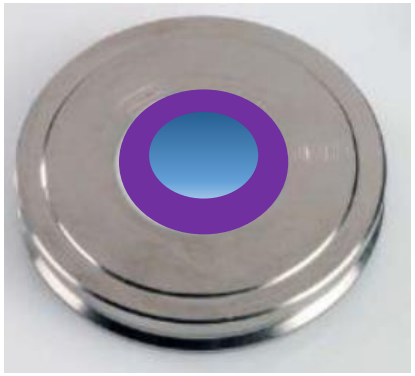
Round C

II. Sample Preparation – 2/2

4. It is important to always have the top of your sample at the SAME height as the top of your sample holder (no exception!)
5. If your sample is not at the same height as your sample holder, the peaks obtained will be incorrectly shifted away from correct positions!

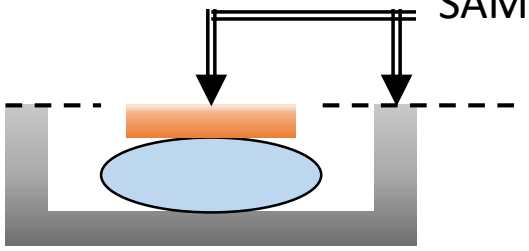
Irregular

Membrane

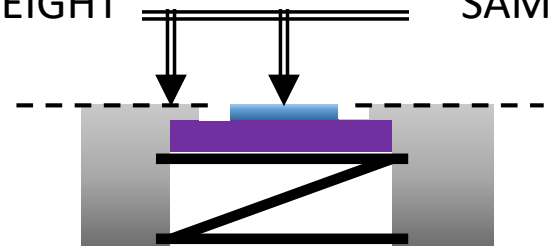


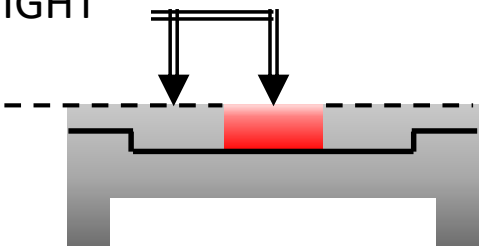
Round

SAME HEIGHT

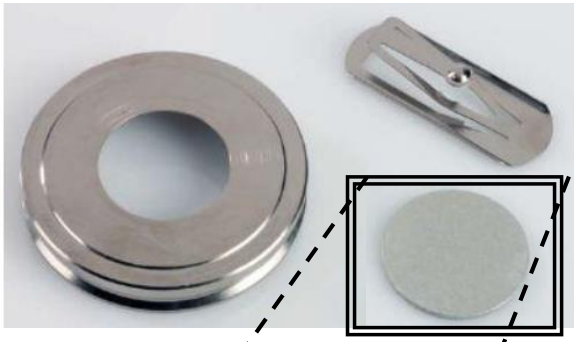


SAME HEIGHT





III. Membrane Holders – 1/1



1. This holder is designed for mounting:
   - dust filters
   - sample mounting plates
   - metal plates
   - pressed pellets
   - silicon substrates

2. Requires a support plate (Diameter = 32 mm)
   - Aluminum support provided (will have background Al peaks)
   - Recommend Si Zero Background Plate
     - These need to be provided by users

MTI Corp: Zero Diffraction Plate 32 mm Dia. x 2.0 mm t, Si Crystal for XRD sample ($150)

MTI Corp: Zero Diffraction Plate with Cavity for XRD sample: 32 Dia x 2.0 t mm with Cavity 10 ID x 0.2 mm, Si Crystal ($199)

Warning: X-ray beam shall be 5 mm dia or less (current installed beam mask is 10 mm) and hit in the center of sample when you use cavity zero diffraction plate! Otherwise the edge may result in a peak. USE AT YOUR OWN RISK!

IV. Irregular Holders – 1/1

1. This holder can be used to analyze solid samples with:
   - Maximum diameter = 45 mm
   - Maximum thickness = 6.5 mm

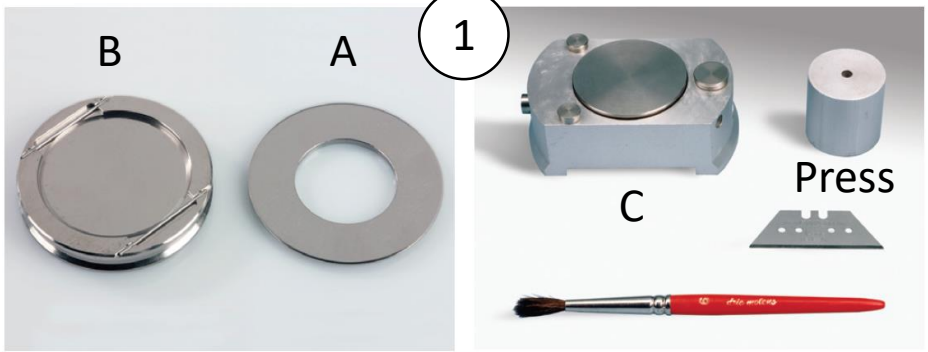
2. The sample can be mounted with clay available from Storehouse:
   - Storehouse Description: SARGENT ART 22-4096 1LB MODELING CLAY, WHITE (Stock #: 48702-108)
3. Recommend using a glass slide or Si zero background plate as support for your sample on top of clay

MTI Corp: Zero Diffraction Plate 32 mm Dia. x 2.0 mm t, Si Crystal for XRD sample ($150)

MTI Corp: Zero Diffraction Plate with Cavity for XRD sample: 32 Dia x 2.0 t mm with Cavity 10 ID x 0.2 mm, Si Crystal ($199)

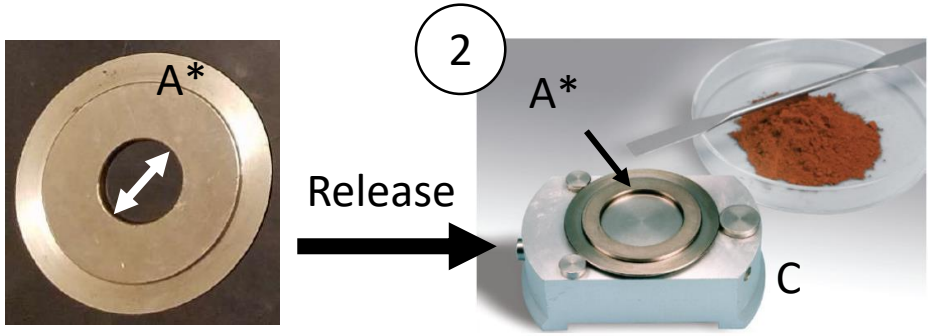
Warning: X-ray beam shall be 5 mm dia or less (current installed beam mask is 10 mm) and hit in the center of sample when you use cavity zero diffraction plate! Otherwise the edge may result in a peak. USE AT YOUR OWN RISK!

V. Round Holders – 1/2



1. Assemble the items for powder samples (user provides razor and brush)

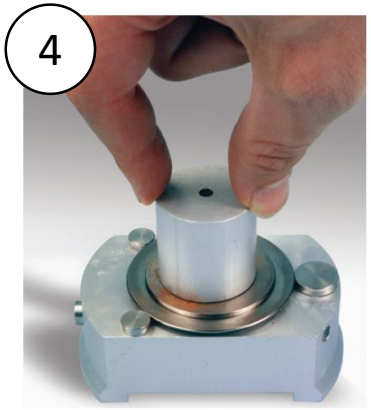
REMEMBER TO CLEAN ALL SURFACES FIRST BEFORE USING!



2. Invert A to get A*. Place on top of C and push the release to have it sit into place.

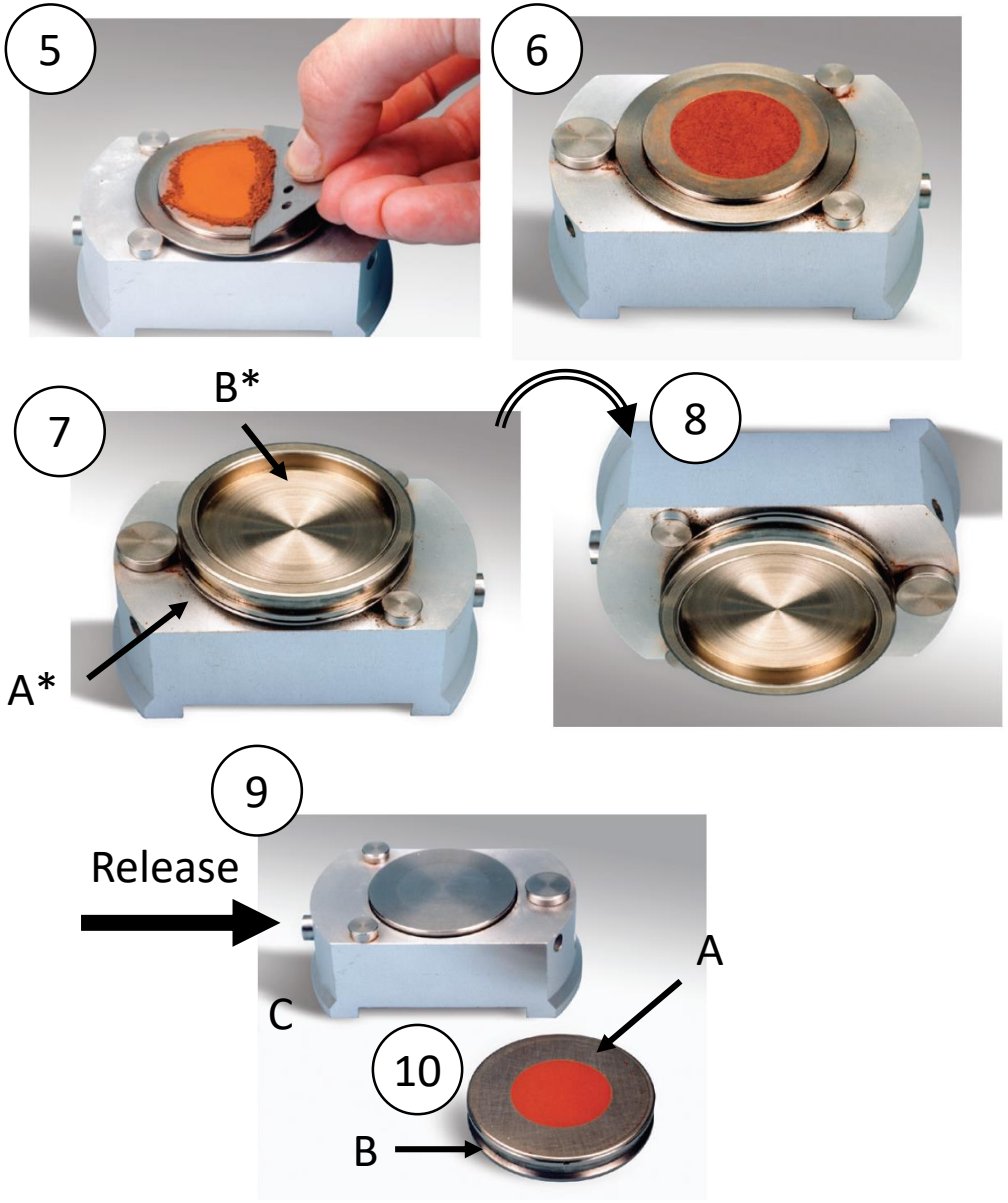
Diameter = 16 mm

3. Spread the powder into the cavity using a spatula but do not pack or compress.



4. Press powder with Aluminum press

V. Round Holders – 2/2



5. Remove excess powder with a straight edge or side of microscope slide

   DO NOT SCRATCH TOP SURFACE!

