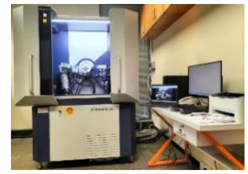
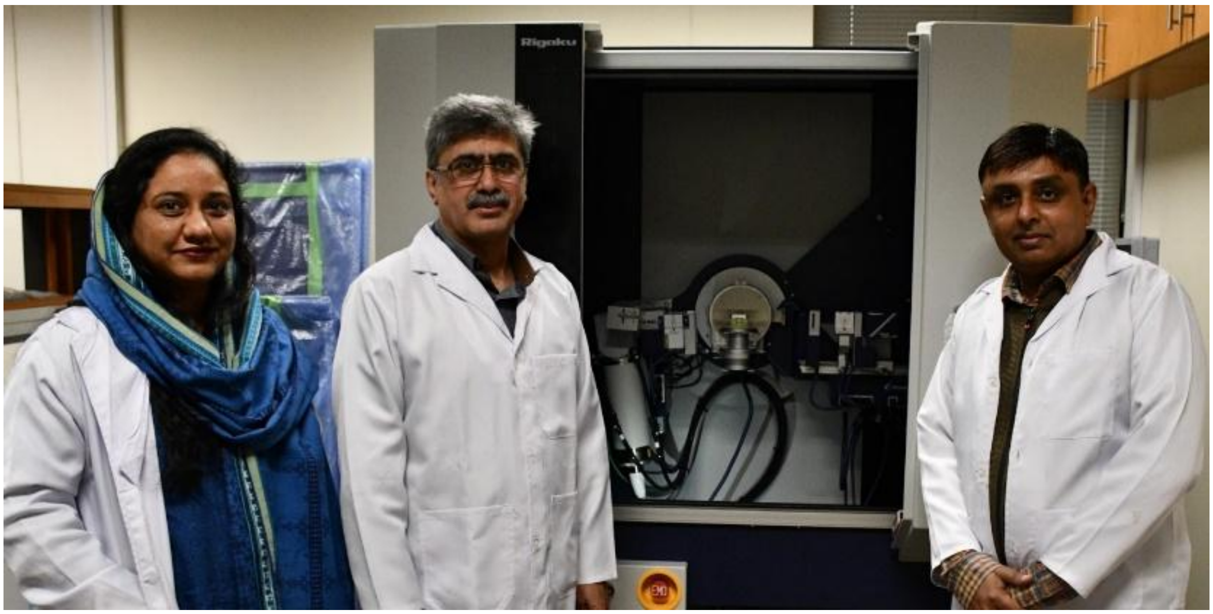


Step by Step Operational Manual of the SmartLab SE



Dr. Ghulam Sarwar Butt, Dr. Sabieh Anwar,

https://physlab.org/, Physical Properties Measurements Lab,

CentralLab, Department of Physics, LUMS

Contents

Turning ON SmartLab SE

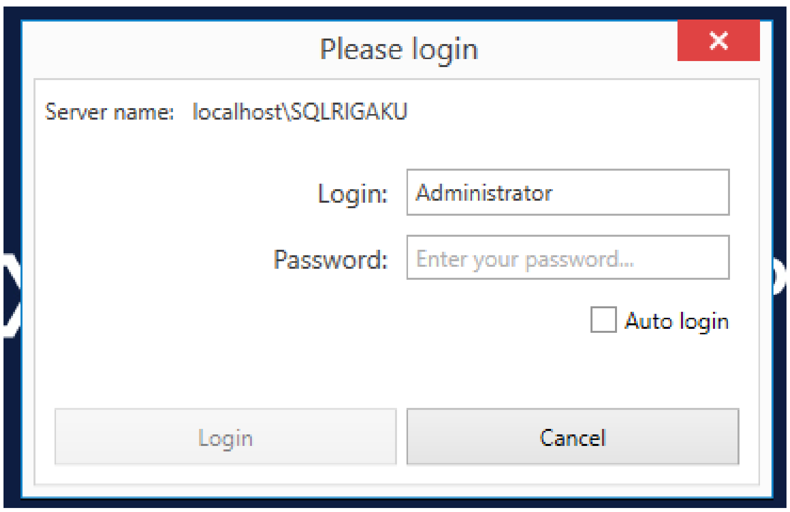
1. Turn ON CPU
2. Turn ON the circuit breaker clockwise on the rear side of the SmartLab SE XRD.

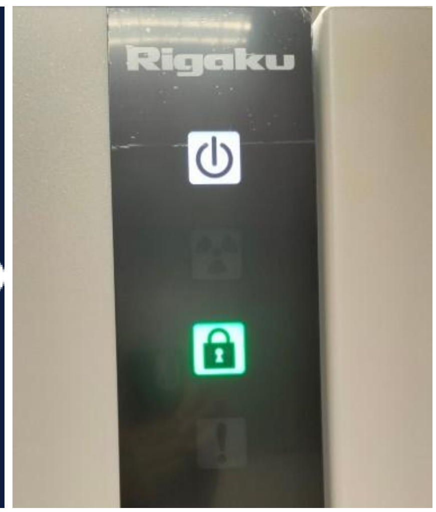
3. Insert the power key into the front panel of XRD. Turn the power key clockwise.

