Standard Operating Procedure: X-ray Diffractometer (XRD)

Rigaku MiniFlex 6G

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1. Lab Safety Information

- ✓ All GMU NFF users are required to complete the Lab Safety Orientation (LSO) before performing any lab work.
- ✓ Proper Personal Protective Equipment (PPE) should always be worn before entering the clean room: safety glasses, hair net, shoe covers, gloves, and lab coat. Additional PPE is available for specialized chemical work as needed.
- ✓ No shorts, sandals, tank tops, or spaghetti-strap shirts are allowed in the clean room!
- ✓ Material Safety Data Sheets (MSDS) are available in a binder in the gowning room.
- ✓ Read the SDS for any chemicals you plan to use before proceeding with your work. Any materials used in the clean room for the first time should be brought in after the approval of NFF staff.
- ✓ A safety buddy is required in the clean room with you when doing chemical work. The safety buddy should be fully trained and qualified to work with the chemical you are using. They must remain in the clean room the entire time you are handling the chemical. Feel free to ask NFF staff if no one qualified is available!
- ✓ Prohibited clean room items: cardboard, pencils, cloth, hats/coats, and contact lenses.
- ✓ Accepted clean room items: plastic, pens, synthetic fabrics, clean room paper.

2. XRD Safety Information

- ✓ Any irregular system behavior should be reported to NFF staff promptly. Never attempt to fix the system yourself! We are here to help.
- ✓ Every user should attend mandatory X-ray safety training and X-ray specific training organized by EHS and stay informed about updates in safety procedures before the equipment training.
- ✓ Hazards:
    - XRD generates strong X-rays that are harmful to the human body and can cause significant radiation damage.
    - X-ray tube windows incorporate metallic beryllium that is harmful if inhaled its powder or touched by a part of the body.
    - The high-voltage unit inside the XRD can cause electric shock.
- ✓ Before starting the equipment, check the instrument to ensure the following:
    - Neither the inside nor outside of the enclosure shall be wet with water.
    - No abnormal odor shall be detected from the main body, power cable, water pump, or any other component.
    - The OPERATE lamp on the front display panel of the instrument lights in yellow.
    - The external chiller temperature should be at the room temperature range.
- ✓ The system has safety devices in place to shut down the X-ray generator in case of any malfunction. Therefore, do not remove or modify the equipment under any circumstances. In particular, removal or disassembly of the X-ray shutter could result in exposure to a massive dose of X-rays.
- ✓ In state of emergency, press the red Emergency Off (EMO) switch at the right area of the front side of the instrument to stop running the equipment. OPERATE lamp on the display panel in front of the main body will light off. The power will be supplied up to the circuit breaker only.
- ✓ Failure to use the system safely and properly may result in your access to the system being reviewed and/or revoked.
- ✓ Fill out the logbook before you begin.

3. Principles of XRD

XRD is used for analysis of unknown crystal structures to determine their composition, and for evaluation of material characteristics. X-rays were first discovered in 1895 by a German scientist named Wilhelm Conrad Röntgen. Röntgen's research was expanded upon by Max von Laue, who determined the wavelengths of X-rays to be in the range of 0.1 Å to 100 Å, and postulated that crystals are comprised of atoms of sizes in the X-ray wavelength range arranged in an orderly manner.

A key principle behind XRD is Bragg reflection. Bragg's Law states that at a certain incident angle θ, the constructive interference of X-rays reflecting off a crystal structure will cause them to scatter at an angle of 2θ. As the spacing between crystal lattice planes increases, the Bragg angle decreases proportionally. Additionally, as the number of lattice planes increases, the number of values for the angle θ that satisfy the condition for Bragg's reflection decreases, and the intensity of the reflected rays increases. In some instances, reflected rays may destructively interfere with one another, reducing intensity and causing what is known as extinction.

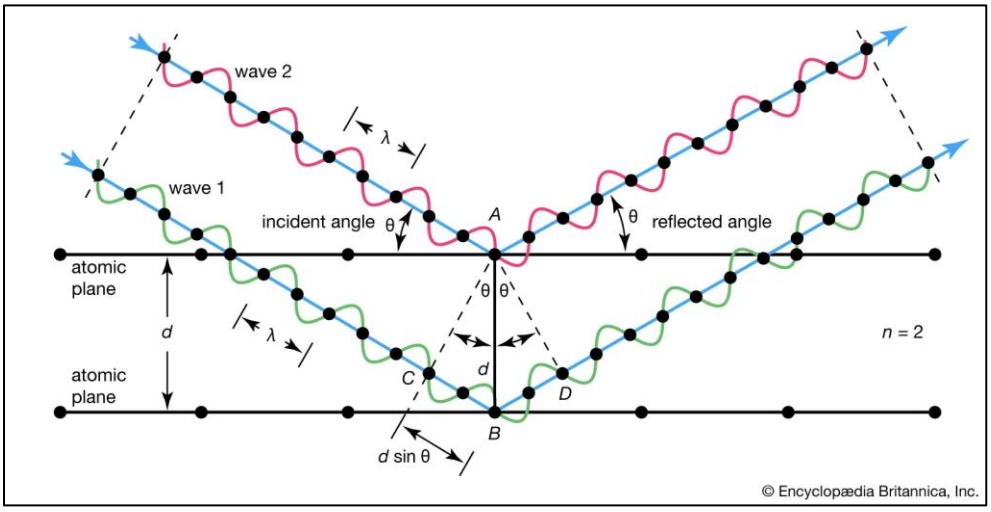


Figure 1. Bragg's Law

An XRD sends focused X-rays towards a sample to be reflected and received at the other side by a detector, which measures the intensity of the reflected rays as the scattering angle 2θ is varied. Intensity peaks at certain angles can be compared against a database of materials with known diffraction profiles to determine the crystal structure and/or identity of the sample.