6. Clean mating surfaces with small brush or provided kim wipe

7. Invert B to get B* and snap on top of A*

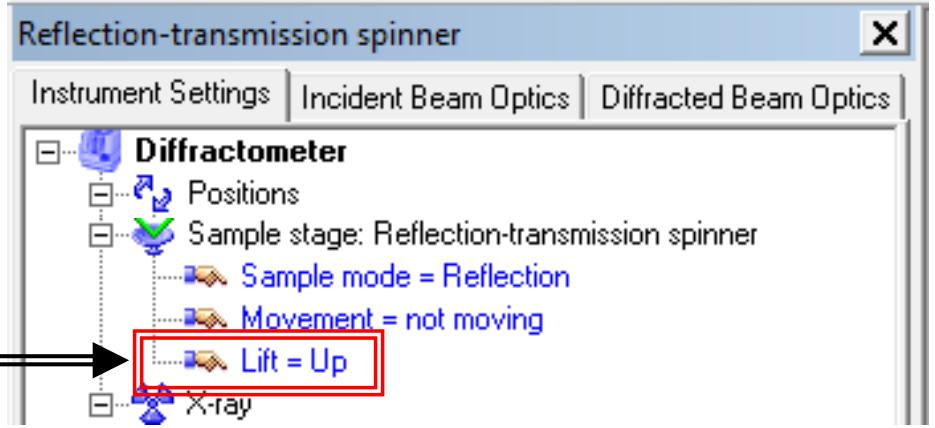
8. Flip entire assembly

9. Push the release to remove the sample holder (A + B) from C

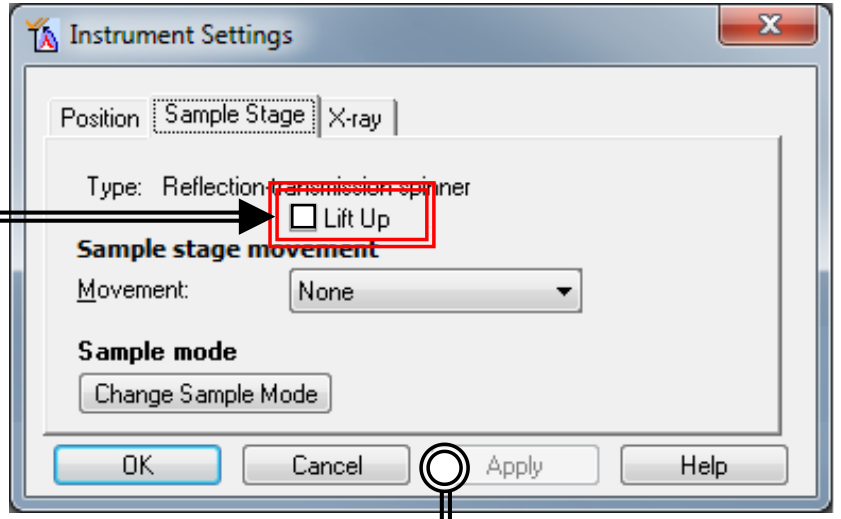
10. The surface of your sample should be smooth via back-filling approach

VI. Sample Loading – 1/4

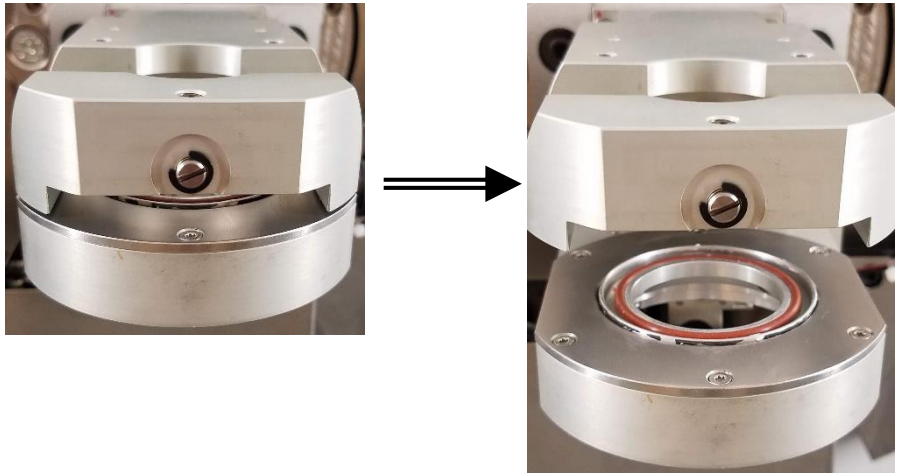
1. Double-click on "***Lift = Up***"



2. ***Uncheck*** the "***Lift Up***" option and click ***Apply***



3. The stage will now drop down

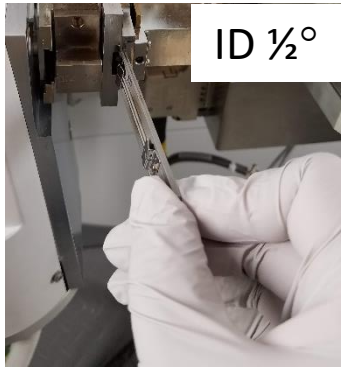


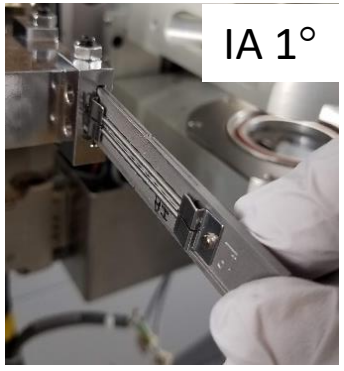
VI. Sample Loading – 2/4

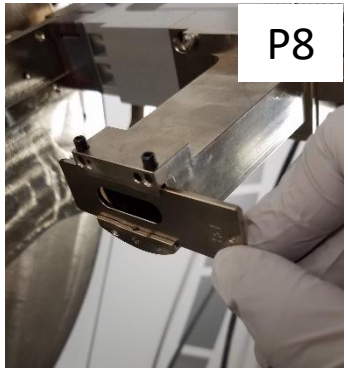
4. Press "***UNLOCK DOORS***" on cabinet

5. Open doors by pulling on the handles at the ends for better leverage

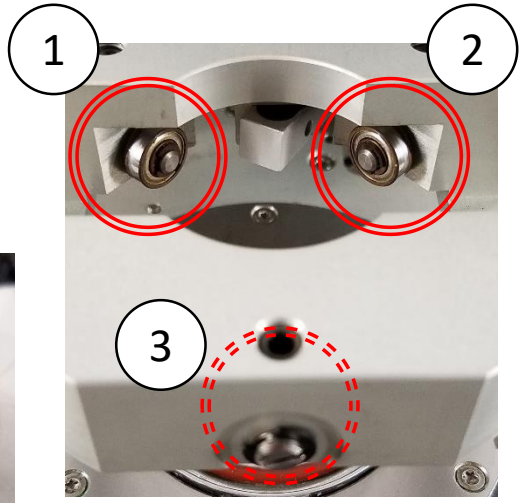
6. Inspect and check if desired slits are installed
   - ***Standard Slits*** are default:
     - **I**nc **D**iv ½°
     - **I**nc **A**nt 1°
     - Dif Ant **P8**





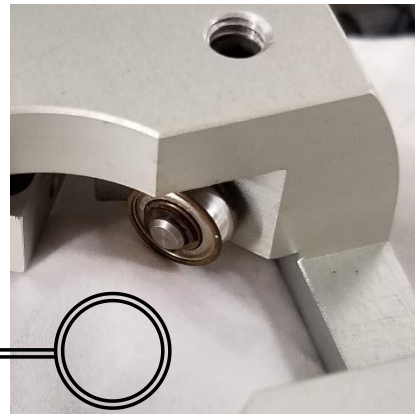


VI. Sample Loading – 3/4



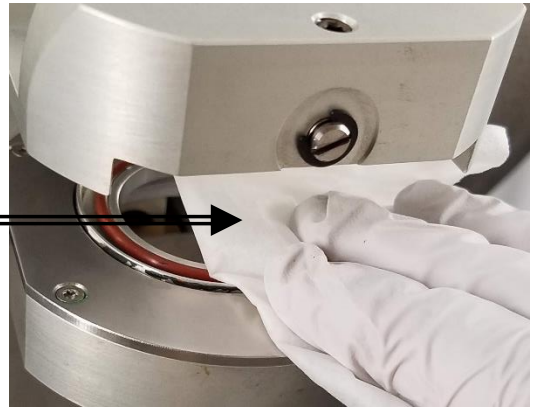
7. Inspect ***Stage*** for any residual sample left stuck on **3** ***Spinner Bearings*** from previous user

8. Take ***Kimwipe*** with ***IPA*** and carefully wipe all ***3 Spinner Bearings***



9. Use fresh area on ***Kimwipe*** to remove residual sample

10. Fold, and use fresh area of ***Kimwipe*** to wipe down the base of the ***Stage***

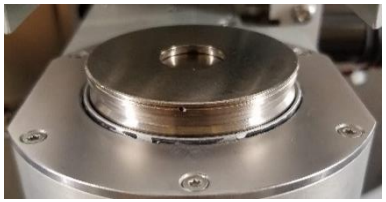


11. If necessary, use provided ***Air Duster*** to dry and remove any remaining dust on ***Stage***

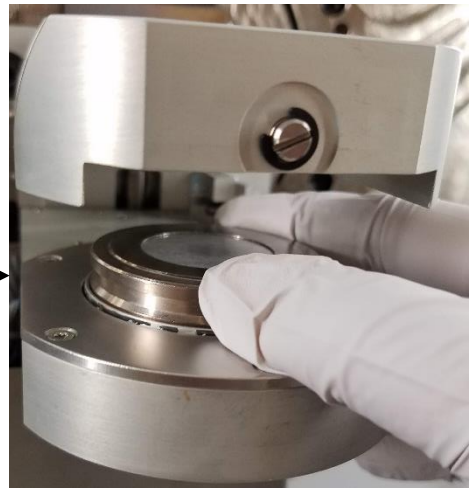
VI. Sample Loading – 4/4

12. Carefully insert ***Sample Holder*** into ***Stage***



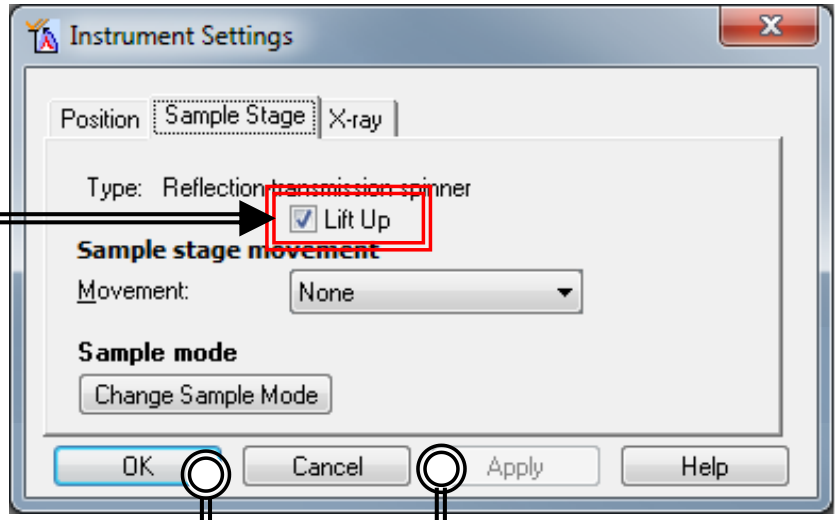


13. Confirm ***Sample Holder*** is properly seated into ***Stage***



14. Close doors of cabinet

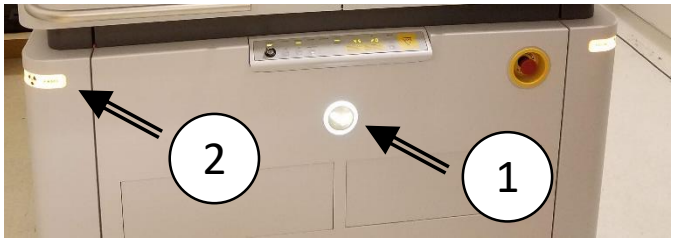
15. Check the "***Lift Up***" option and click ***Apply***