4. Power ON indicator turn ON

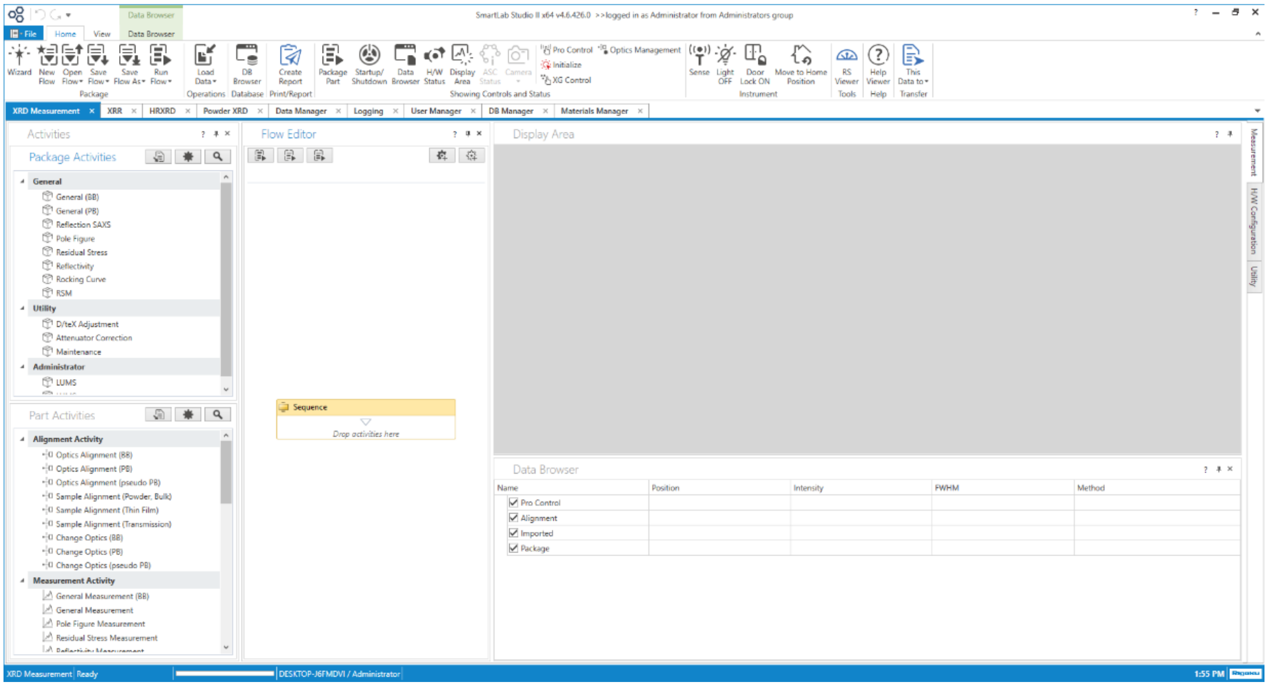
5. Double Click Smart Lab Studio II shortcut on desktop.

6. Enter password xxxxxx. (Tick sound after login. Door is auto locked).

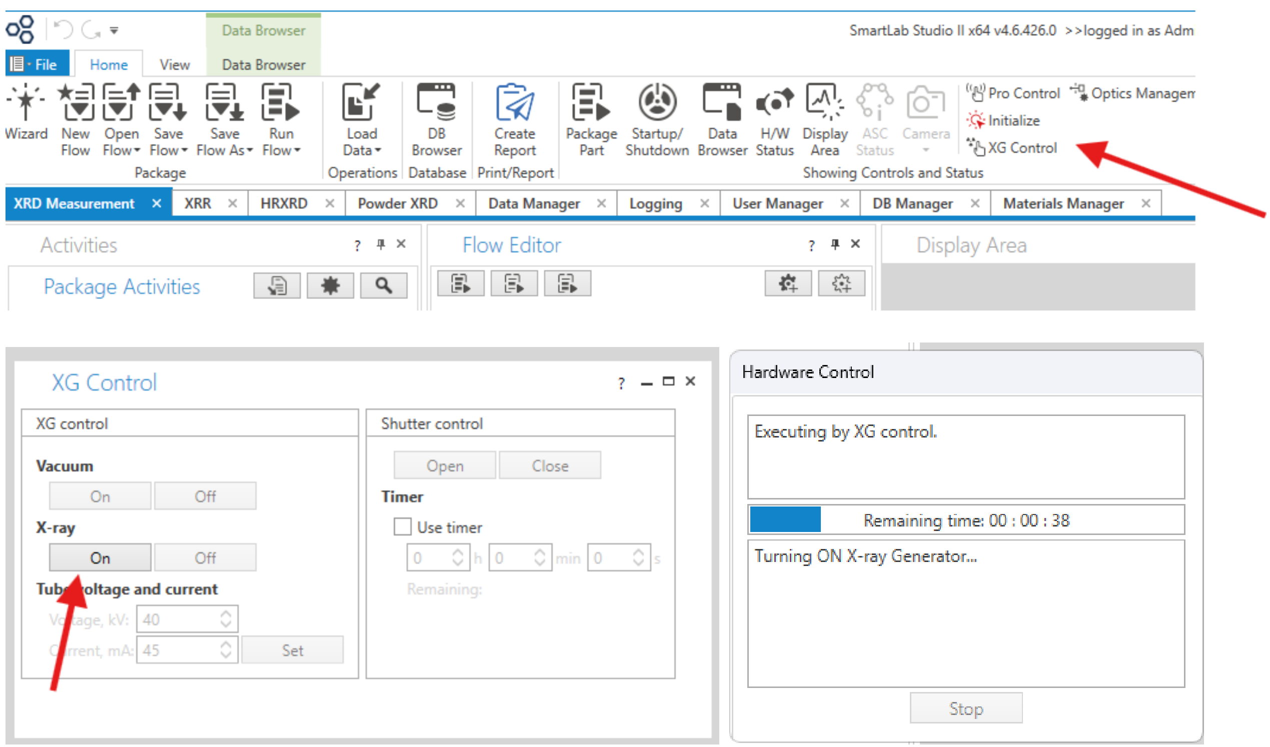




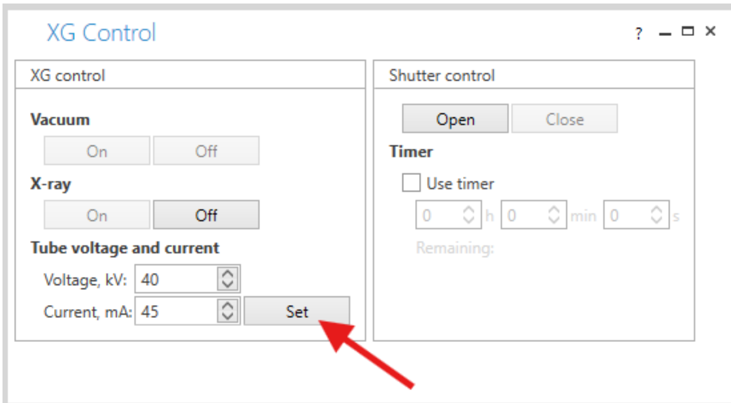
7. See the bottom left; do nothing unless you see “Ready”.