The Rigaku MiniFlex 6G at NFF is a tabletop XRD equipped with a 600 W Copper X-ray source and a D/TEX Ultra detector. Several options for sample holders are available, including the

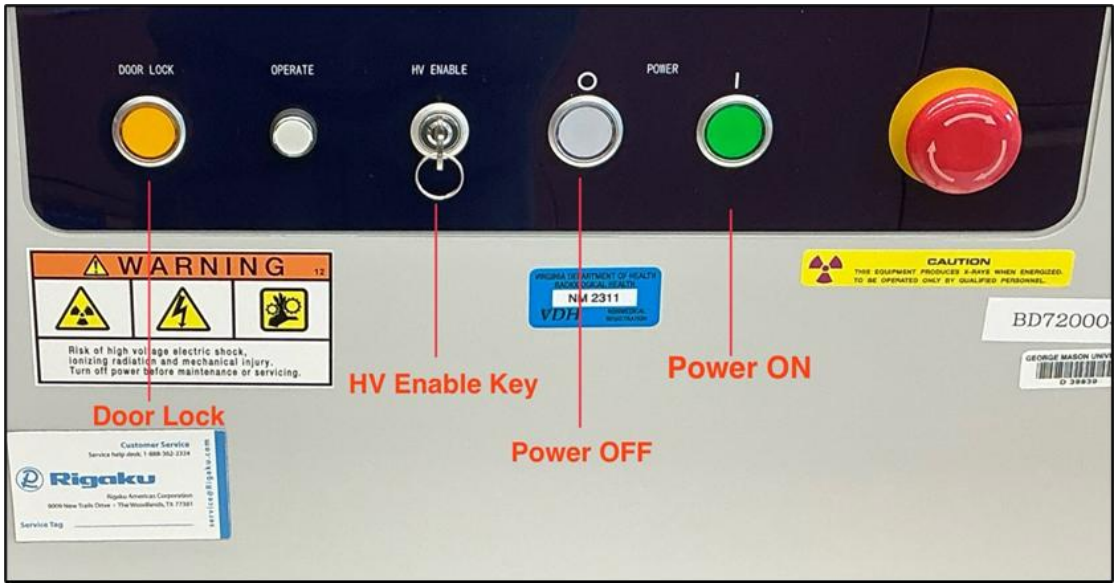
standard sample holder, and an 8-slot automatic sample changer that allows multiple measurements and rotation. The basic package includes tools for data processing, phase identification, user database creation, RIR quantification, crystallite size analysis using the Scherrer method, and crystallite size and lattice strain analysis using the FP method.

Figure 2. Rigaku MiniFlex 6G XRD

4. Operation Manual

4.1. Turning on the XRD

1. Turn the HV enable key to the horizontal position.
2. Press the green power button to turn on the machine.
3. The door can be opened when the yellow door lock button is blinking.
4. Make sure the OPERATE lamp next to the door lock turns yellow.



4.2. Sample preparation and loading

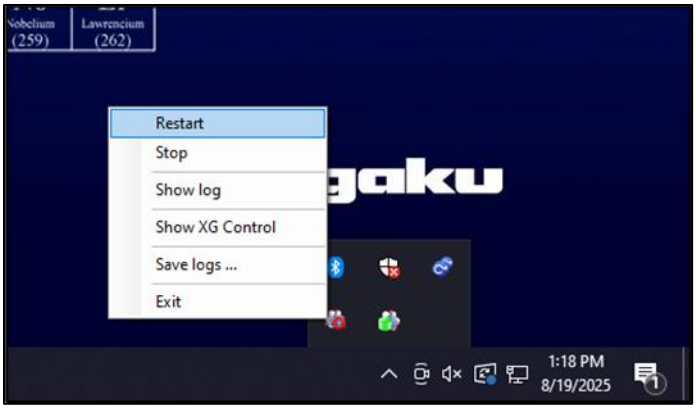
5. Sample preparation methods vary depending on the type of sample (powder, bulk, etc). Prepare the sample according to Rigaku instructions, or ask NFF staff for help.
6. In general, pour your sample into the sample holder. Flatten and compress the sample surface. For powders, the top of the sample surface should be perfectly level with the sides of the sample holder.
7. Wipe around the sample filling section to remove any sample remains by using a cleaning paper soaked in IPA in the lab.
8. Lift up the knife edge to facilitate easier sample holding.
9. Place the sample with the sample holder in any position of the 8-slot automatic sample changer. Which position is chosen does not matter as long as you note the corresponding position number.
10. Lower the knife edge after placing the sample and before closing the door.

11. Make sure that the HIS slit, DS slit, Soller slit, Kβ filter, and direct beam stop are always installed. Do not attempt to remove them.
12. Close the chamber. Lock the door by pressing door lock button. When the door lock is engaged, the yellow light should be solid and not blinking.

4.3. Software

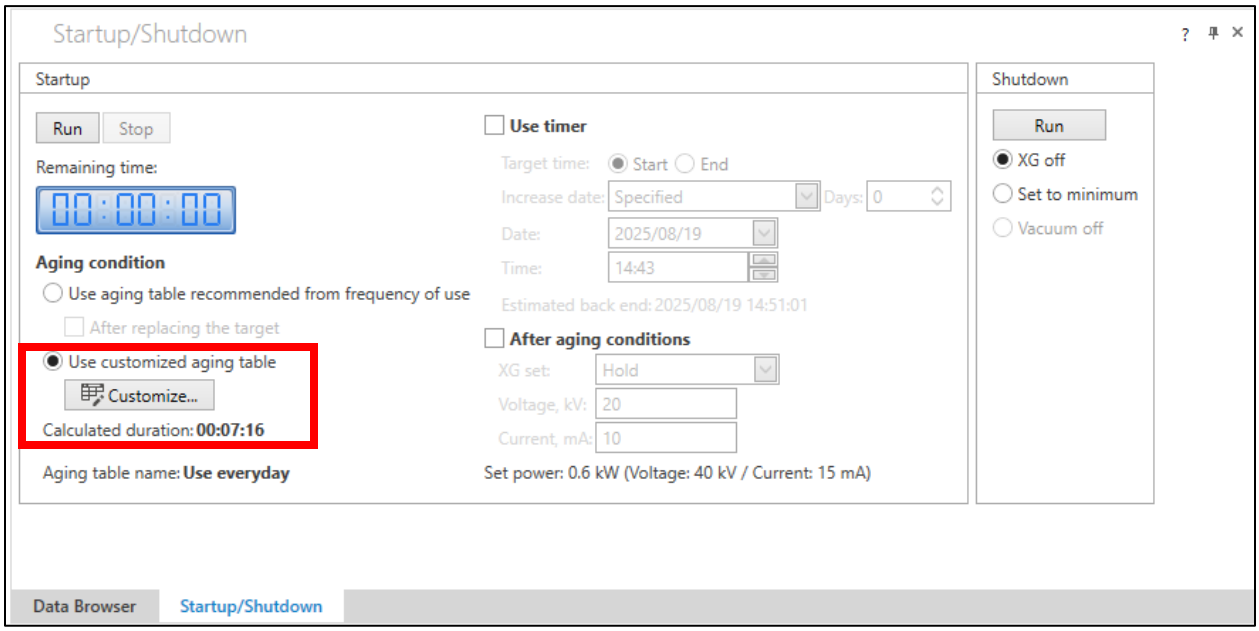
13. Open the SmartLab II Studio Software by right-clicking on the program icon on the desktop, and select "Run as administrator."