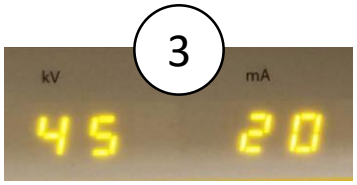


16. Click ***OK***

VII. XRD Cabinet – 1/5

1. Always remember to check 3 indicators that XRD is ***OK***
   - ***White Power Light*** is ***On***
   - ***X-Rays On Light*** is ***On***
   - ***X-Ray*** settings are ***45 kV*** and ***20 mA***





Note: If above 3 indicators are missing, contact Lab Manager

X-Rays On Indicator

Safety Key: ***ON*** Position

Power Off Button

Power On Button

Unlock Doors Button

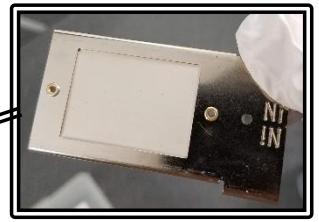
X-Rays Setting

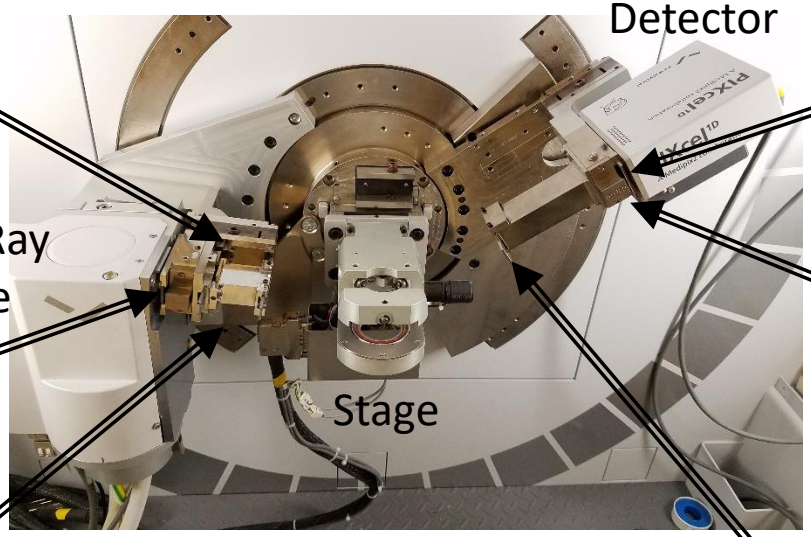
Lights On Button

VII. XRD Cabinet – 2/5

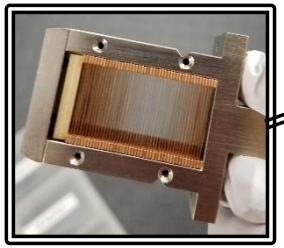
10 mm Beam Mask

Ni Beta Filter

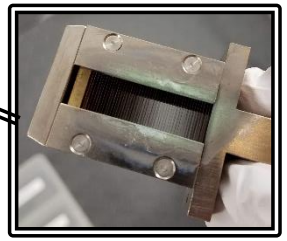


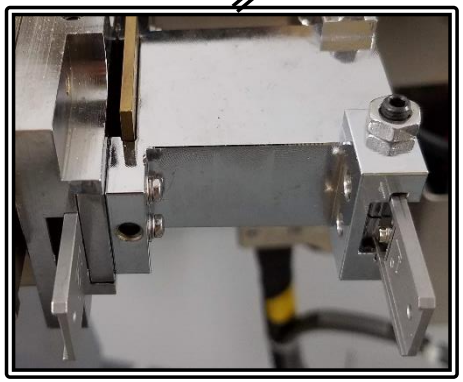


Soller Slit



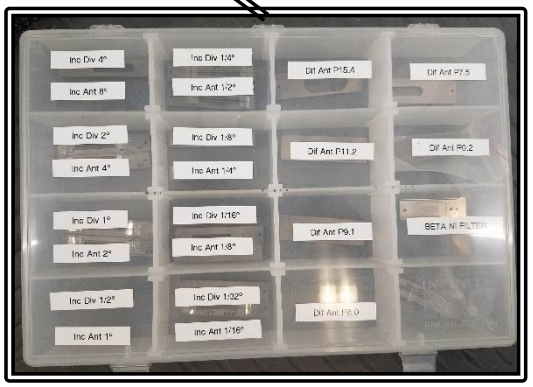
Soller Slit



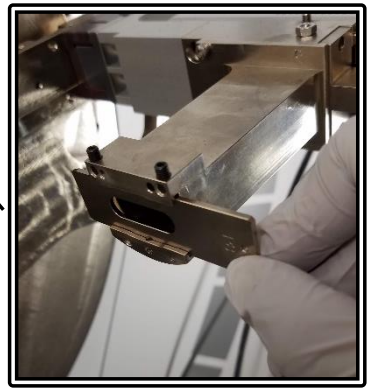


Incident Divergent Slit

Incident Anti-scatter Slit



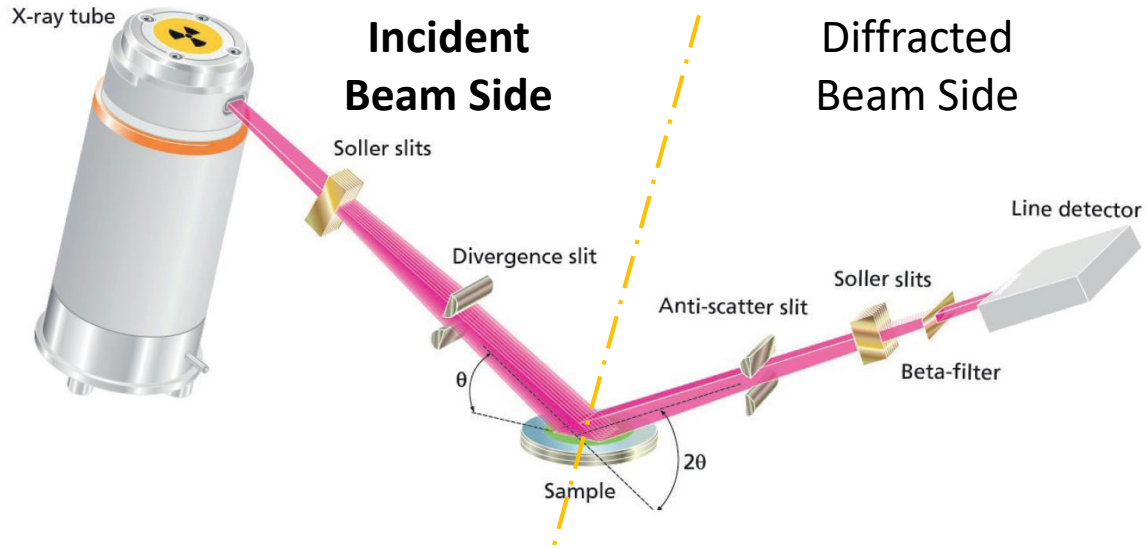
Slits Storage Box



Diffracted Anti-scatter Slit

VII. XRD Cabinet – 3/5

The following table describes the components for the **Incident Beam Side**

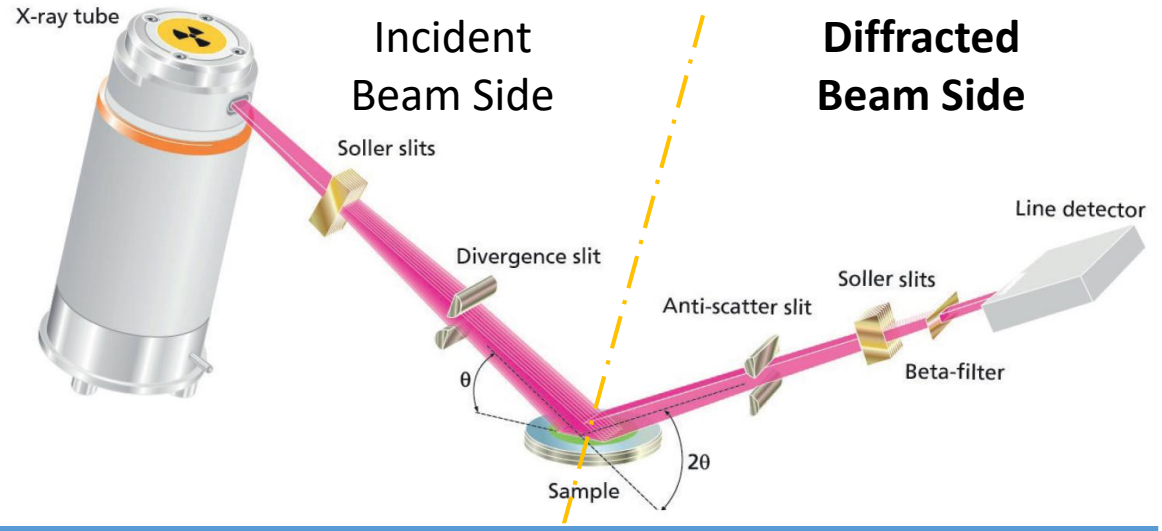


Incident Beam Side

Component Name	Function or Description
Soller Slit	Prevents axial divergence and improves peak shape and symmetry
Divergence Slit	Controls the irradiated length of the X-Ray beam on the sample. Slit size depends on sample size and starting scan angle.
Incident Anti-scatter Slit	Reduces X-Ray beam scatter and reduces background. Typically double the selection of the divergent slit.
Beam Mask (not pictured)	Controls axial width of the X-Ray beam. Match to sample size.