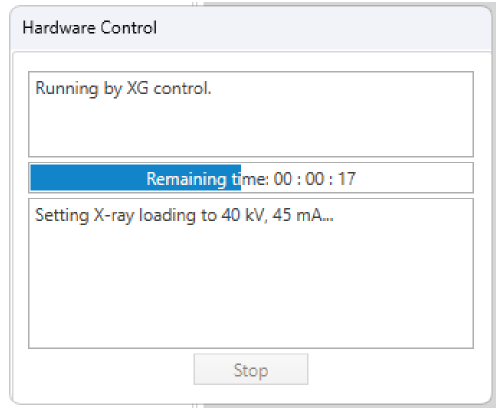


8. When the status is ready, then press XG control and press “ON”.



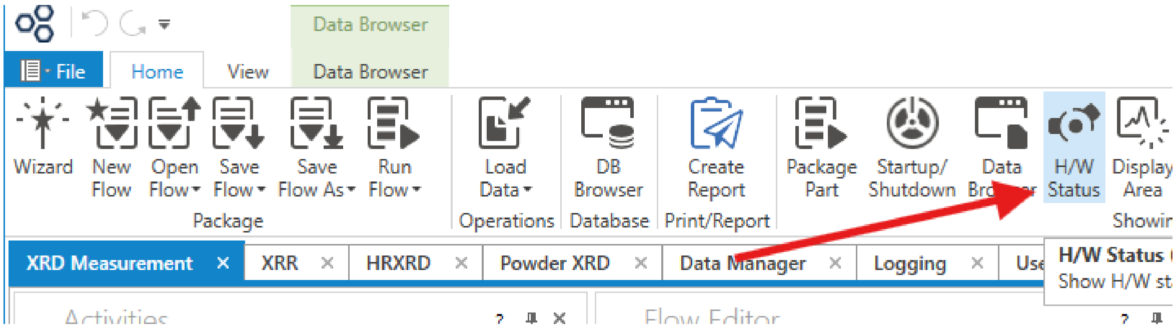
9. Chiller will turn on automatically; do nothing till the status changes to ready.
10. Press set on XG Control window for the Default Settings as
a. Voltage KV 40
b. Current mA 45

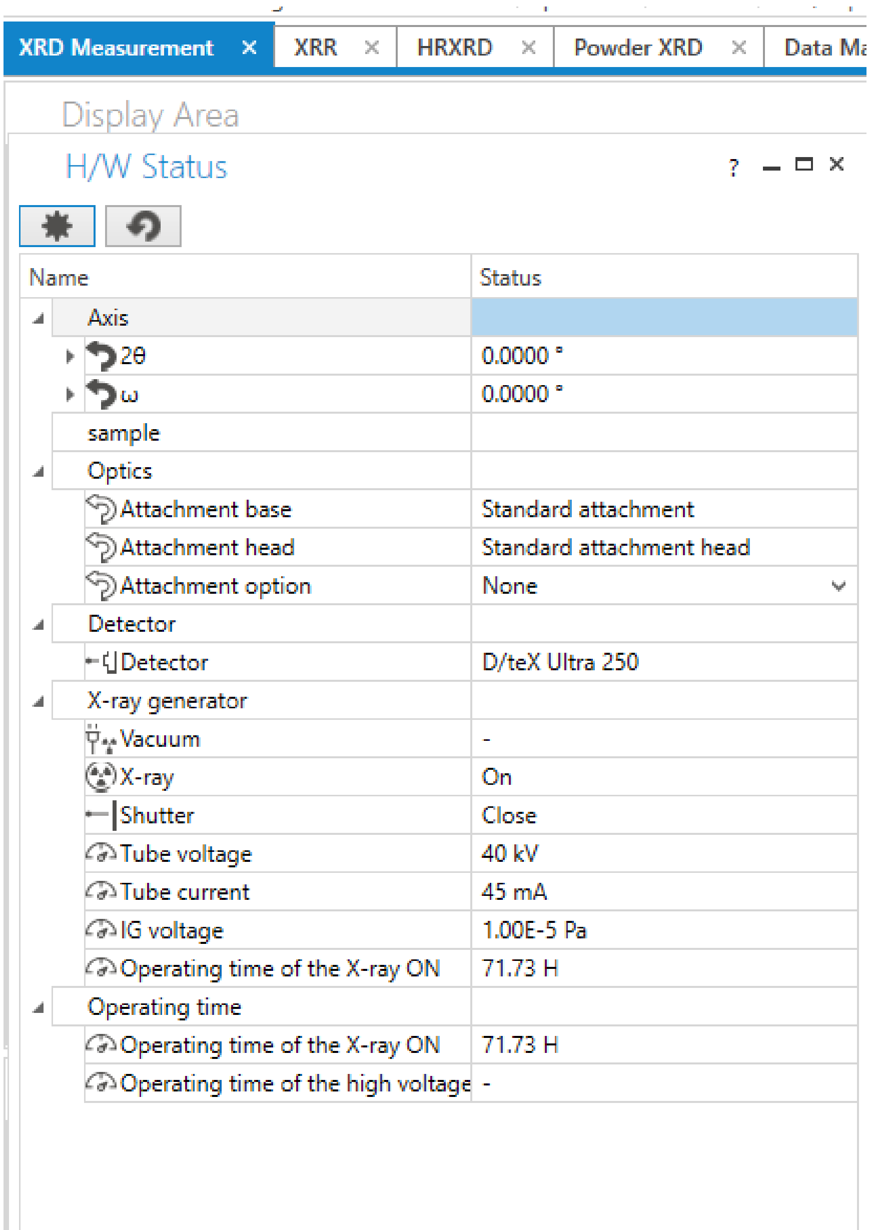




11. When X-RAYS generator turns ON, the X-RAYS lamps and X-RAYS ON indicator will turn ON. The X-RAYS lamps are at the left and right locations.