14. In the bottom left of the screen, check that the H/W status is ready and connected.
    a. If it is not ready, close the software.
    b. Go to the Windows task tray and restart the I/C server icon, and reopen the software afterwards.

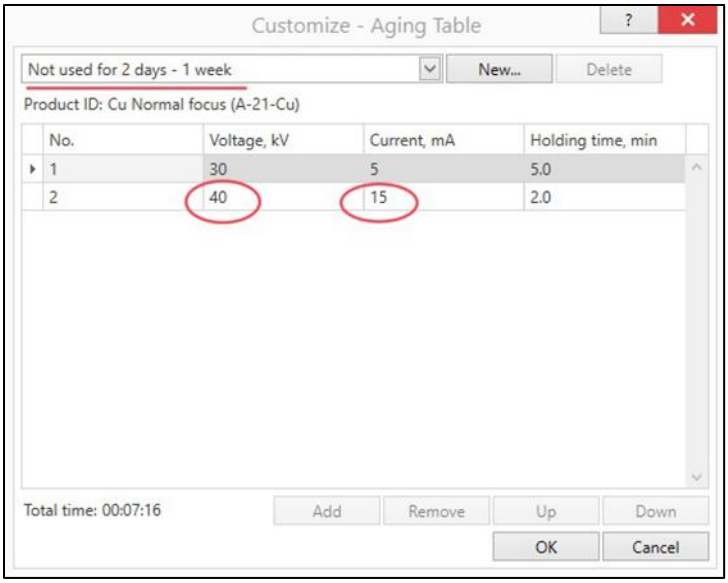


4.4. Turning on the X-ray

15. In the “Startup/shutdown” menu, select the option to “use customized aging table”

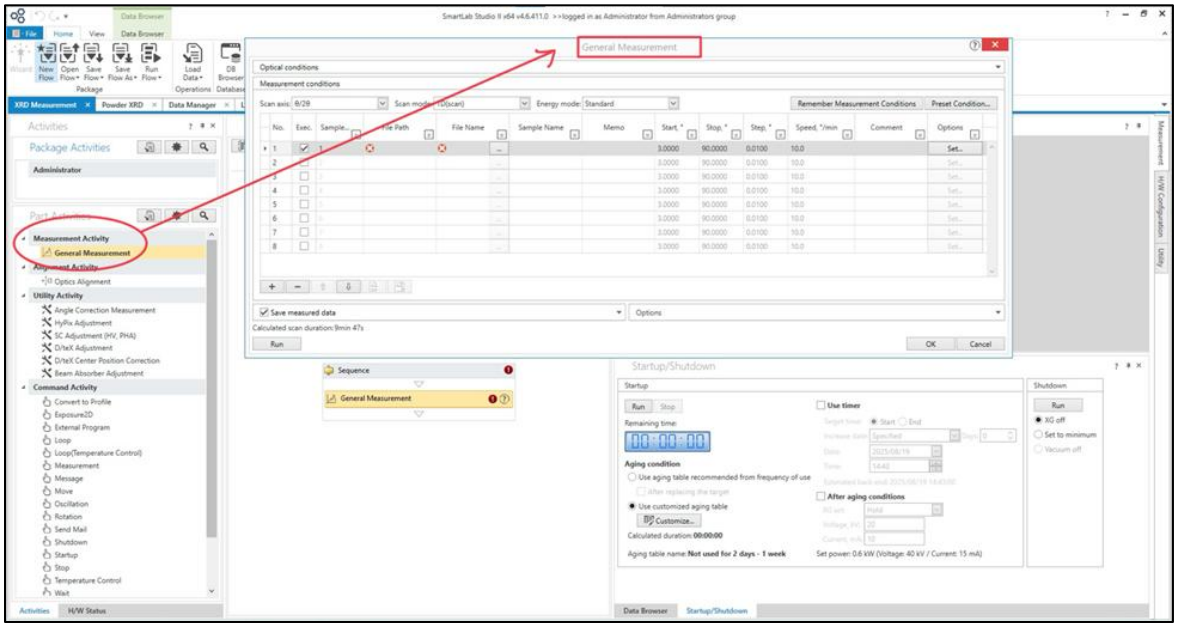


16. In the window that pops up, select “Not used for 2 days - 1 week”, and make sure that the final operating conditions are at 40kV and 15mA.
17. Click “Ok”, and “Run” the startup. It should take 7 minutes.



