## PANALYTICAL X-Ray Diffractometer – Quick START by Kennedy Nguyen

Before your session:

1. Book time on **Google Calendar** for the Panalytical XRD – "Name/PI" on your time slot.

2. Have your **samples ready** to mounted on the sample holders (or already mounted) During your session:

- 1. Sign in on Login-sheet
- 2. **Check**: HT is on (40kV and 10mA). Check that the water chiller is running with temperature between 68-72°F. If not, contact the IMF lab manager.
- 3. Login to the computer with your user account.
- 4. Click on **Data Collector** and login using your UCMID and password
- 5. Make sure the **doors of the XRD are closed** (shutter lights are off).
- In the top menu: Instrument -> Connect. The XRD is connected to the computer when you see the Instrument settings/Incident Beam Optics/Diffracted Beam Optics menu pop up the left side. Use Reflection – Transmission Spinner.
- 7. In instrument settings, click on Tension value, **change settings to 40kV and 45 mA** for Cobalt source. Be sure to keep as line focus.
- 8. Insert your sample into correct position (usually default is position 1)
- 9. If you already have a program set up, you can edit it by going to File -> Open Program. If you need a new program, you can also <u>File-> New Program</u>. Chose Absolute Scan. Edit the parameters and then save (Make sure in settings, the "Actual detector" is changed to "X'celerator" to get an accurate time estimate). Sample (changer) batch programs can also be created here. Use reflection transmission spinner and gonio stage.
- 10. From the top menu, go to **Measure->Program**... and select your program. Double check that the files will be saved where you want them to be saved. Here is also where you edit the file names. The sample will be analyzed. *Be sure to wait and not leave the room immediately, since errors may popup and you may need restart the program. Check your run occasionally if you have a long run.* After your scan is complete, set the tension back to **40KV and 10mA**.
- 11. Go to Instrument->Disconnect.
- 12. Close the Data collector Program.
- 13. Sign off on the log sheet. Report any issues, otherwise put "ok".
- 14. Remove your sample and return the sample holder (cleaned).
- 15. Convert your raw files into the appropriate format (ie. Excel) and then logout of the computer.

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Editing your data and converting Data Collector data to alternative format (Quick Guide)

- 1. Open "HighScore" Plus Software
- 2. Open your file (saved from Data collector: \*.XRDML)
- 3. To remove background: Treatment -> Determine Background...(Subtract and replace if needed)
- 4. To smooth out the data: Treatment -> Smooth
- 5. To convert Co source data to Cu data: Treatment -> Corrections -> Convert Wavelength
- 6. If you have sharp peaks: Analysis -> Search & Match -> Execute Search & Match...
- 7. To compare you data to a database:
  - a. Reference Patterns ->Retrieve Patterns By ->Restrictions...
    - i. Select the elements present in your sample (you should know a bit about your sample)
    - ii. Select the appropriate "Quality" in the quality tab. If you have too many results, you can choose the appropriate quality accordingly.
  - b. Reference Patterns ->Retrieve Patterns By ->Reference Code...
    - i. If you know the reference code
    - ii. HighScore plus software has it.
- 8. Compare the Pattern List to identify your sample in "Analysis View" (bottom tab of spectrum).
- 9. To compare data files, after opening the file, in the same tab, click on insert Data file (top tab of spectrum).
- 10. Save file as: select appropriate file format
  - a. File -> Save As...
  - b. Choose the appropriate location and file name
  - c. For Save as type: Choose your preferred format type for data presentation
- 11. To convert the data into Excel, save the file as "General ASCII scan (\*.ASC)"
  - a. Then open the file in Excel (Remember to chose "All Files" to view the \*.asc file.
  - b. Choose Delimited and Next.
  - c. Choose your "Delimiters" as "Space" and click Finish
  - d. Save the file as an Excel file afterwards, since the default is Text (Tab delimited)

For more advance usage, refer to the help menu of the HighScore Plus Software.

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Starting system from Shutdown

- 1. Turn on the water chiller (breaker switch and power to water chiller)
- 2. Turn on the breaker switch for the XRD (if it's off)
- 3. Turn the HT key switch on the control and display panel clockwise.
- 4. Make sure the doors are closed.
- 5. Press the "Power On" button. The "Standby" lights will go off and the "Power On" light will be lit.
- 6. Login to the computer and start initialization (There are not pre-module instruments for our instrument).
- 7. If the instrument has been off for more than 100 hrs, it's recommended to do tube breeding.

Shutting down the instrument

- 1. Set HT to minimum values: 15kV and 5mA.
- 2. Disconnect from instrument and logout of the computer.
- 3. Switch the HT generator off by turning the HT key switch on the control and display panel counter-clockwise. ("X-rays On" Lamp will turn off)
- 4. Press the "Standby" button on the control and display panel. The Power On light will switch off and the Standby light will turn on.
- 5. Turn off the water chiller.
- 6. If there is an emergency or planned power outage, also turn the power breaker switches down for the water chiller and XRD.