12. Press H/W Status to confirm the set values of voltage and current.

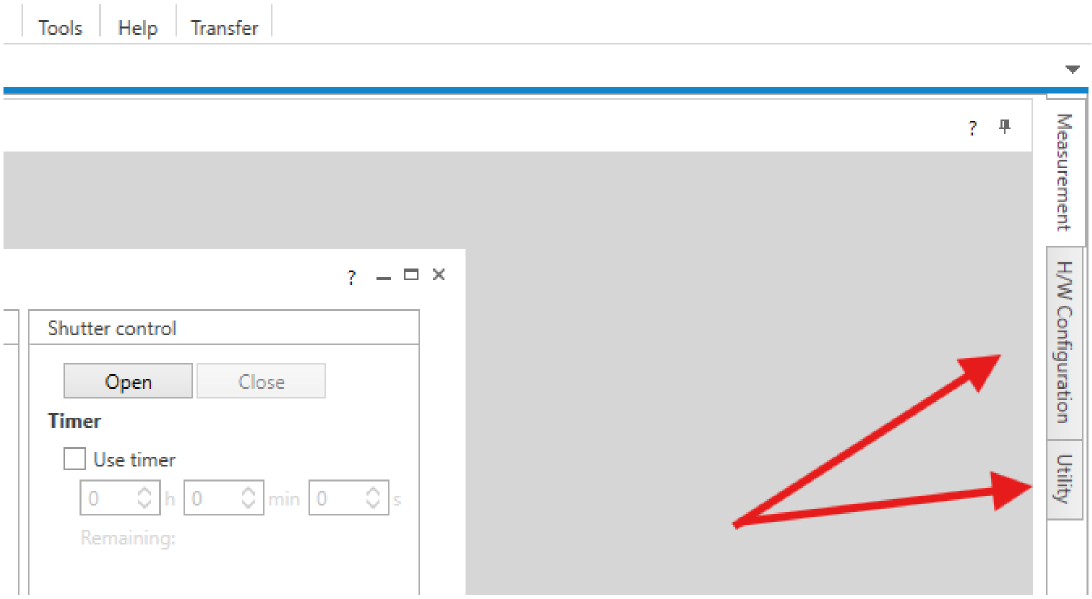




Warning!!

a. Do not Click H/W configuration.

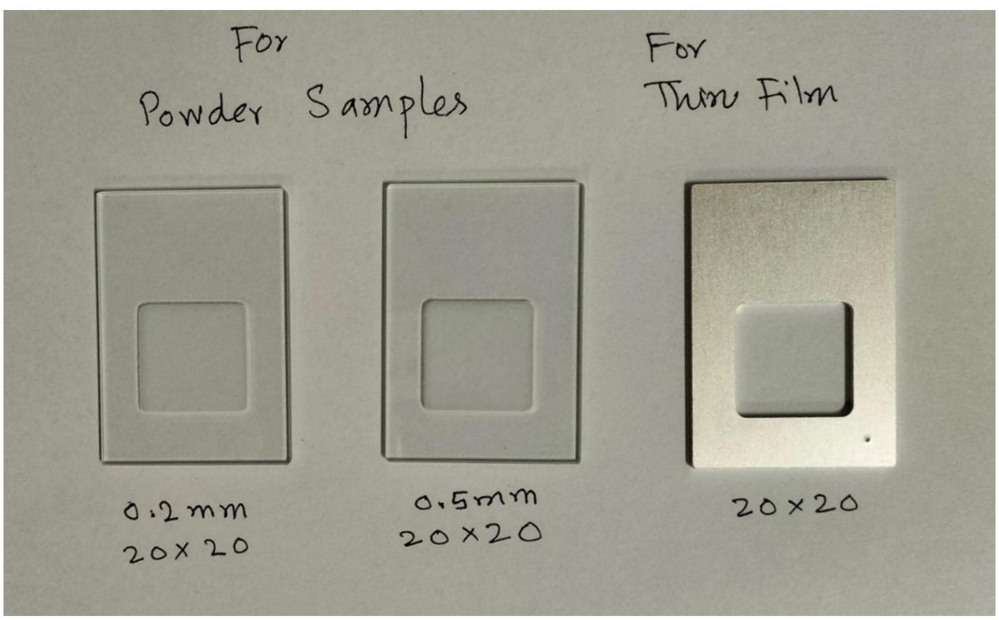
b. Do not click Utility.
c. Do not change Hardware Assembly

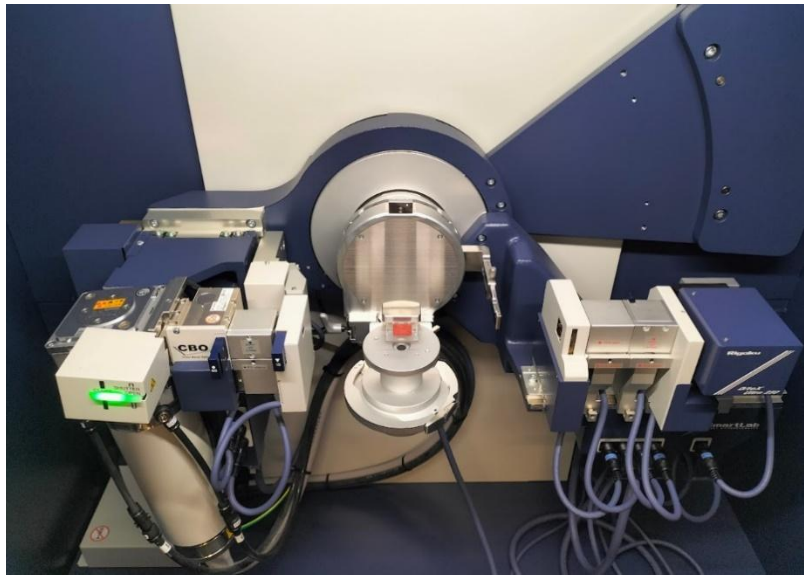


Now SmartLab SE Instrument is ready for measurements.