VII. XRD Cabinet – 4/5

The following table describes the components for the **Diffracted Beam Side**



Diffracted Beam Side

Component Name	Function or Description
Receiving Slit	Controls the resolution of the instrument, common setting is 0.1 mm.
Soller Slit	Match with incident selection, typically 0.04 radians.
Diffracted Anti-scatter Slit	Match to the selection of the Divergent Slit.
Beta-filter	Used to remove beta radiation.
Detector	PIXcel 1D

VII. XRD Cabinet – 5/5

Standard Slit Configuration = 1/2°, 1°, 8 mm

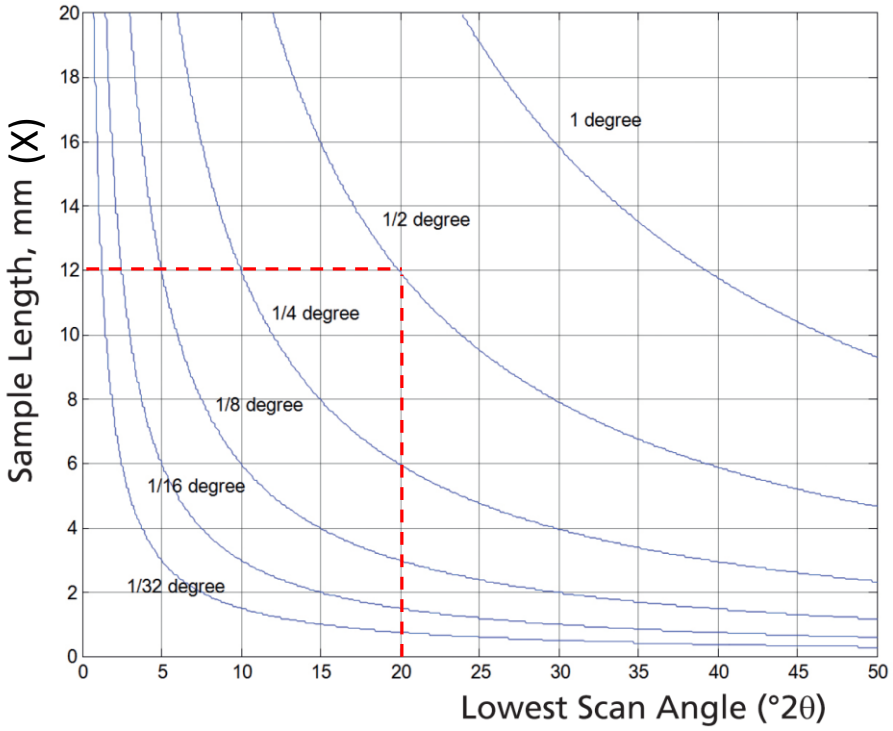
Slit Configurations		
Incident Beam Side		**D**iffracted Beam Side
Incident **D**ivergence	**I**ncident **A**nti-scatter	**D**iffracted **A**nti-scatter
ID 4°	IA 8°	P15.4
ID 2°	IA 4°	P11.2
ID 1°	IA 2°	P9.1
ID **1/2°**	IA **1°**	**P8.0**
ID 1/4°	IA 1/2°	P7.5
ID 1/8°	IA 1/4°	P7.5

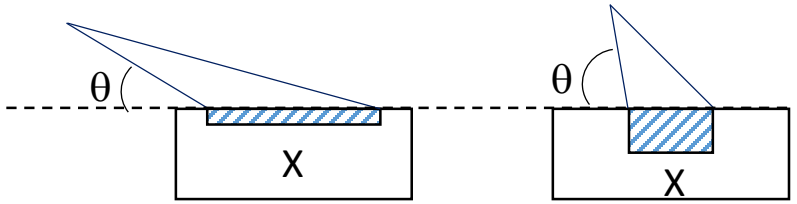
Decreasing Slit Size

Choose smaller slit sizes for removing background intensity at low angles

Effects of Low Scan Angles:
Irradiated Sample Length X vs. °***2θ***

(Ex: °2θ = 20° with ID ½° => X = 12 mm)





VIII. X-Ray Settings – 1/1

1. Double-click on the "***Current = 20 mA***"

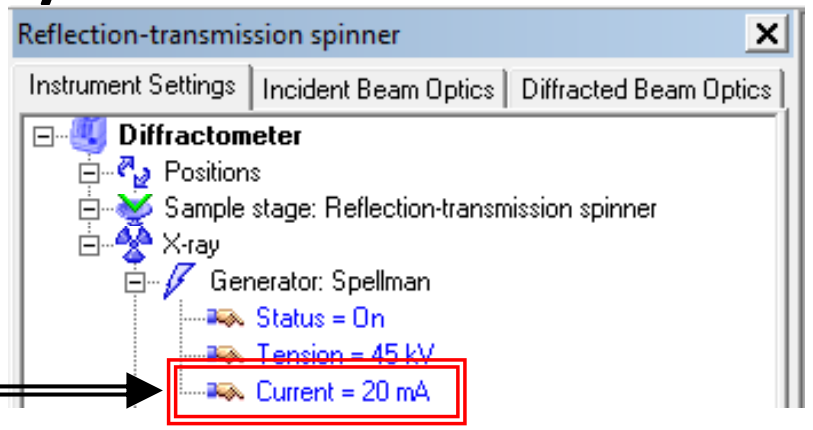
2. Tension should be kept at ***45 kV***

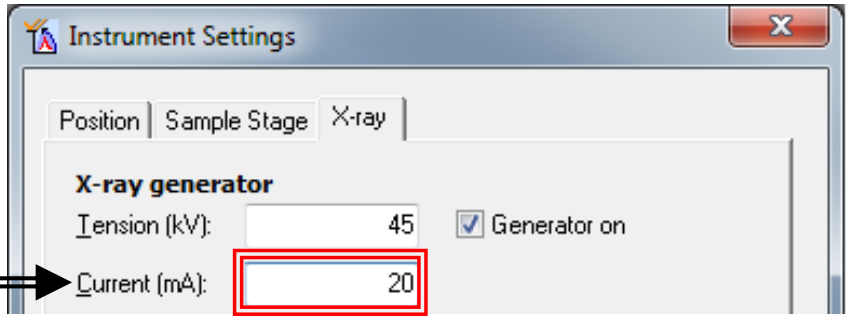
   Current = 20 mA when not in use

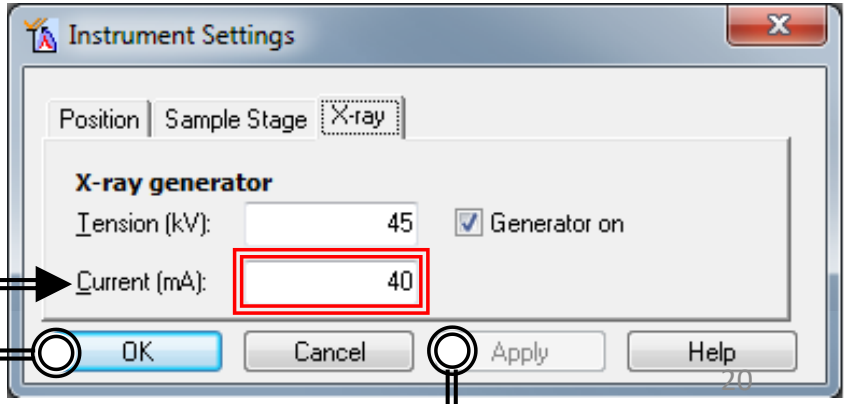
   Current = 40 mA for experiments

3. Change current to ***40 mA*** for actual experiments

4. Click ***Apply***, then ***OK***



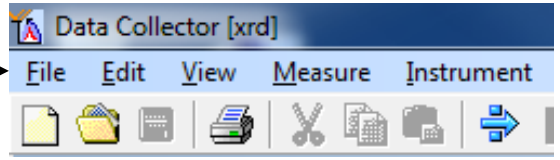




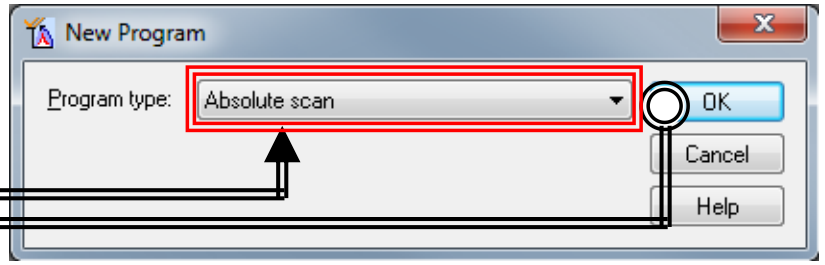
IX. New Measurement Program – 1/7

Note: **SKIP** to ***X. Editing Measurement Program*** if you already have a program

1. Click ***File*** and choose ***New Program*** to create a new program (required for training)

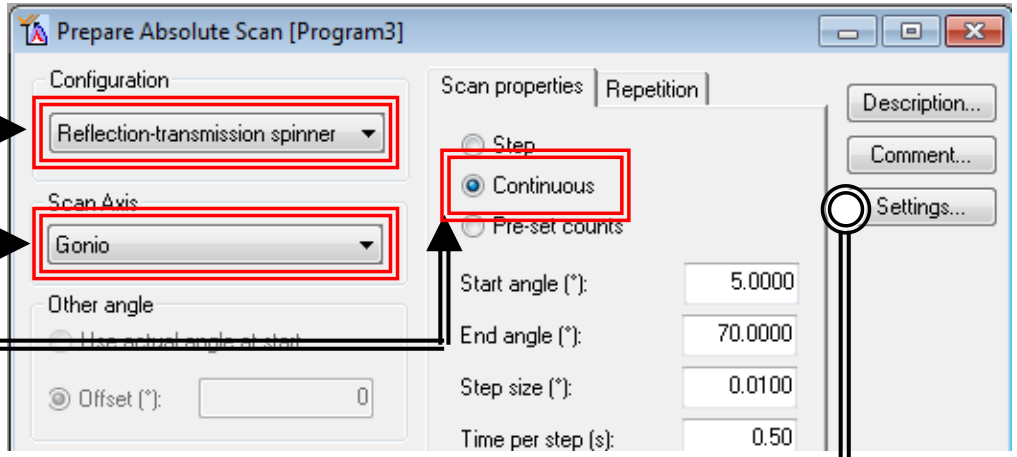


2. Choose ***Absolute Scan***, click ***OK***



3. Confirm ***Reflection-transmission spinner*** and ***Gonio*** are selected

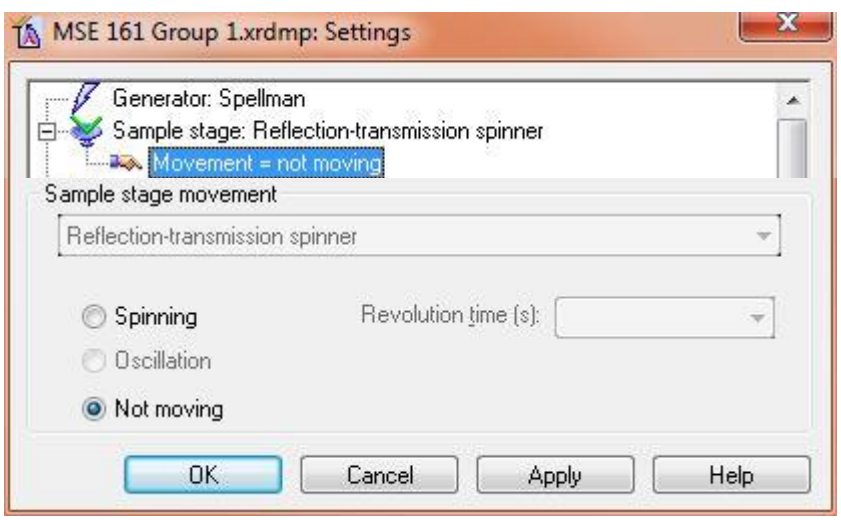
4. Choose ***Continuous***



5. Click ***Settings***

IX. New Measurement Program – 2/7

6. Click ***Movement***
   - Set to ***Spinning Enabled*** (recommended)
     - Set ***Revolution Time = 2 seconds***
   - Set to ***Not moving*** if homogeneity is not an issue but sample ejection is