4.5. Preparing for measurement

18. Go to the "Activities" tab and click "General measurement." This should bring up a new window.

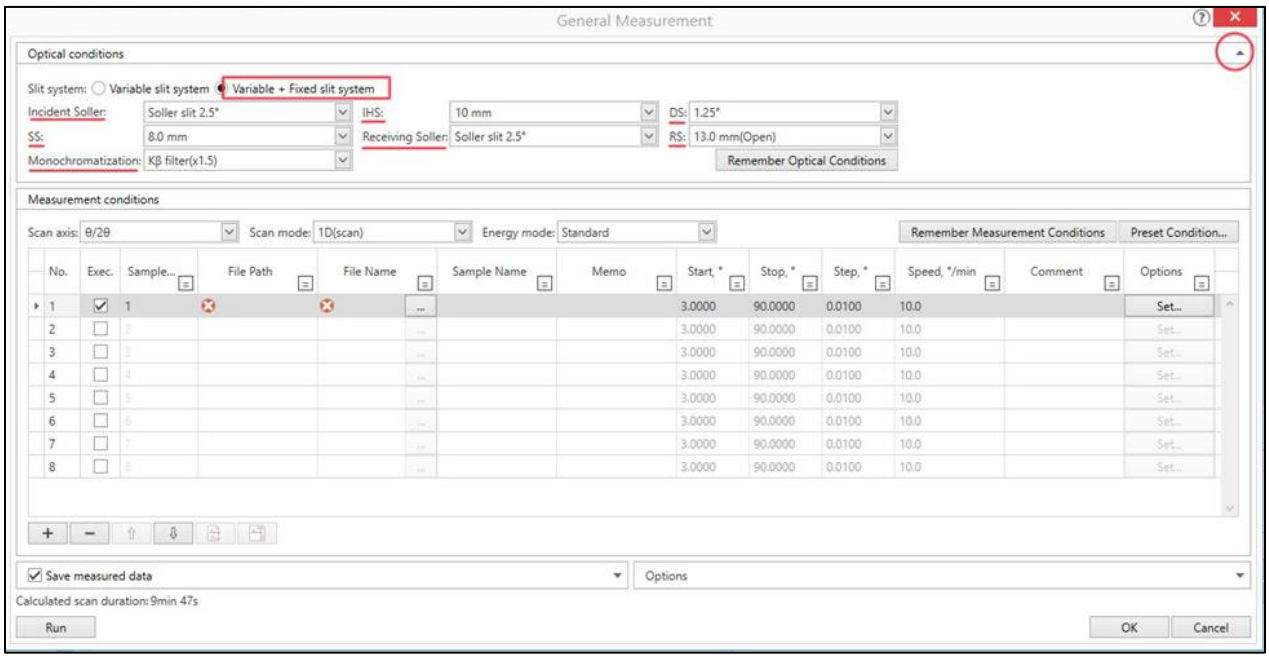


19. Open the "optical conditions" menu, which is collapsed by default. Check that the following conditions are true:
    a. "Variable + fixed slit system" is selected
    b. Monochromatization: kβ 1.5

c. Incident soller: 2.5

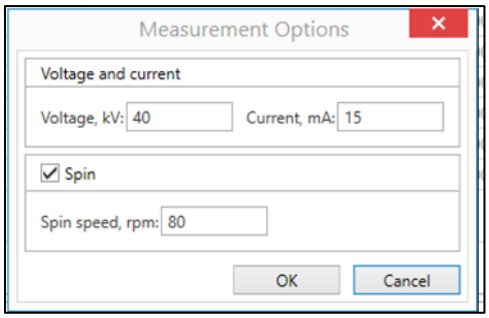
d. IHS: 10 mm

e. DS: 1.25

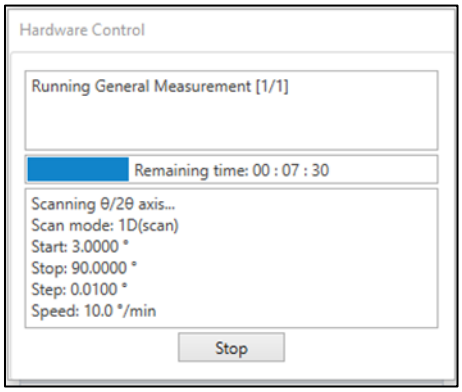


20. Continue to the "measurement conditions" selection area.
21. The scan axis (θ/2θ) and scan mode (1D scan) cannot be changed at this time. For the energy mode, it is recommended to always start with the standard scan. If the background is unusually high, or if the sample contains large amounts of Mn, Co, and/or Fe, then the XRF mode can be used after first attempting a scan in the standard mode.
22. In the table, a checkmark on a row indicates that a scan will be performed with the specified conditions.
23. Select the "Sample…" cell and input the numeric position the sample was loaded in.
24. Select the three dots ("…") in the cell in the "File Name" column to choose where to save the scan file. Type the name of the file to be created as well.
25. The default start/stop angles are 3° to 90°. It is recommended to start with these values to avoid missing any peaks. If you are more familiar with your samples, users scanning organic samples can stop around 40°, while inorganic samples may have peaks appearing up to 80°-90°.

26. Keep the step size at the default value of 0.01. If the data is noisy, retry the scan with a step size equal to 1/10th of the peak width at half maximum.
27. Generally, faster scan speeds result in lower resolutions, and it is up to the user to determine what scan speed to use. However, lower scan speeds are recommended to reduce noise. The minimum scan speed is 0.1°/min, and the maximum scan speed is 100°/min.
28. If the crystal grain size is large, or to avoid the preferred orientation problem, the sample may be spun to obtain a more averaged view of the peaks. To access this option, click the "Set" button under the "Options" column to enable the spin, as well as to set a spin speed. The maximum spin speed is 80rpm.

