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 DataCollector password
XRD Operation

I. Initiate Software
II. Sample Preparation
III. Sample Loading
IV. X-Ray Settings
V. Measurement Program
VI. Start Measurement
VII. Data Viewing and Exporting
VIII. Data Analysis (under construction)
IX. Sample Unloading
X. Cleanup
XI. 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 Login User = <Faces Username> and 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 – 1/4

1. Depending on your sample, the sample holder and preparation will vary...

2. Several sample holders are available for use located in the storage container

3. **CLEAN UP AFTER EACH USE AND WIPE DOWN!**

<table>
<thead>
<tr>
<th>Membrane A</th>
<th>Membrane B</th>
<th>Membrane C</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1" alt="Membrane A" /></td>
<td><img src="image2" alt="Membrane B" /></td>
<td><img src="image3" alt="Membrane C" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Irregular</th>
<th>Miscellaneous Tools</th>
<th>Press</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image4" alt="Irregular" /></td>
<td>Tweezers Scissors</td>
<td><img src="image5" alt="Press" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Round A</th>
<th>Round B</th>
<th>Round C</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image6" alt="Round A" /></td>
<td><img src="image7" alt="Round B" /></td>
<td><img src="image8" alt="Round C" /></td>
</tr>
</tbody>
</table>
II. Sample Preparation – 2/4

Membrane Holders: Sample holder designed for mounting dust filters, sample mounting plats, metal plates, pressed pellets, or silicon substrates.

Diameter = 32 mm
Si Zero Background Plate:
These need to be provided by user.

Irregular Holders: Sample holder can be used to analyze solid samples with a maximum diameter of 45 mm and a maximum thickness of 6.5 mm. The sample can be mounted with wax or clay.
II. Sample Preparation – 3/4

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

CLEAN ALL SURFACES FIRST BEFORE USING!

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

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

Press powder with Aluminum press

Diameter = 16 mm
II. Sample Preparation – 4/4

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

**DO NOT SCRATCH TOP SURFACE!**

Clean mating surfaces with small brush or provided kim wipe.

Invert B to get B*.

Snap on top of A*.

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

The surface of your sample should be smooth via back-filling approach.
III. Sample Loading – 1/1

1. Double-click on “Lift = UP”

2. Uncheck the “Lift Up” option and click **Apply**

3. The stage will now drop down

4. Press “Unlock Doors” on cabinet

5. Open doors

6. Insert sample holder into stage

7. Close doors

8. Check the “Lift Up” option and click **Apply**, then **OK**

9. The stage will now lift up to secure your sample
IV. 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**
V. Measurement Program – 1/10

The following table describes the components for the Incident Beam Side

### Incident Beam Side

<table>
<thead>
<tr>
<th>Component Name</th>
<th>Function or Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soller Slit</td>
<td>Prevents axial divergence and improves peak shape and symmetry</td>
</tr>
<tr>
<td>Divergence Slit</td>
<td>Controls the irradiated length of the X-Ray beam on the sample. Slit size depends on sample size and starting scan angle.</td>
</tr>
<tr>
<td>Incident Anti-scatter Slit</td>
<td>Reduces X-Ray beam scatter and reduces background. Typically double the selection of the divergent slit.</td>
</tr>
<tr>
<td>Beam Mask (not pictured)</td>
<td>Controls axial width of the X-Ray beam. Match to sample size.</td>
</tr>
</tbody>
</table>
The following table describes the components for the **Diffracted Beam Side**

<table>
<thead>
<tr>
<th>Component Name</th>
<th>Function or Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Receiving Slit</td>
<td>Controls the resolution of the instrument, common setting is 0.1 mm.</td>
</tr>
<tr>
<td>Soller Slit</td>
<td>Match with incident selection, typically 0.04 radians.</td>
</tr>
<tr>
<td>Diffracted Anti-scatter Slit</td>
<td>Match to the selection of the Divergent Slit.</td>
</tr>
<tr>
<td>Beta-filter</td>
<td>Used to remove beta radiation.</td>
</tr>
<tr>
<td>Detector</td>
<td>PIXcel 1D</td>
</tr>
</tbody>
</table>
V. Measurement Program – 3/10

The following table describes the relationship between Divergence Slit Size and the Anti-scatter Slit Size for the PIXcel 1D Detector

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

<table>
<thead>
<tr>
<th>Incident Beam Side</th>
<th>Diffracted Beam Side</th>
</tr>
</thead>
<tbody>
<tr>
<td>Divergence Slit Size (°)</td>
<td>Anti-scatter Slit Size (°)</td>
</tr>
<tr>
<td>------------------</td>
<td>-----------------</td>
</tr>
<tr>
<td>Inc Div</td>
<td>Inc Ant</td>
</tr>
<tr>
<td>4°</td>
<td>8°</td>
</tr>
<tr>
<td>2°</td>
<td>4°</td>
</tr>
<tr>
<td>1°</td>
<td>2°</td>
</tr>
<tr>
<td>1/2°</td>
<td>1°</td>
</tr>
<tr>
<td>1/4°</td>
<td>1/2°</td>
</tr>
<tr>
<td>1/8°</td>
<td>1/4°</td>
</tr>
<tr>
<td>1/16°</td>
<td>1/8°</td>
</tr>
<tr>
<td>1/32°</td>
<td>1/16°</td>
</tr>
</tbody>
</table>
V. Measurement Program – 4/10

1. Click **File**
   a. Choose **New Program** if you wish to create a new program (or training)
   b. Choose **Open Program** if you wish to edit an existing program, then skip to Step 23