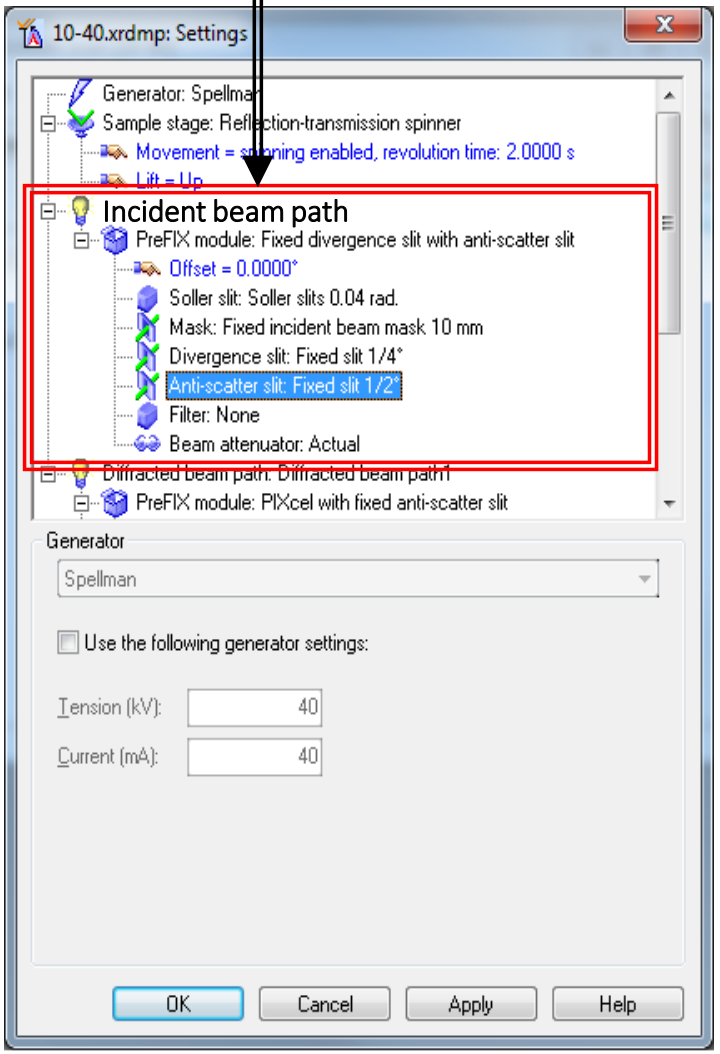


7. The default settings show "***Actual***" (meaningless) for all entries

IX. New Measurement Program – 3/7

8. Set the following ***Incident beam path*** entries as follows:

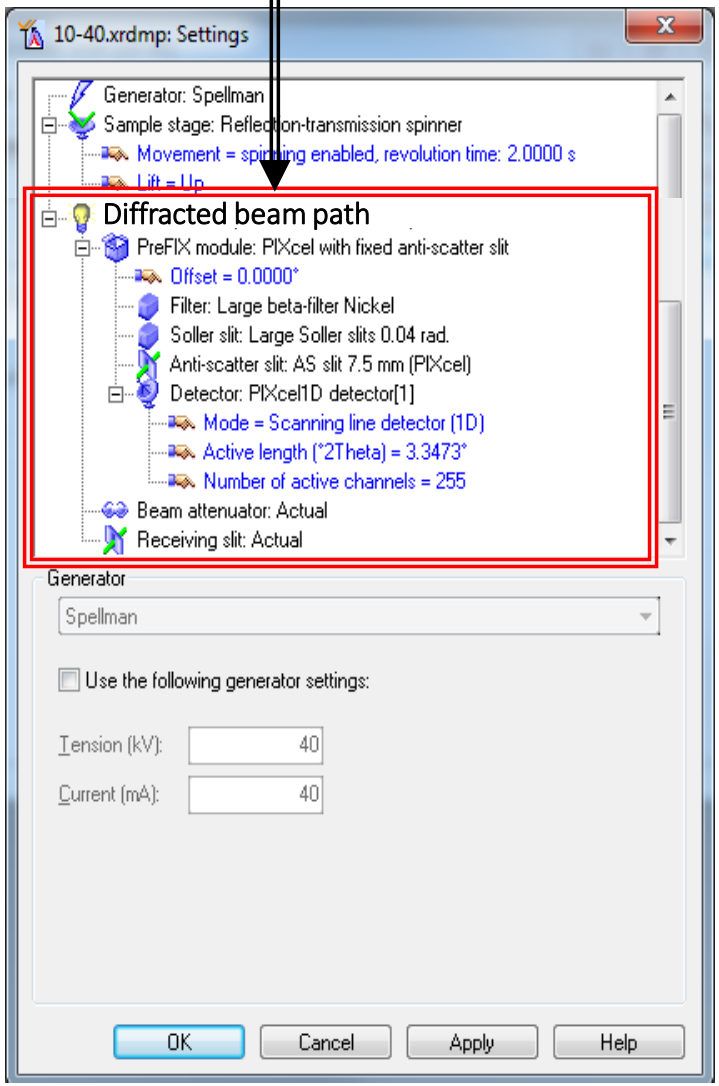
- ❑ PreFIX module: ***Fixed divergence slit with anti-scatter slit***
- ❑ Soller slit: ***Soller slits 0.04 rad***
- ❑ Mask: ***Fixed incident beam mask 10 mm***
- ❑ Filter: ***None***
- ❑ Beam attenuator: ***None***
- ❑ Divergence slit: ***<Enter what you're using>***;
  if ***Standard Slits*** then ***Fixed slit 1/2 °***
- ❑ Anti-scatter slit: ***<Enter what you're using>***;
  if ***Standard Slits*** then ***Fixed slit 1 °***



IX. New Measurement Program – 4/7

9. Repeat for the ***Diffracted beam path*** entries as follows:

- ❑ PreFIX module: ***PIXcel with fixed anti-scatter slit***
- ❑ Filter: ***Large beta-filter Nickel***
- ❑ Soller slit: ***Large soller slits 0.04 rad***
- ❑ Detector: ***PIXcel1D detector[1]***
- ❑ Beam attenuator: ***None***
- ❑ Receiving slit: ***None***
- ❑ Anti-scatter slit: ***<Enter what you're using>***;
  if ***Standard Slits*** then ***AS slit 8.0mm (PIXcel)***