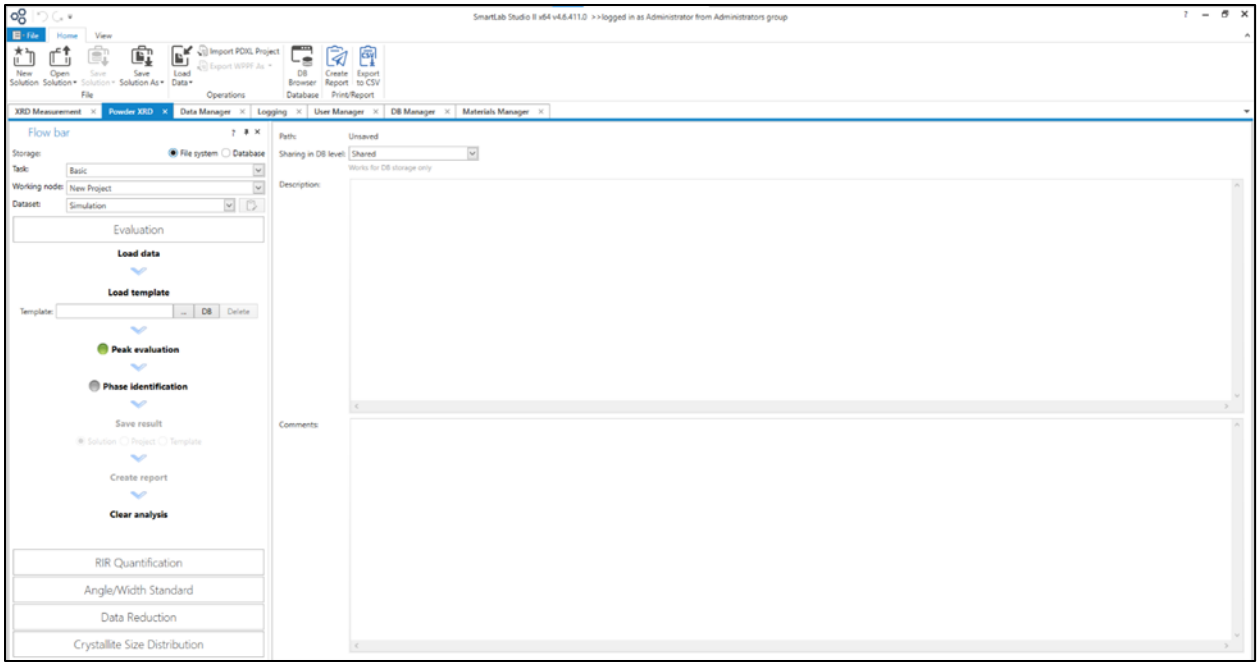


29. Click "Run" to start the measurement. The estimated scan time is displayed above this button.

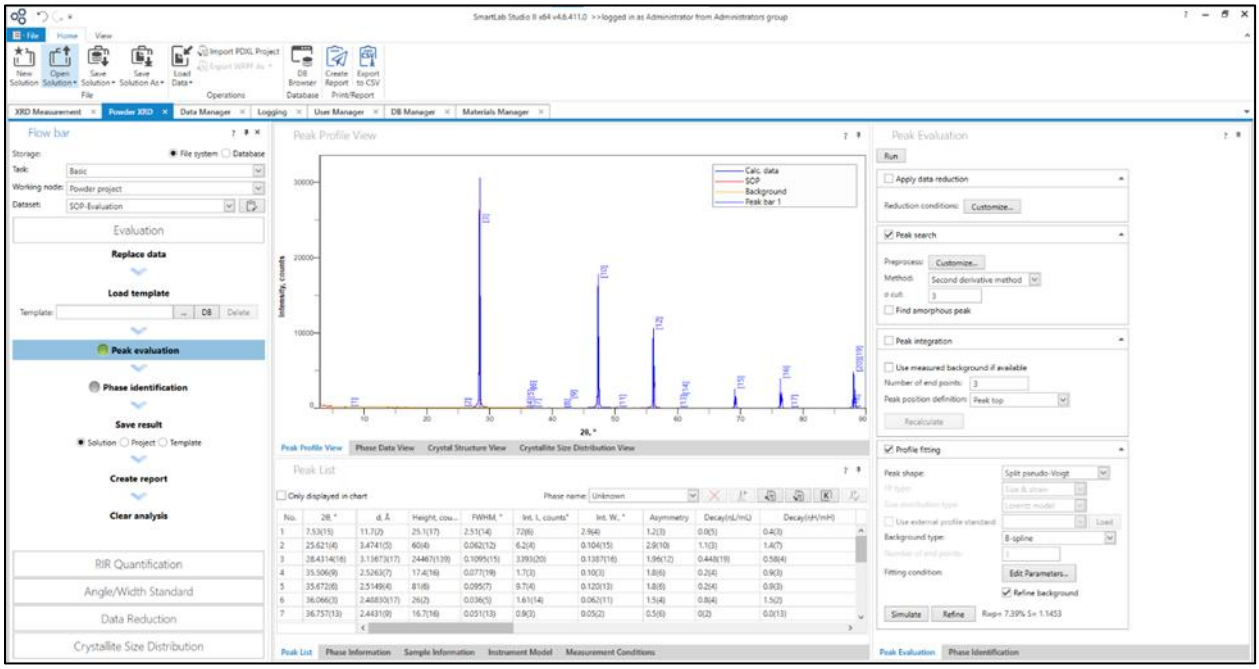


4.6. Analysis

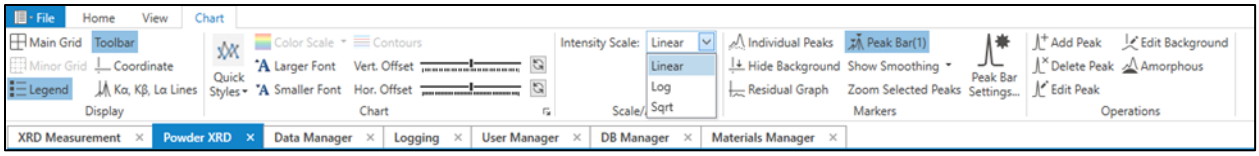
30. In the Powder XRD tab, the left sidebar shows the order of steps to be taken.
31. Click "Load Data" to load the measured data from a saved measurement file, which should be in rasx format.