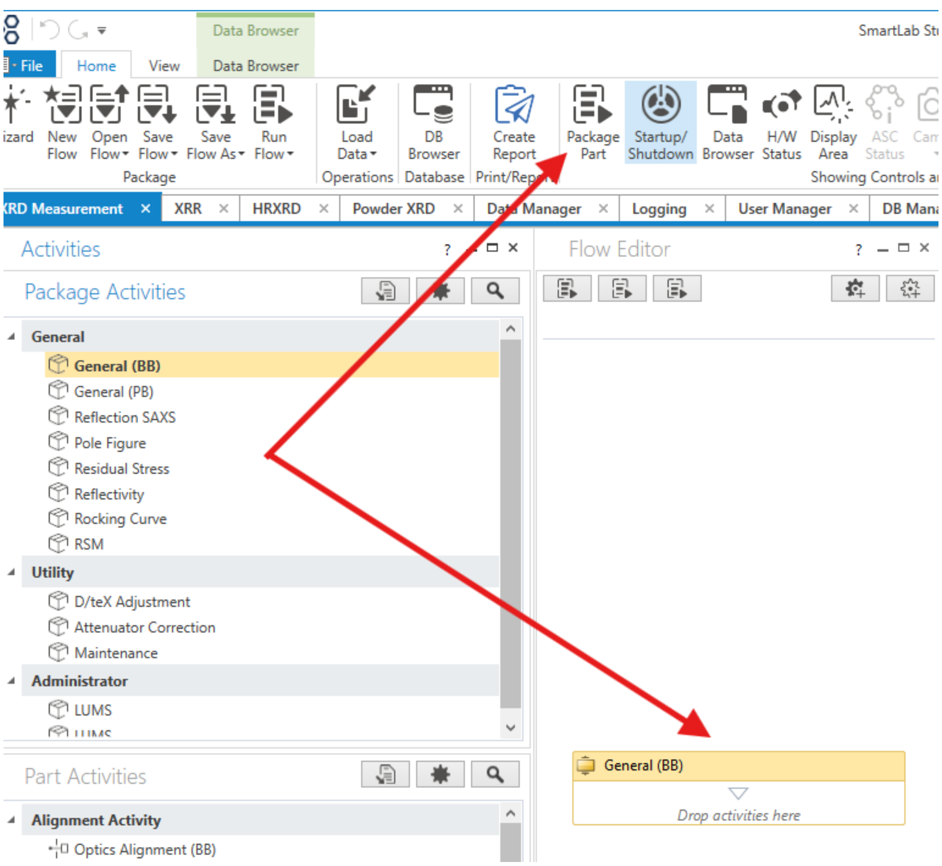
Taking measurements of powder / thin film