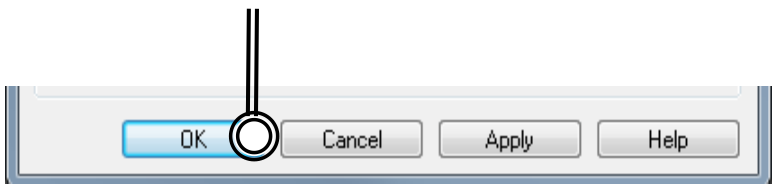
IX. New Measurement Program – 5/7

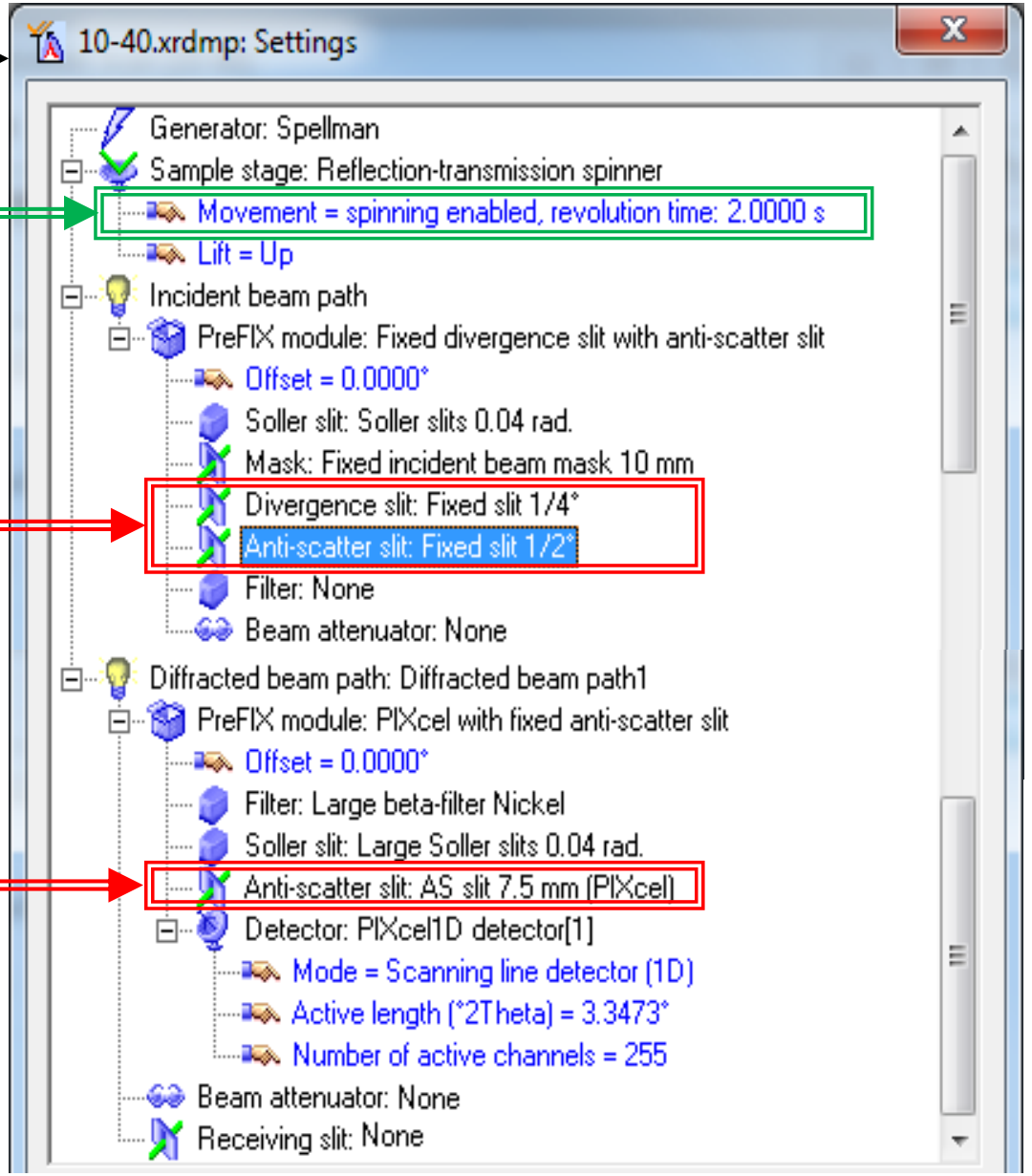
10. Confirm the settings

YOU CONTROL STAGE MOVEMENT CHOICE

VALUES WILL CHANGE DEPENDING ON ACTUAL SLITS USED

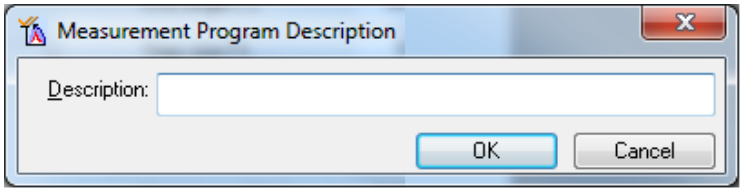
11. Click ***OK***

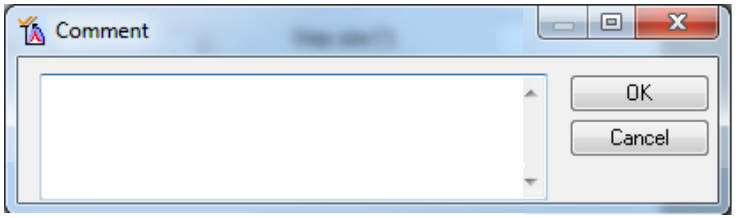




IX. New Measurement Program – 6/7

12. Click ***Description*** and ***Comment*** tabs to enter information if desired





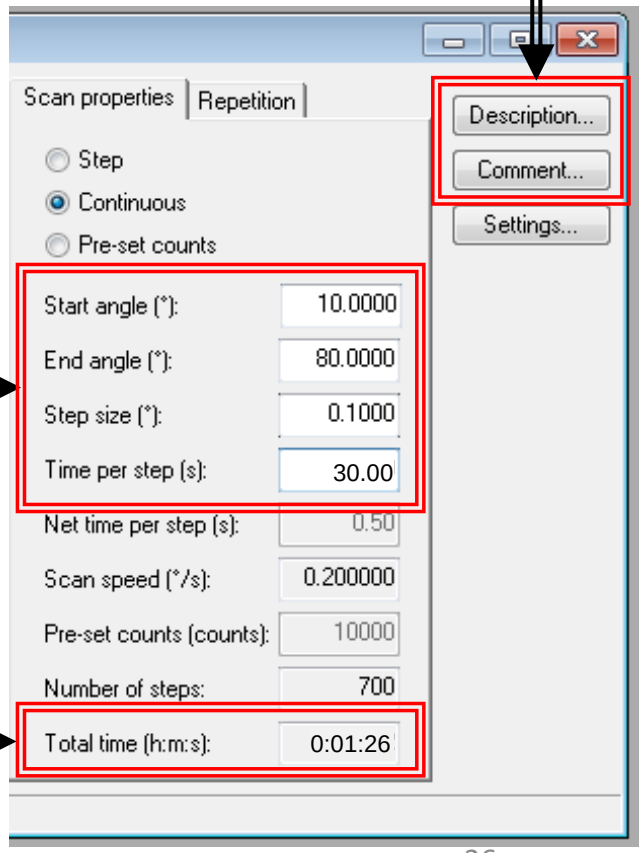
13. Set ***Start Angle***, (eg. **10°**)

14. Set ***End Angle***, (eg. **80°**)

15. Set ***Step Size***, (eg. **0.1°**)
    - ***decrease*** to enhance ***resolution***

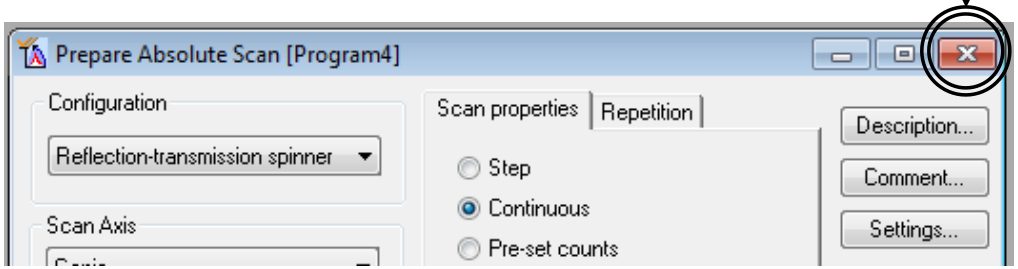
16. Set ***Time Per Step***, (eg. **30 sec**)
    - ***increase*** to enhance ***signal/noise***

17. The ***Total Time*** (**h:m:s**) for the scan will automatically update



IX. New Measurement Program – 7/7

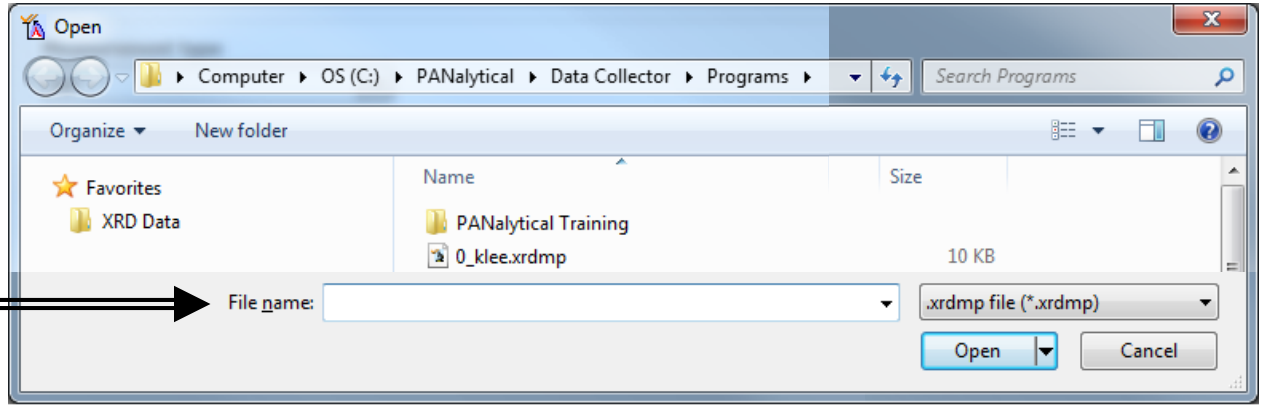
18. Click the ***Close X*** to close the window



19. Choose to ***SAVE*** your program

20. Select your <***PI'S NAME***> folder

21. Name your ***Measurement Program*** file



22. Default unsorted folder is "**C:\PANalytical\Data Collector\Programs**"

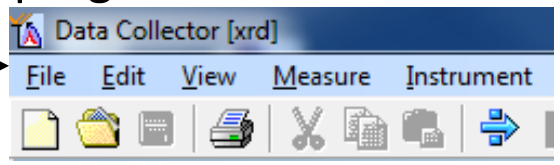
23. Continue to ***XI. Start Measurement*** and SKIP ***X. Edit Measurement Program***

X. Editing Measurement Program – 1/1

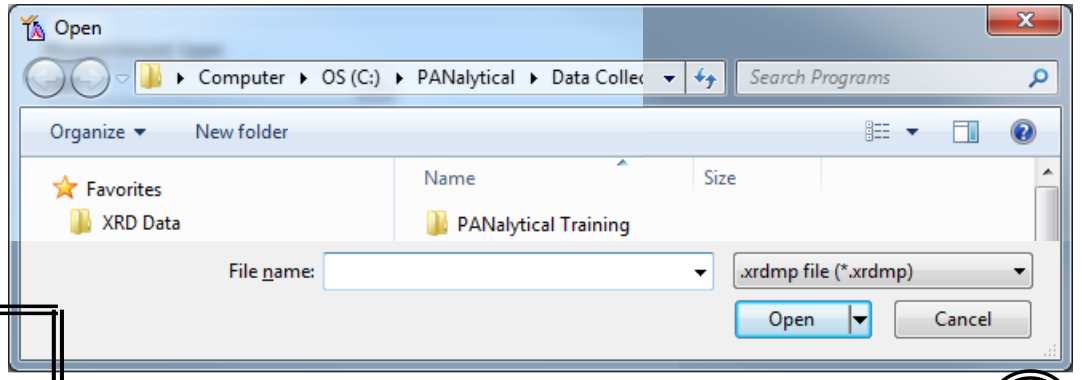
The following steps are for **EDITING** existing program you already created only!