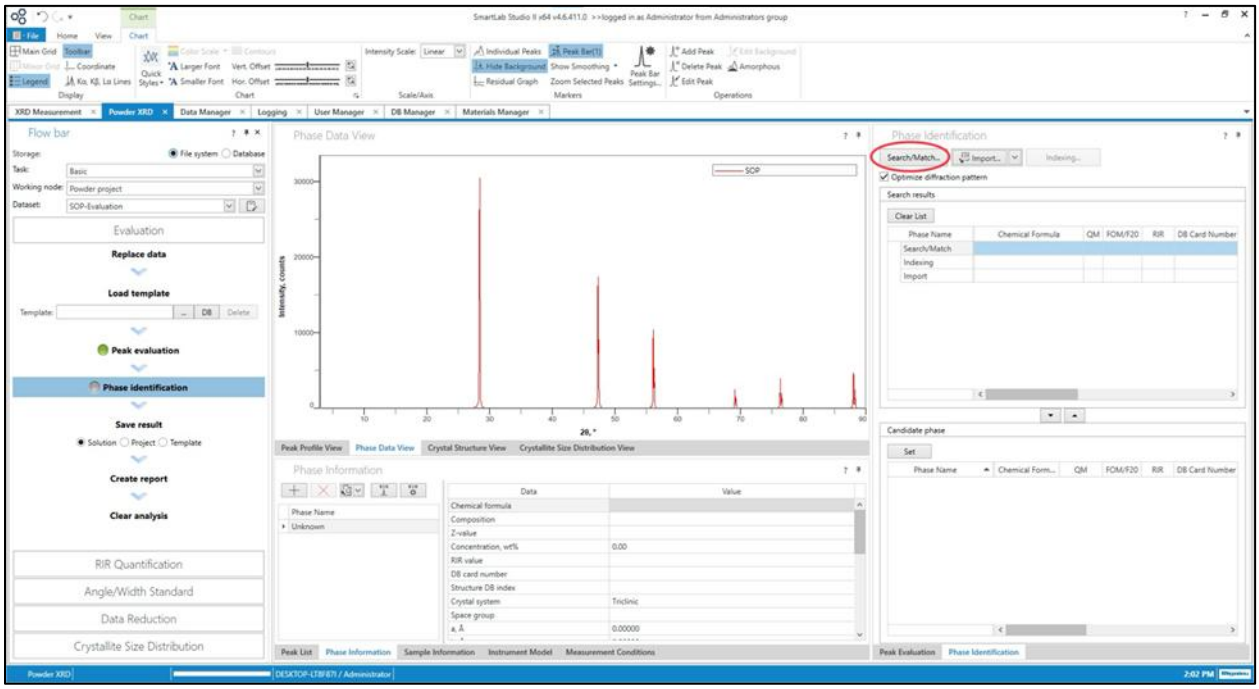
32. Once the data is loaded, the peak evaluation window should appear. The blue lines indicate calculated data, while the red indicates measured data.

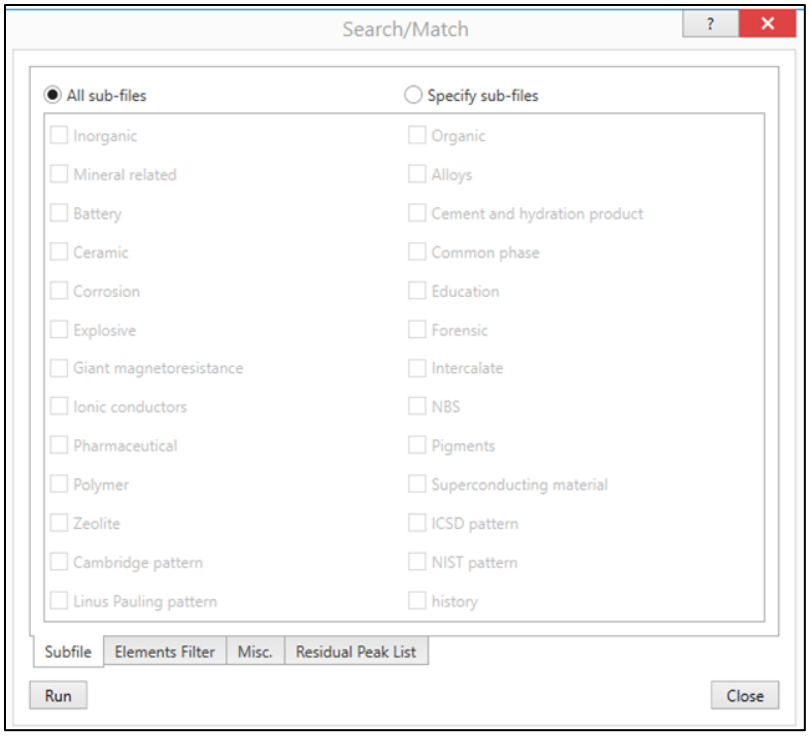


33. Depending on the measured data, it may be beneficial to change to a different intensity scale, which is available in the top taskbar.



