Instructions for using Rigaku Miniflex X-ray Diffractometer (XRD)

Startup – if XRD is already started, with X-ray tube on, skip to step 4

1. Turn on cooling water (water chiller to the left of the Miniflex), wait for two minutes
2. Turn on main switch to XRD in the back
3. Prepare sample(s). *Ensure no sample is being X-rayed before sliding open the door of the XRD.* Load sample into XRD chamber [Note position on the sample changer. Magnetic clamps should hold disc in place.].
4. When green ready light turns on, turn X-ray tube on (green ready light will go out, X-ray light takes 10-15 sec to turn on)

Sample runs

5. Boot up computer if not started already (User: Administrator, Password: rigaku). Double-click **Standard Measurement** from the desktop.
6. Fill in the condition line(s), from left to right (click or double-click the cell to alter parameters):
   - *Use* – Toggle between "No" and "Yes" on whether or not to use the condition line
   - *Print* – Toggle between "No" and "Yes" on whether or not to print the output
   - *Folder Name* - Directory path where you would like the data to be saved
   - *File name* - Filename of your x-ray scan (keep it simple) [both Folder and File name can be altered using "Browse…"]
   - *Sample Name* - Description of your sample for identification purposes
   - *Attach.* – Sample rotation setting. Double-click on the number, a pop-up table will appear that associates numbers with the sample rotation status (pull down menu).
   - *Condition* – Double clicking the number opens the conditions table, in which numerical tabs are associated with different settings. Specify the conditions of the scan (Default: 5 to 60 deg. 2θ, 0.04 deg. step size, 20 deg/min scan speed). The units for scanning width and speed are degree and degree per minute, respectively.
   - *Sample* – Sample position in the circular changer (1-6). If you wish to scan more than one sample, add more condition lines and alter the sample number
7. Press yellow **Execute measurement** button in the upper left of the window to begin scan

Shut down

8. After data is collected, it will be saved in your chosen directory. Use **PDXL2** (administrator: no password) to analyze patterns [PDXL2 User manual available through "Help" menu].
9. Turn off X-ray tube and computer when finished. Wait 10-15 min for X-ray tube to cool down before switching off the cooling water and main power supply.
10. Sign up the logbook and Log off the login system in the hall way outside the lab.

Basic Instructions for Operation of PDXL2 Software

Starting software

1. Double click on **PDXL2** icon (located on desktop or found in start menu).
2. Login: administrator | Password: <leave blank. No password>
3. In the "Flow" column on the left, click **Data Process**. Confirm on the right column that **Auto** is selected in the "Analysis – Data process" bar.

Opening a file

1. Click on **File -> Load Measurement Data…** or Click on **Load…** on left side "Flow" window
2. Find the folder, where your data is stored (typically "C:\\Windmax\Data\<username>\"). Choose your file.

Identifying crystalline phases

1. Click on **Auto Search** on left side "Flow" window
2. Click on **Auto Search…** in right side "Analysis – Identification (Auto Search)" window
3. Depending on your application (within Sub-file tab), either use "All sub-files" or "Specify sub-files" to limit your database search
4. Select **Elements filter** tab. A periodic table will open up.
5. Choose elements present in your sample by clicking on them. Clicking on "Unknown, Not Included, Included, Include One at Least" buttons will select all elements to that setting.
6. Select **Other** tab. Click **Default** button and check **Show only phases with RIR value** box.
7. Click **Execute** button when ready to initiate the search.
8. "Candidate phase" window will populate with best (low) **figure of merit (FOM)** values.
9. Organize what candidates you believe are included in the composition of your material. Click on the up or down arrows to either remove or add candidates (respectively). If you click on "+" of a candidate phase, options for similar phases will open for your selection.
10. Once candidate phases are chosen, click **Set** in the lower right. Peaks shown in the "Information" window will have the appropriate phases assigned to them (scroll to the right to see them).
11. Click on **Load Card Info…** on the left side "Flow" window. This transitions from searching the database to being able to look at how the chosen/set candidate phases match up to your spectrum. Click **Card Info…** in the "Analysis" window on the right side. A card search window will pop up. You will notice that while this search window is open, there is additional labeling on your spectrum. Your candidate phases have markings where the peaks are registered in the database.
12. Pull up database card information as needed (by limiting your search field and searching). This allows you to visualize how the pattern changes with different crystal symmetries (clicking the checkbox of a particular search result will overlay the database information on your spectrum data).
13. If a better candidate phase is found, click **Add** in the lower right of the database search window. This will add your chosen entry into the "Analysis" window "Search Results"

sub-window in the “Database” category. Make your chosen element a candidate phase by selecting it and clicking the down arrow, then clicking **Set**.

14. To print the phase-spectrum associations, click **Create Report…** in the “Flow” window and select **Phase Identification** only. A Word document report will be generated of the currently active region of the spectrum (so you can change the zoom if you wish… see next section). Save it as necessary.

Selecting only part of spectrum and going back to whole spectrum

1. Make sure the magnifying glass icon is selected from the top toolbar menu.
2. Click and drag the mouse on the top panel to select a smaller portion of spectrum.
3. Double-click anywhere on the spectrum to go back to full view.

Quantitate composition

1. Click **RIR Method** in the “Flow” window to initiate a quantitative method based on reference intensity ratio (RIR) and integral intensity of the strongest peak of each phase. The method uses the candidate phases set in the previous step to match peaks and quantify compositional content.
2. A colored graph will appear showcasing the % composition of your material based on the candidate phases previously set. A more detailed report can be obtained from **Create Report…** in the “Flow” window, selecting “RIR Method” only.

Loading crystal structure parameters

1. Click the **Phase Information** tab in the “Information” window, then the **Crystal structure information** tab.
2. Select the phase candidate you wish to import crystal information, then click **Import CIF…** to select the .cif file and import the crystal structure parameters.

Saving data using a text editing program (Notepad or Excel)

1. Click **Edit -> Copy Data -> Measurement/Processed Data**
2. Two column data set (angle and intensity) is now copied to the clipboard.
3. Paste the data to a text editing program and svae.

Changing axes

1. Click on “View” -> “Change axes”.
2. Select the desired axes (typically 2Theta for x-axis and Counts for y-axis).

Printing (HP Officejet 6100 is just to the left of the machine). Be patient. Takes time to spool.

Data Transfer: Computer is connected to the internet. If you have a server or cloud solution, you are welcome to use it. We would prefer server usage over flash drives as they often introduce malware/viruses into computer systems.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. If unsure, leave system as is.
3. Try to turn off X-rays and power down the system.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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