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

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

4. Choose **Continuous**

5. Click **Settings**
V. Measurement Program – 5/10

6. The default settings show “Actual” for most entries

7. Click Movement, and
   a. Set to Spinning Enabled with Revolution Time = 2 seconds
   b. Set to Not moving if homogeneity is not an issue

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 then Fixed slit 1/2 °
   - Anti-scatter slit: <Enter what you’re using>; if standard then Fixed slit 1 °
V. Measurement Program – 6/10

9. Set the following **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 then *AS slit 8.0mm (PIXcel)*
V. Measurement Program – 7/10

10. Confirm the settings

YOU CONTROL STAGE MOVEMENT CHOICE

VALUES WILL CHANGE DEPENDING ON ACTUAL SLITS USED

11. Click OK
V. Measurement Program – 8/10

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

13. Set Start Angle, (eg. **10°**)
14. Set End Angle, (eg. **80°**)
15. Set Step Size, (eg. **0.1°**)
16. Set Time Per Step, (eg. **30 sec**)

17. Total Time (**h:m:s**) for the scan will automatically update.
V. Measurement Program – 9/10

18. Click the Red X to close the window
19. Choose to **SAVE** your program
20. Name your measurement program file

21. Default location is “C:\PANalytical\Data Collector\Programs”
22. Create a **New Folder** if desired or place in an existing folder, and continue to **VI. Start Measurement** (skip steps 23-27)
V. Measurement Program – 10/10

NOTE: The following steps are for **EDITING** existing program you already created

23. To *Edit* an existing measurement program, click *Browse* and find it in “C:\PANalytical\Data Collector\Programs” or in your own folder

24. Click *Open*

25. Modify any parameters

26. Click the Red X when done

27. Choose to **SAVE** your program
VI. Start Measurement – 1/3

1. Select **Measure -> Program**

2. The list on the right shows the most recent measurement programs created

3. You may find your program faster if you click on the **Browse**

4. Default location is “C:\PANalytical\Data Collector\Programs”

5. Find your program, and click **Open**
VI. Start Measurement – 2/3

6. Enter a Name for your scan
7. Click to change file location
8. Default is "C:\XRD Data"
9. Enter a Comment if desired
10. Enter a sample ID if desired
11. Enter a Name if desired
12. Select your Username
13. Click **OK** when ready for scan
14. If message appears, perform the actions and click on **OK**

**NOTE:** THE MESSAGE SHOULD ONLY BE SWITCHING TO THE COMBINATION OF SLITS PREVIOUSLY RECORDED. IF ANYTHING ELSE, STOP AND CONTACT THE LAB MANAGER
VI. Start Measurement – 3/3

15. The scan will initiate
16. Scale changes as the measurement proceeds
17. Scan is complete when "No program executing" is shown
18. Once scan is complete, click the Red X to close the scan window
19. DO NOT CLOSE THE DATACollector WINDOW
VII. Data Viewing and Exporting – 1/1

1. Double-click **Data Viewer icon**
2. Click **File -> Open**
3. Find your saved file
4. Default folder is “C:\XRD Data”
5. Click **OK** to view your scan
6. To export your data for plotting, click **File -> Convert**
7. Check “Comma separated file (*.csv)” and uncheck everything else
8. Click **Convert**
9. A **.CSV** file will now be present in the folder you specified
VIII. 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 using a flash drive or transfer them to the “Z” drive directory (computers are networked)

2. Double-click **High Score icon**

3. Refer to “*Introduction to PANalytical X’Pert HighScore Plus v3.0*” guide by Scott A. Speakman

4. Also available on MSE XRD website under Useful Documentation:
   
   http://www.mse.ucr.edu/facilities_xrd.html
IX. Sample Unloading – 1/1

1. Double-click on “Lift = UP”

2. Uncheck the “Lift Up” option and click Apply

3. The stage will now drop down

4. Press “Unlock Doors” on cabinet

5. Open doors

6. Remove sample holder from stage

7. Close doors

8. Check the “Lift Up” option and click Apply, then OK

9. The stage will now lift up
X. 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
XI. Troubleshoot – 1/3

1. You may attempt to troubleshoot the XRD for only the following reason:
   1. If the DataCollector software crashes or freezes and you observe the following...
      1. Cannot control the “Lift-Up” feature to remove the sample
      2. Cannot change the current settings back to 20 mA

2. Select **Instrument -> Disconnect** if not already disconnected

3. Attempt to reconnect to the instrument by selecting **Instrument -> Connect**

4. If you receive the following message continue on, else email the lab manager (Perry: pcheung@ucr.edu) or stop by the office in MSE 311 ext. 3378

5. Select **File -> Exit** to close DataCollector and log out
XI. Troubleshoot – 2/3

6. Press the “Off” button
7. The instrument will perform a controlled power-down of the system
8. Wait approximately 30 seconds
9. Toggle the Safety Key from “On” to “Off” 5 times
10. Set Safety Key in “On” position
11. Press and hold the “On” button
12. Wait until the following occurs:
   1. HT-enabled light turns on
   2. X-Rays On light turns on
   3. 15 kV and 5 mA is shown on the console
13. Log-in to DataCollector
14. Select Instrument -> Connect
15. Select Reflection-Transmission Spinner and click OK
XI. Troubleshoot – 3/3

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

17. Click **Yes** and wait for the system to initialize

18. Click **OK**

19. Reset the **Tension = 45 kV** and **Current = 20 mA**

20. Continue with experiments