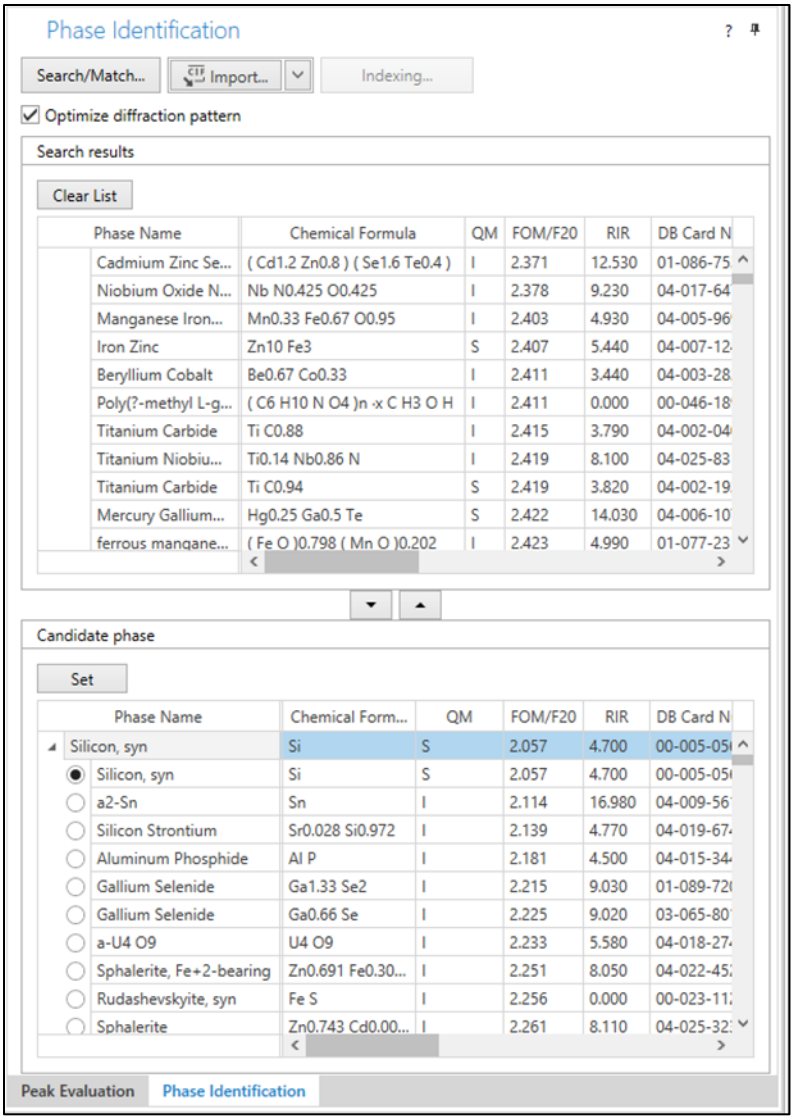
34. In the phase identification step, click Search/Match to start comparing and identifying peaks. This should open a new window with options to refine search criteria and select databases to search within.
    a. Select the “All sub-files” option, and click “Run”.

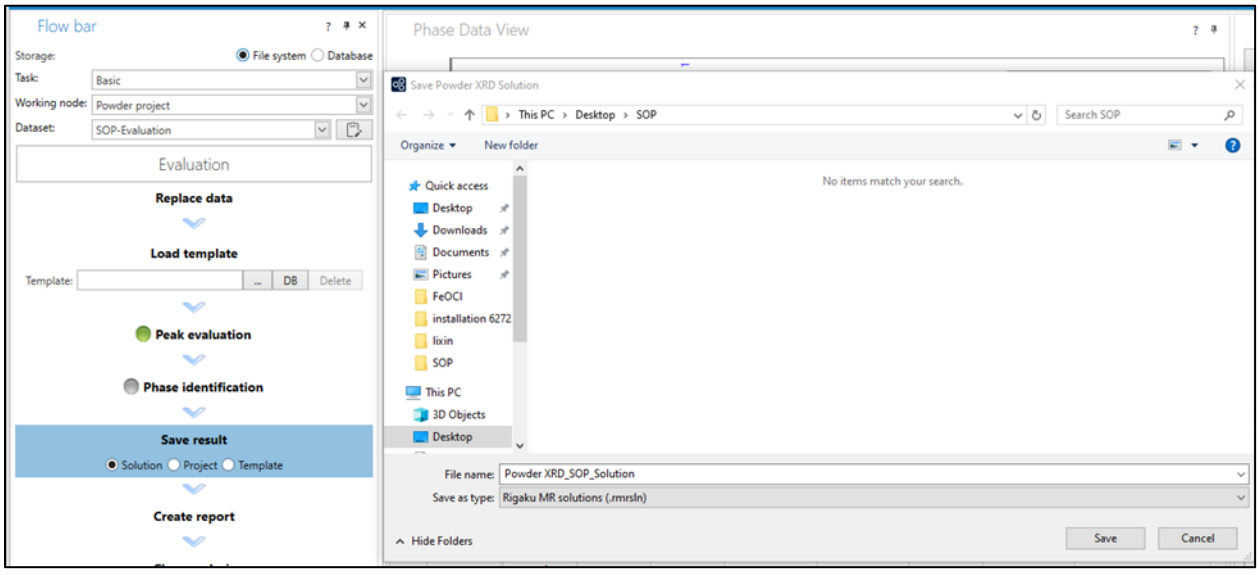




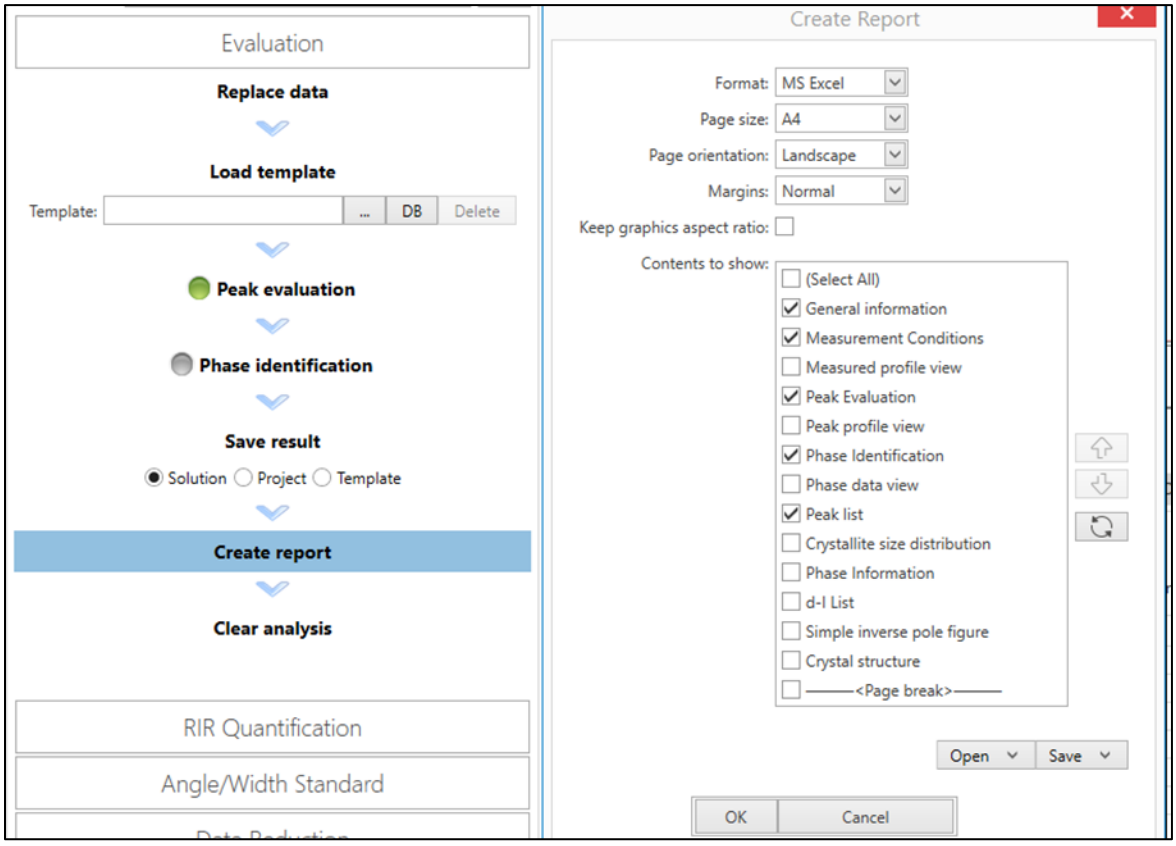
b. If you already know what your sample is comprised of, the search can be narrowed by elements in the "Elements Filter" tab, which allows you to choose which elements to include, not include, or include one at least.
c. The "Misc." tab allows you to select which database to be searched. Multiple databases cannot be searched simultaneously.