SKIP to ***XI. Start Measurement*** if you don't need to edit your program

1. Click ***File*** and choose ***Open Program***



2. Click ***Browse*** and find program in <***PI'S NAME***> folder in "**C:\PANalytical\Data Collector\Programs**"

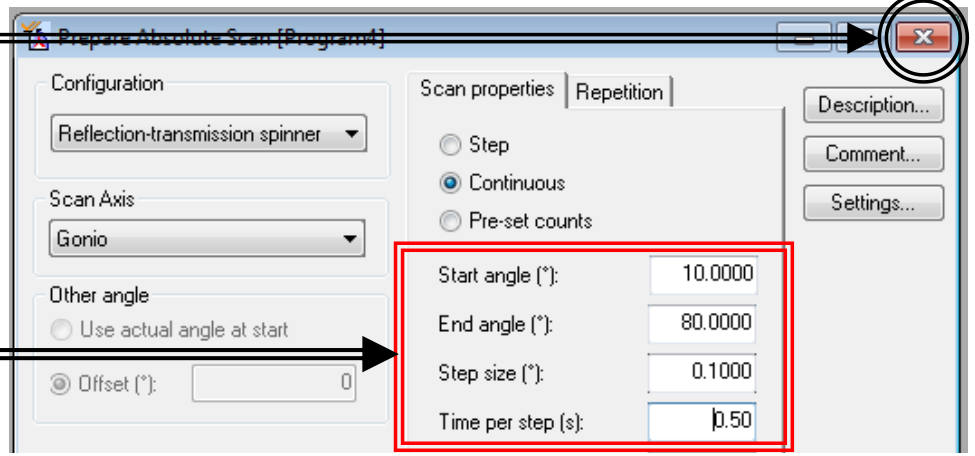


3. Click ***Open***

4. Modify desired parameters

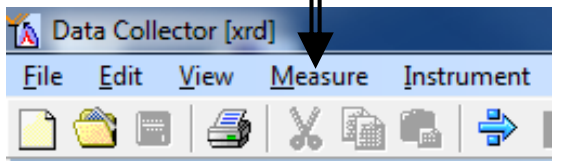
5. Click ***Close X*** when done

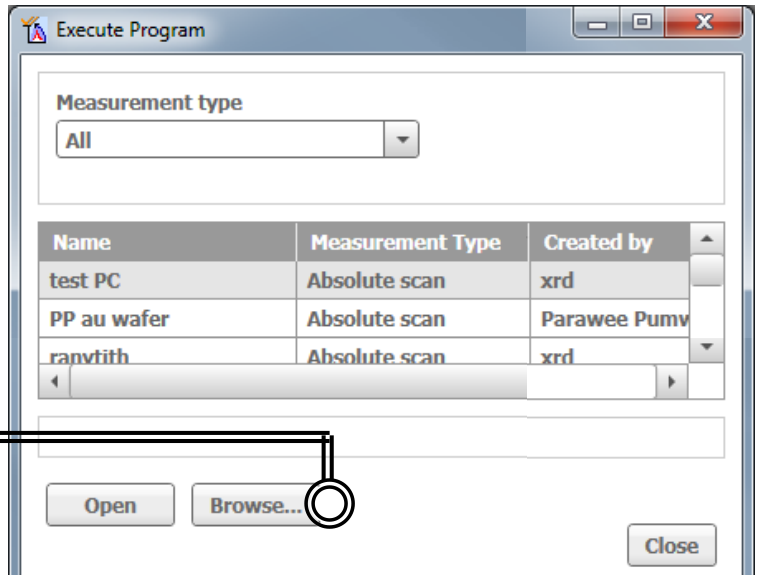
6. Choose to ***SAVE*** your program

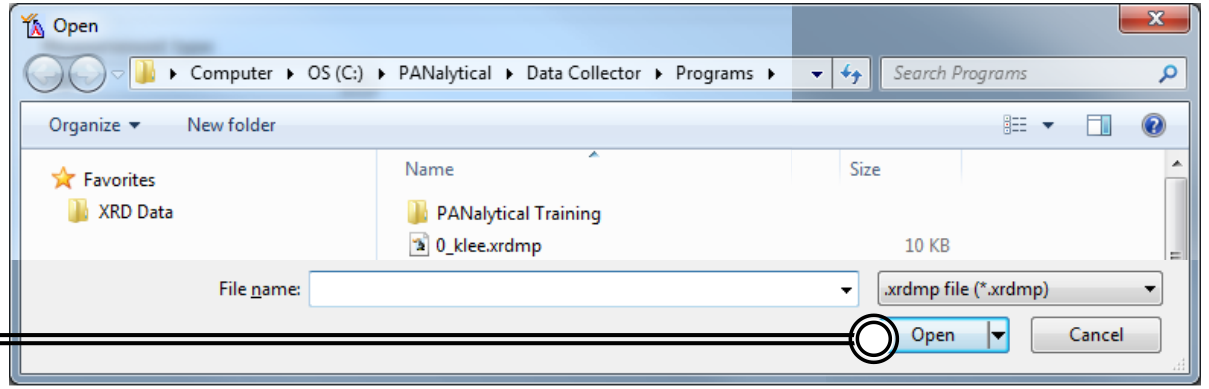


XI. Start Measurement – 1/3

1. Select ***Measure -> Program***
2. Click ***Browse***
3. Default location is "**C:\PANalytical\Data Collector\Programs**"
4. Find your program in <***PI'S NAME***> folder, and click ***Open***

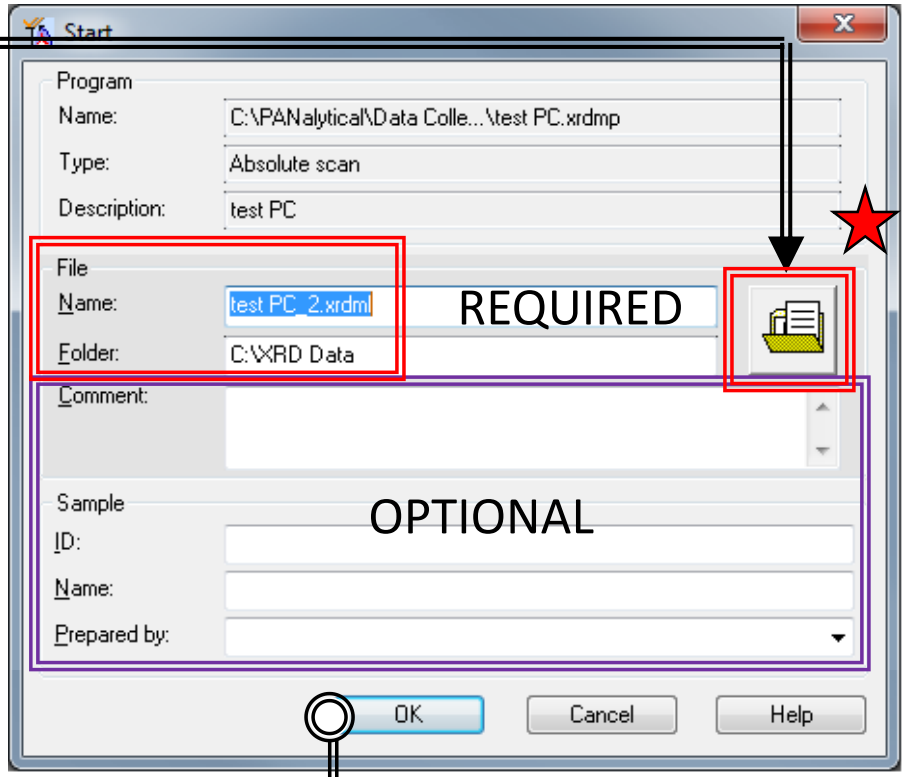






XI. Start Measurement – 2/3

5. ★ Click  icon to change file location
6. Default is unsorted in "***C:\XRD Data***"
7. Select your <***PI'S NAME***> folder
8. Select your ***Folder*** for this scan
9. Enter a ***Name*** for your scan
10. Confirm correct ***File Folder*** location
11. Enter a Comment, Sample ID, Sample name, or Username if desired
12. Clicking ***OK*** will start your scan!
13. If message appears, perform the actions and click on ***OK***



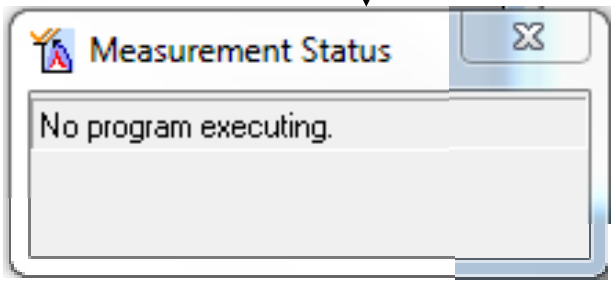
NOTE: THE MESSAGE SHOULD ONLY BE ABOUT CHECKING THAT THE COMBINATION OF SLITS YOU HAVE INDICATED IN YOUR PROGRAM ARE INSTALLED

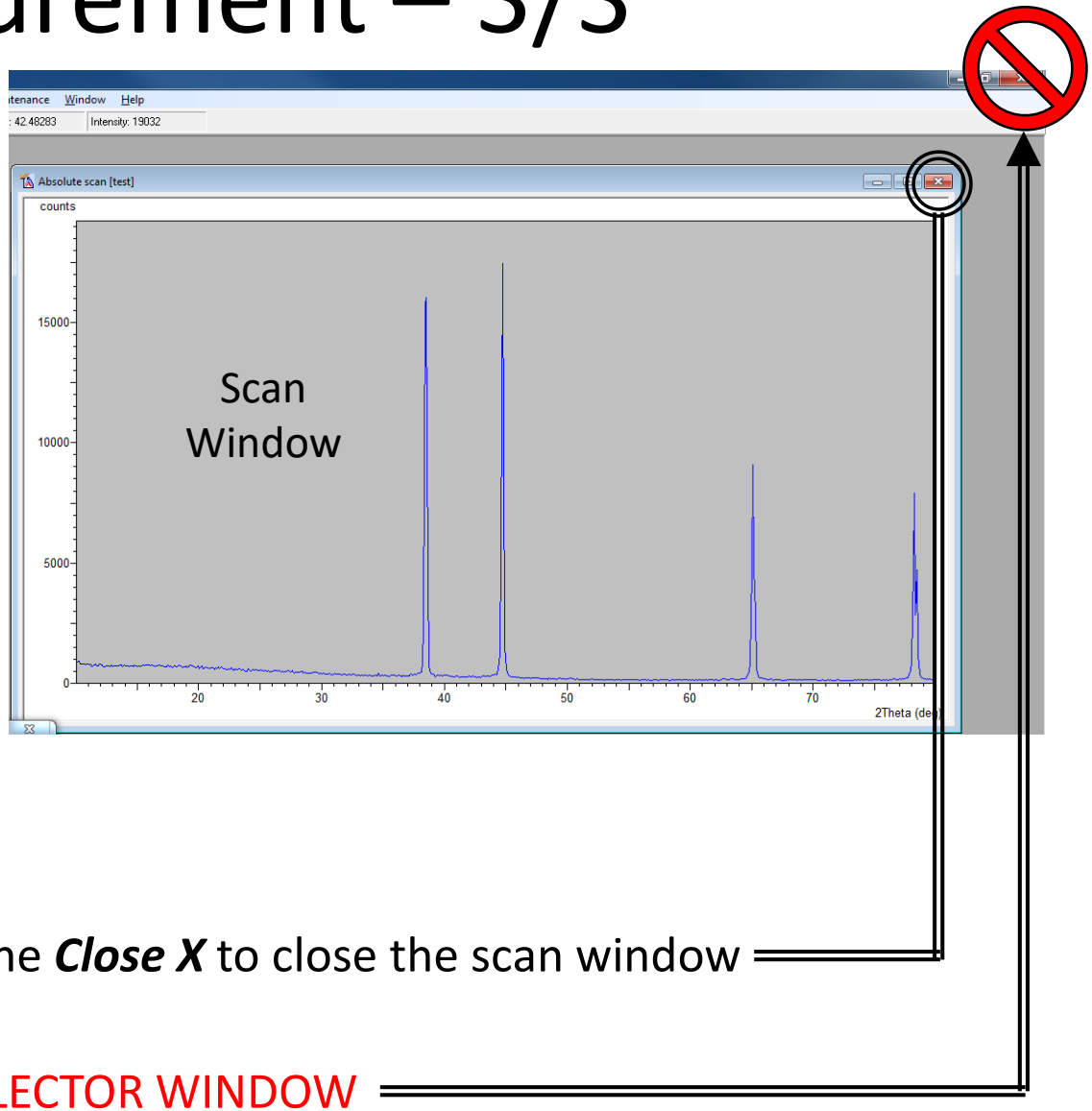
XI. Start Measurement – 3/3

14. The scan will initiate

15. Scale changes as the measurement proceeds

16. Scan is complete when "***No program executing***" is shown



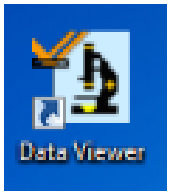


17. Once scan is complete, click the ***Close X*** to close the scan window

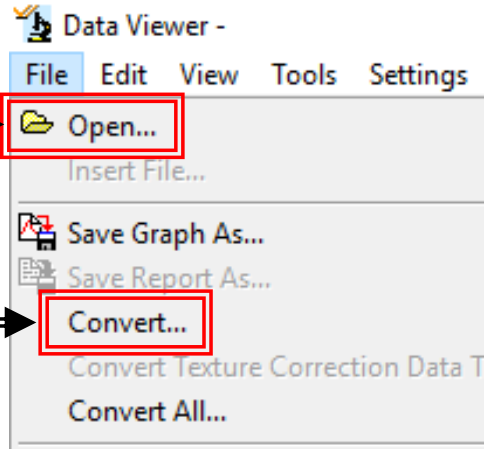
18. DO NOT CLOSE THE DATACOLLECTOR WINDOW