1. Prepare and Attach sample

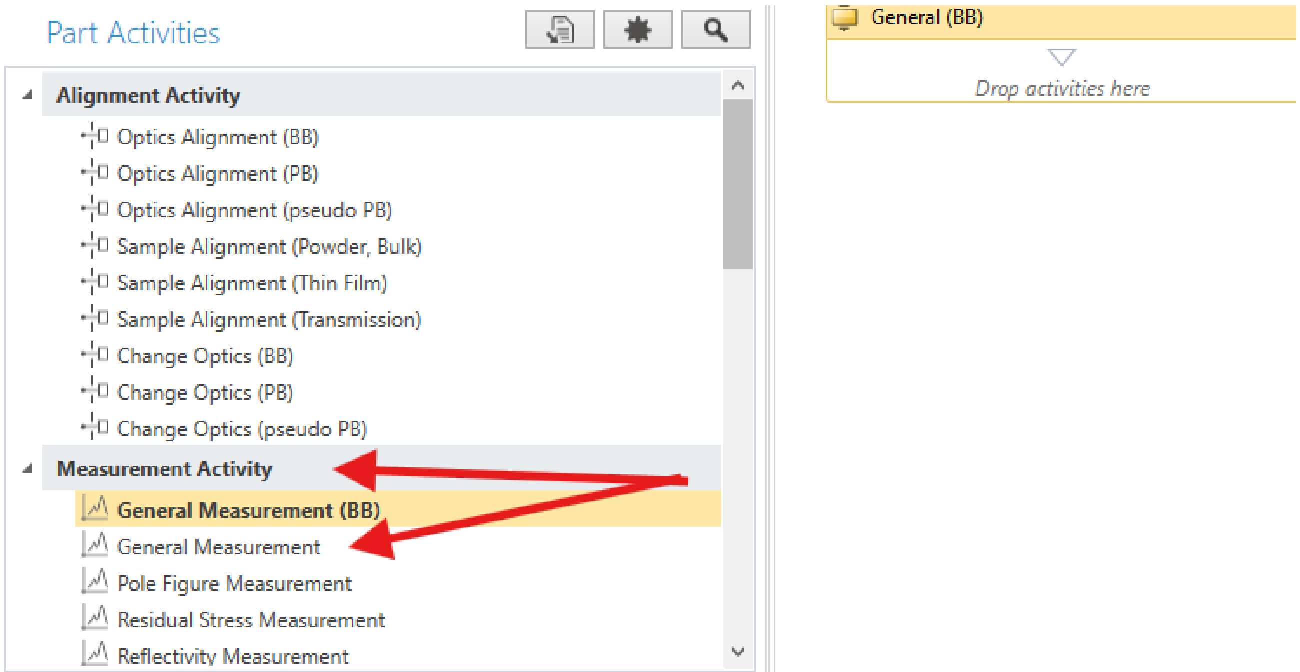


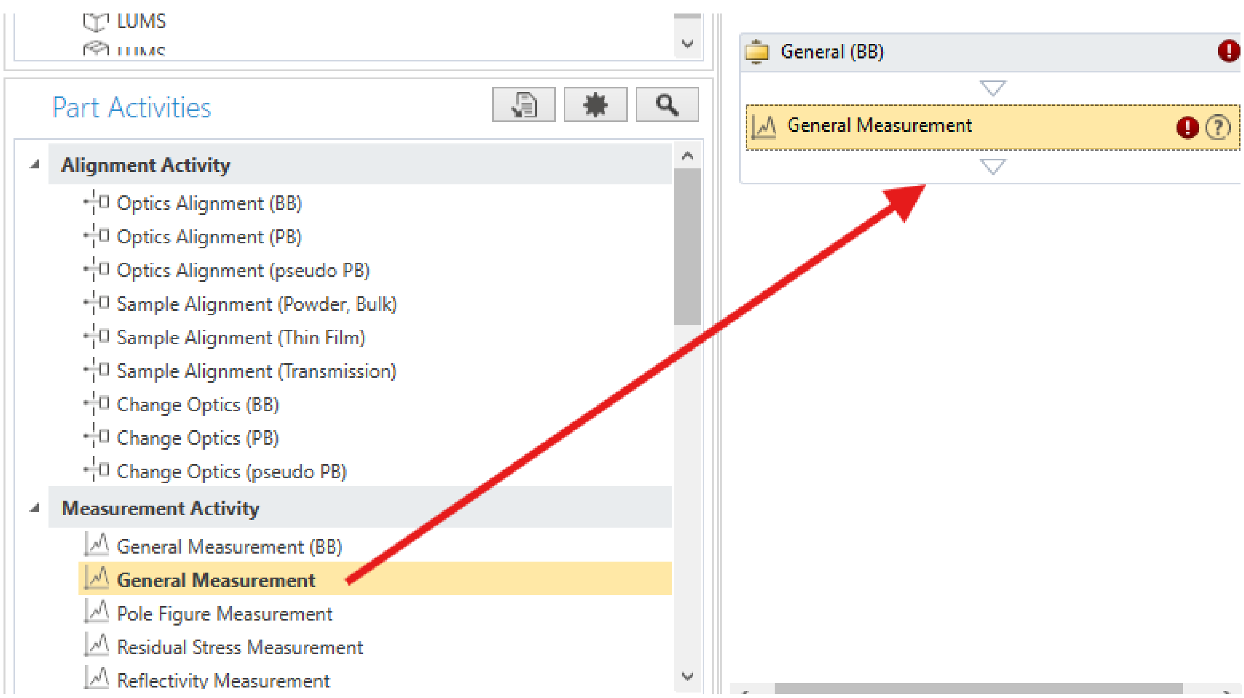


2. Click package part to run the Flow Editor

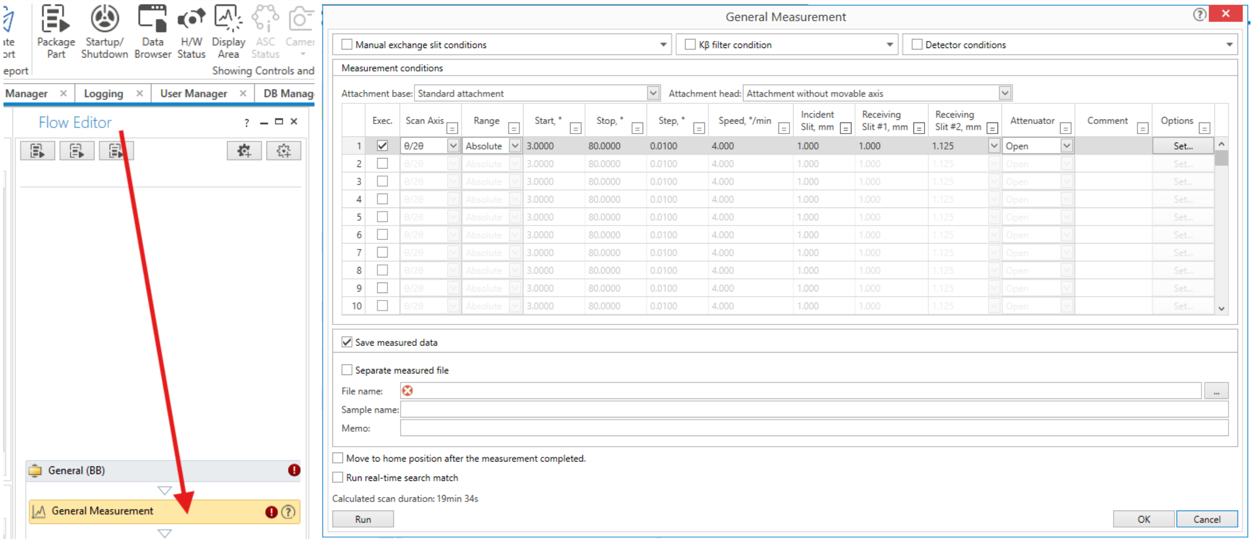


3. Double Click General Measurement under Measurement Activity

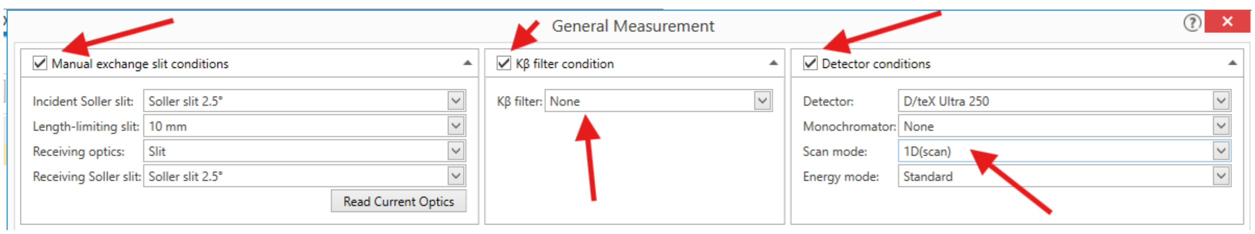




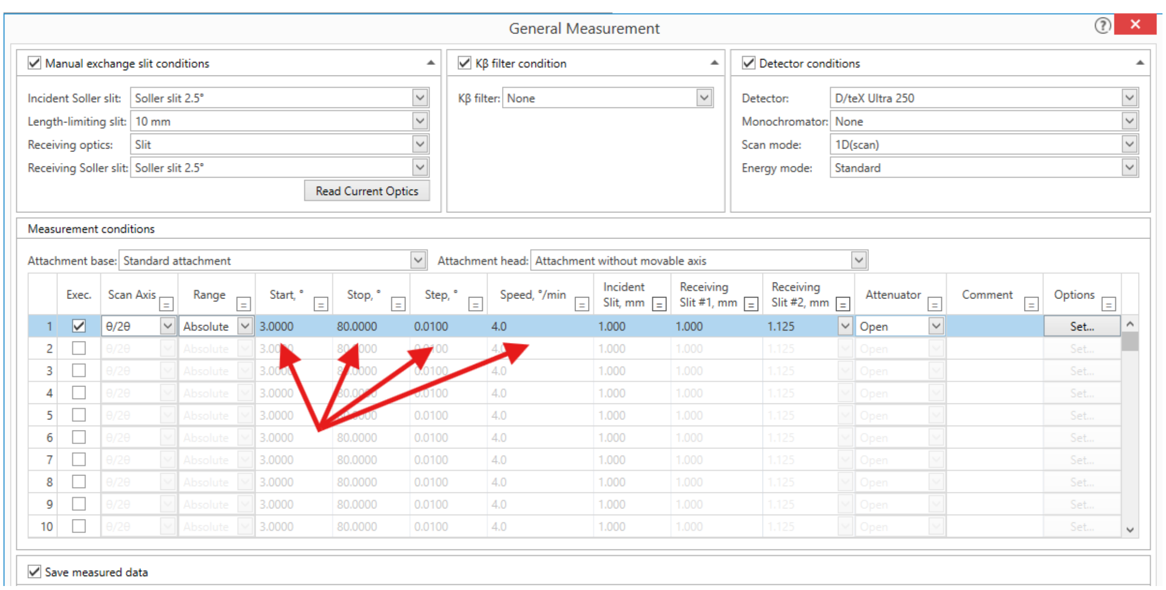
4. Now click General Measurement under the Flow Editor, new window of general measurement will appear.