35. Once the initial search/match is conducted, the right sidebar should give a list of search results in the upper window, and the best candidate phase matches in the lower window.
    a. Candidate phase entries can be expanded by clicking the triangle in the leftmost column.
    b. Double clicking on candidates opens up the material's card information.
    c. Lower FOM values indicate a closer match.


Phase Identification
Search/Match...
Import...
Indexing...
Optimize diffraction pattern
Search results
Clear List
Phase Name
Chemical Formula
QM
FOM/F20
RIR
DB Card N
Cadmium Zinc Se...
(Cd1.2 Zn0.8) (Se1.6 Te0.4)
I
2.371
12.530
01-086-75
Niobium Oxide N...
Nb N0.425 O0.425
I
2.378
9.230
04-017-64
Manganese Iron...
Mn0.33 Fe0.67 O0.95
I
2.403
4.930
04-005-96
Iron Zinc
Zn10 Fe3
S
2.407
5.440
04-007-12
Beryllium Cobalt
Be0.67 Co0.33
I
2.411
3.440
04-003-28
Poly(?-methyl L-g...
(C6 H10 N O4)n -x C H3 O H
I
2.411
0.000
00-046-18
Titanium Carbide
Ti C0.88
I
2.415
3.790
04-002-04
Titanium Niobiu...
Ti0.14 Nb0.86 N
I
2.419
8.100
04-025-83
Titanium Carbide
Ti C0.94
S
2.419
3.820
04-002-19
Mercury Gallium...
Hg0.25 Ga0.5 Te
S
2.422
14.030
04-006-10
ferrous manganе...
(Fe O)0.798 (Mn O)0.202
I
2.423
4.990
01-077-23
Candidate phase
Set
Phase Name
Chemical Form...
QM
FOM/F20
RIR
DB Card N
Silicon, syn
Si
S
2.057
4.700
00-005-05
Silicon, syn
Si
S
2.057
4.700
00-005-05
a2-Sn
Sn
I
2.114
16.980
04-009-56
Silicon Strontium
Sr0.028 Si0.972
I
2.139
4.770
04-019-67
Aluminum Phosphide
Al P
I
2.181
4.500
04-015-34
Gallium Selenide
Ga1.33 Se2
I
2.215
9.030
01-089-72
Gallium Selenide
Ga0.66 Se
I
2.225
9.020
03-065-80
a-U4 O9
U4 O9
I
2.233
5.580
04-018-27
Sphalerite, Fe+2-bearing
Zn0.691 Fe0.30...
I
2.251
8.050
04-022-45
Rudashevskyite, syn
Fe S
I
2.256
0.000
00-023-11
Sphalerite
Zn0.743 Cd0.00...
I
2.261
8.110
04-025-32
Peak Evaluation
Phase Identification


Flow bar
Storage:
File system
Database
Task:
Basic
Working node:
Powder project
Dataset:
SOP-Evaluation
Evaluation
Replace data
Load template
Template:
DB
Delete
Peak evaluation
Phase identification
Save result
Solution
Project
Template
Create report
Phase Data View
Save Powder XRD Solution
This PC
Desktop
SOP
Search SOP
Organize
New folder
Quick access
Desktop
Downloads
Documents
Pictures
FeOCl
installation 6272
lixin
SOP
This PC
3D Objects
Desktop
No items match your search.
File name:
Powder XRD_SOP_Solution
Save as type:
Rigaku MR solutions (.rmrsln)
Hide Folders
Save
Cancel

36. To save the completed phase identification data, go to the flow bar and click “Save result”, and then save to the computer as a solution.
37. The program also has the option to create a report, with multiple format options. If creating a report, the .csv filetype is most recommended.



4.7. Turning off the X-ray

38. Switch back to the XRD measurement tab.
39. In the same startup/shutdown window that was used to turn on the X-ray, there is a column to the right of the startup options, which is titled “Shutdown”. Select the “XG off” option and click “Run”.
40. Once the X-ray shutdown has completed, the “X-ray on” light (orange) on top of the machine should turn to transparent.

4.8. Taking the sample out and turning off the equipment

41. After confirming that X-rays are stopped, shut down SmartLab Studio II software.
42. **WAIT AT LEAST 3 MINUTES** after X-ray generation stopped, and take out your sample.
43. Press the Power Off button (white one) on the front side of Miniflex.
44. Turn the HV enable key to the vertical position.