XII. Data Viewing and Exporting – 1/1

1. Double-click ***Data Viewer icon***



2. Click ***File*** -> ***Open***



3. Find your file in "***C:\XRD Data\<PI'S NAME>***"

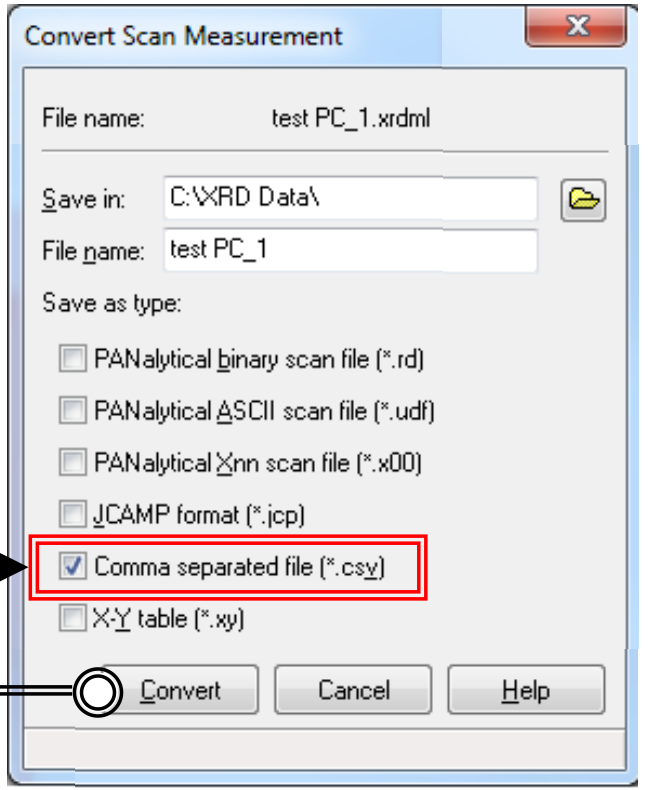
4. Click ***OK*** to view your scan

5. To export your data for plotting in Excel, click ***File*** -> ***Convert***

6. Check "***Comma separated file (*.csv)***" and uncheck everything else

7. Click ***Convert***

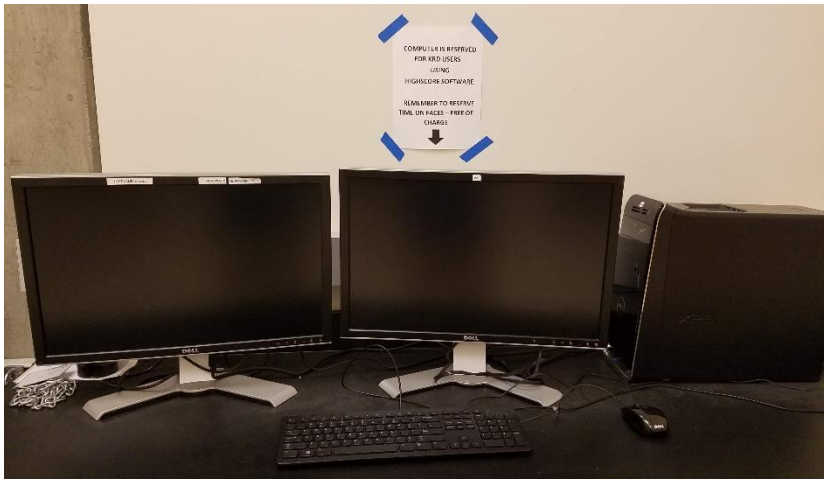
8. A ***.CSV*** file will now be present in the folder you specified



XIII. Data Analysis – 1/1

NOTE: High Score can only be used on "High Score" computer outside

1. If you plan on using ***High Score***, transfer your files directly to computer outside by transferring them to the "***Z***" drive directory (computers are networked) or use a flash drive



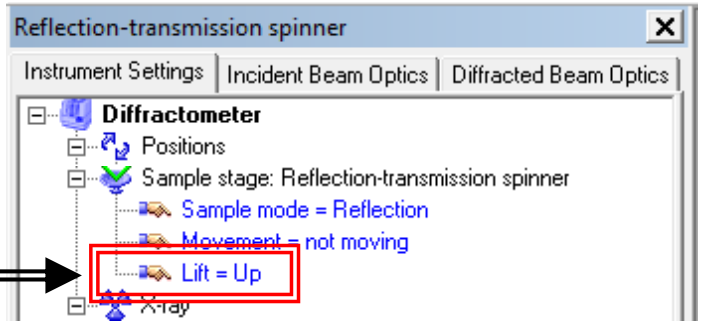
2. Refer to "***Introduction to PANalytical X'Pert HighScore Plus v3.0***" guide by Scott A. Speakman available on desktop of "High Score" computer

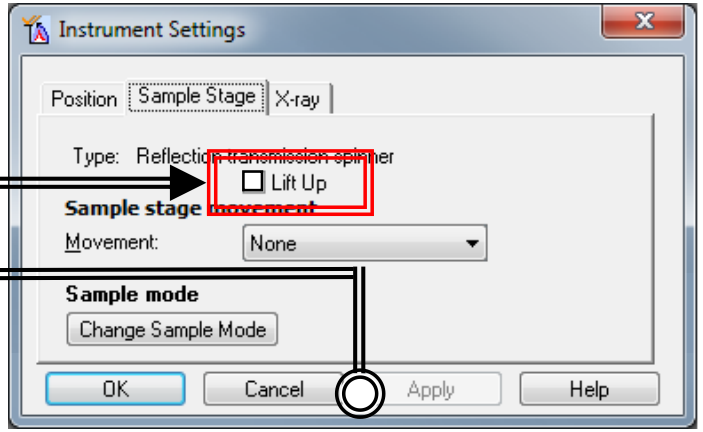
3. Guide is also available on MSE XRD website under Useful Documentation:

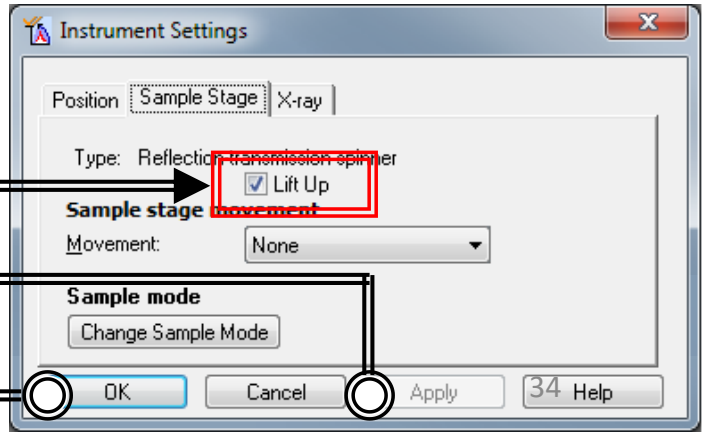
   http://www.mse.ucr.edu/facilities_xrd.html

XIV. Sample Unloading – 1/1

1. Double-click on "***Lift = UP***"
2. Uncheck the "***Lift Up***" option and click ***Apply***
3. Press "***Unlock Doors***" on cabinet
4. Open doors and remove sample holder from stage
5. Carefully wipe down all ***3 Spinner Bearings***
6. Close doors
7. Check the "***Lift Up***" option and click ***Apply***, then ***OK***

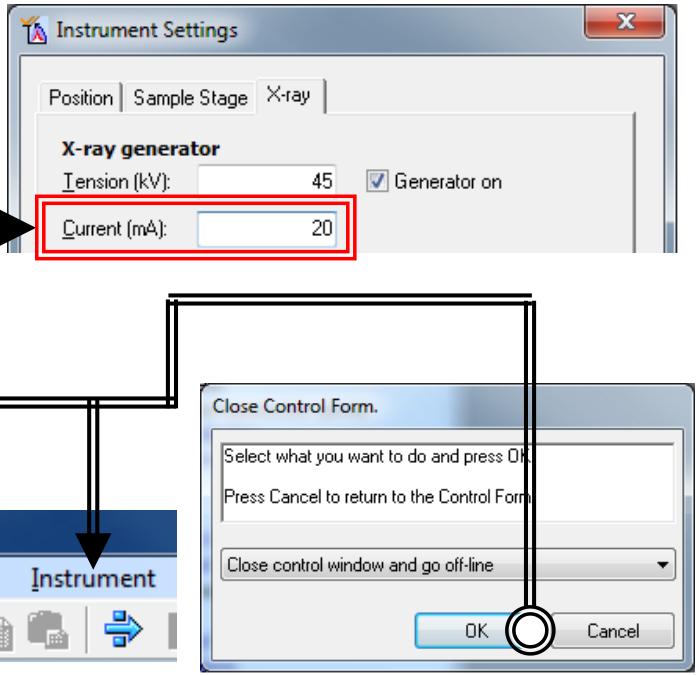




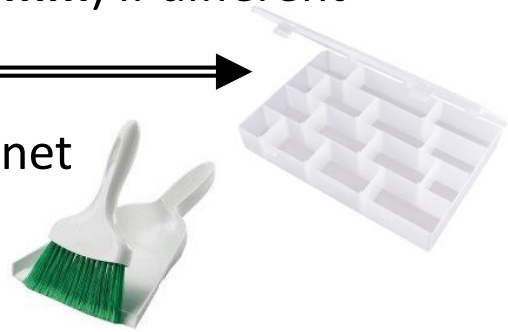


XV. Cleanup – 1/1

1. Double-click on the "***Current = 40 mA***"
2. Change current back to ***20 mA***, click ***OK***
3. Select ***Instrument –> Disconnect***, click ***OK***



4. Select ***File –> Exit*** to log out
5. **CLEAN THE SAMPLE HOLDER!**
6. Return the sample holder pieces to its storage box
7. Replace slits with ***Standard Slit Configuration*** (**1/2°, 1°, 8 mm**) if different
8. Return any other used slits back to its storage box



9. Brush up any sample that may have dropped into the cabinet
10. Turn ***OFF*** the lights to the cabinet (if ON)
11. Close doors (if open)
12. Record your **time-out** on the **sign-in sheet**, slits used, and any issues encountered like dirty sample holders or instrument errors

XVI. Troubleshoot – 1/1

1. If XRD is powered ***ON*** but with ***Tension = 0 kV*** and ***Current = 0 mA …***
2. You can turn on the X-Ray Generator by double-clicking on the ***Status***
3. Proceed to ***Check*** the ***Generator on*** box
4. ***WAIT*** for the Generator to warm up and ***Tension = 30 kV*** and ***Current = 10 mA***
5. Proceed to bring the Generator back to ***Tension = 45 kV*** and ***Current = 20 mA***
6. For **ANY** other issue, please contact the lab manager ASAP
   - Call the lab manager at (951) 827-3378
   - E-mail the lab manager (Perry: pcheung@ucr.edu)
   - Stop by the lab manager's office at MSE 311