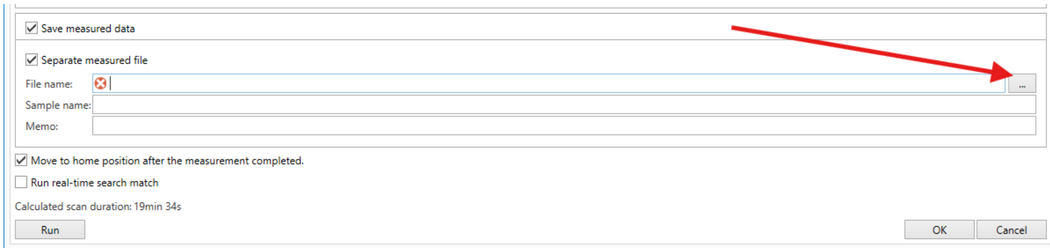
5. Check the Manual exchange condition, Kβ filter condition and detector conditions. Select the Kβ filter condition to none and detector conditions to 1D(scan).

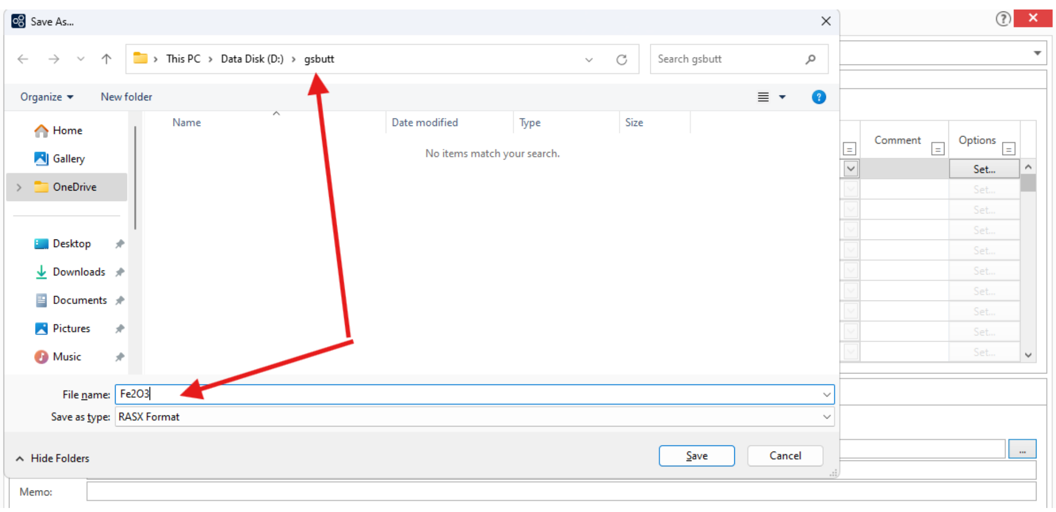


6. Set the scan conditions for start, stop angle, step size and speed. The standard values are given in the picture below and press set.

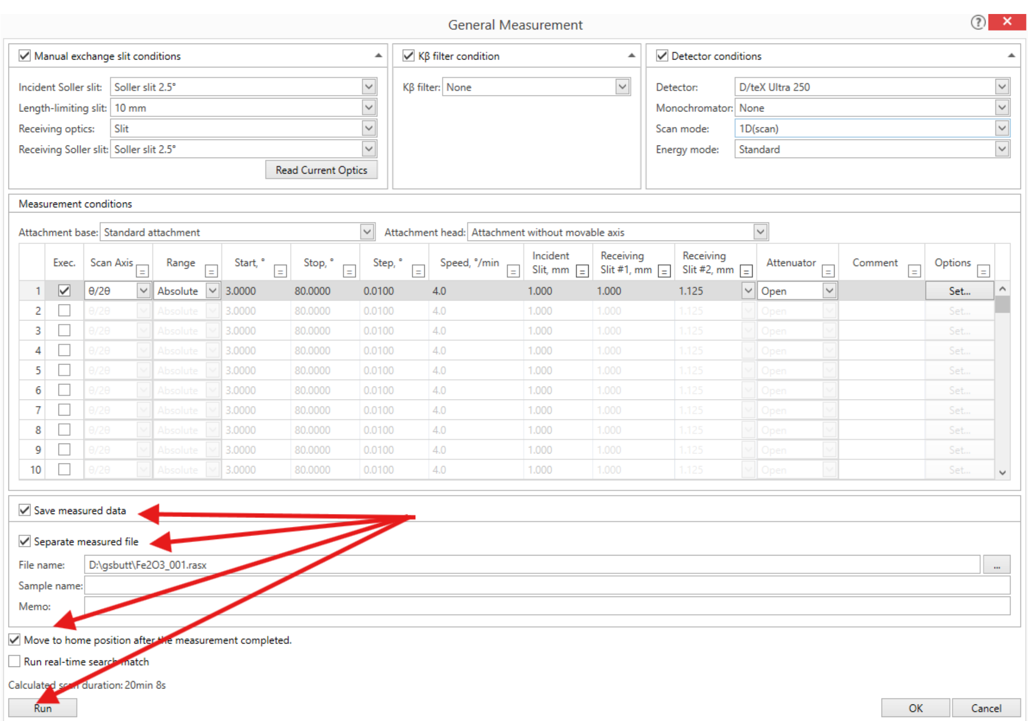


7. Click three dots as given in figure below and select the folder where the data file is to be saved, enter the sample name and press save.

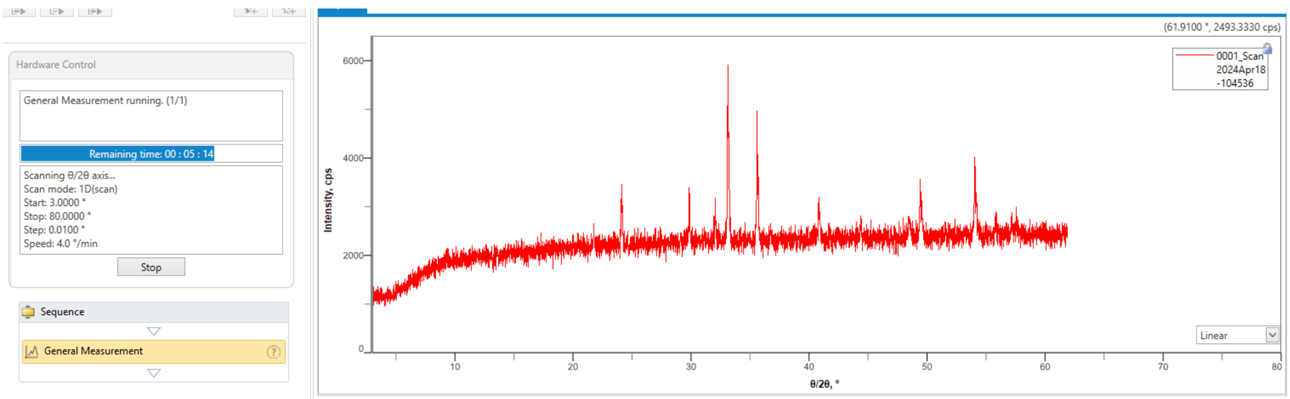




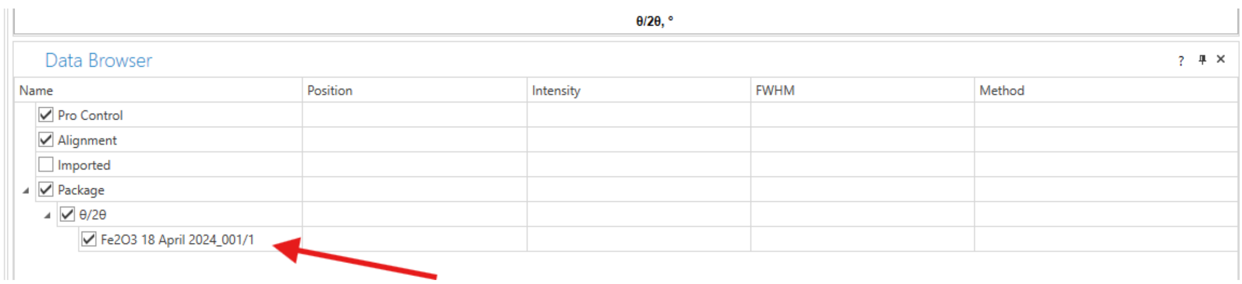
8. Check all the boxes mentioned with red arrows and Press RUN.



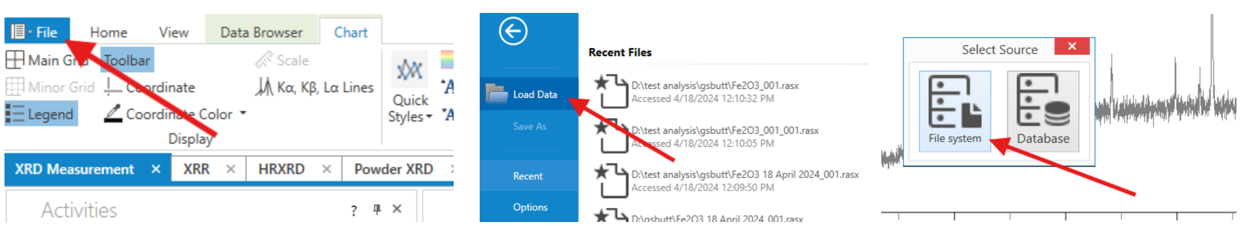
9. Measurements will start.

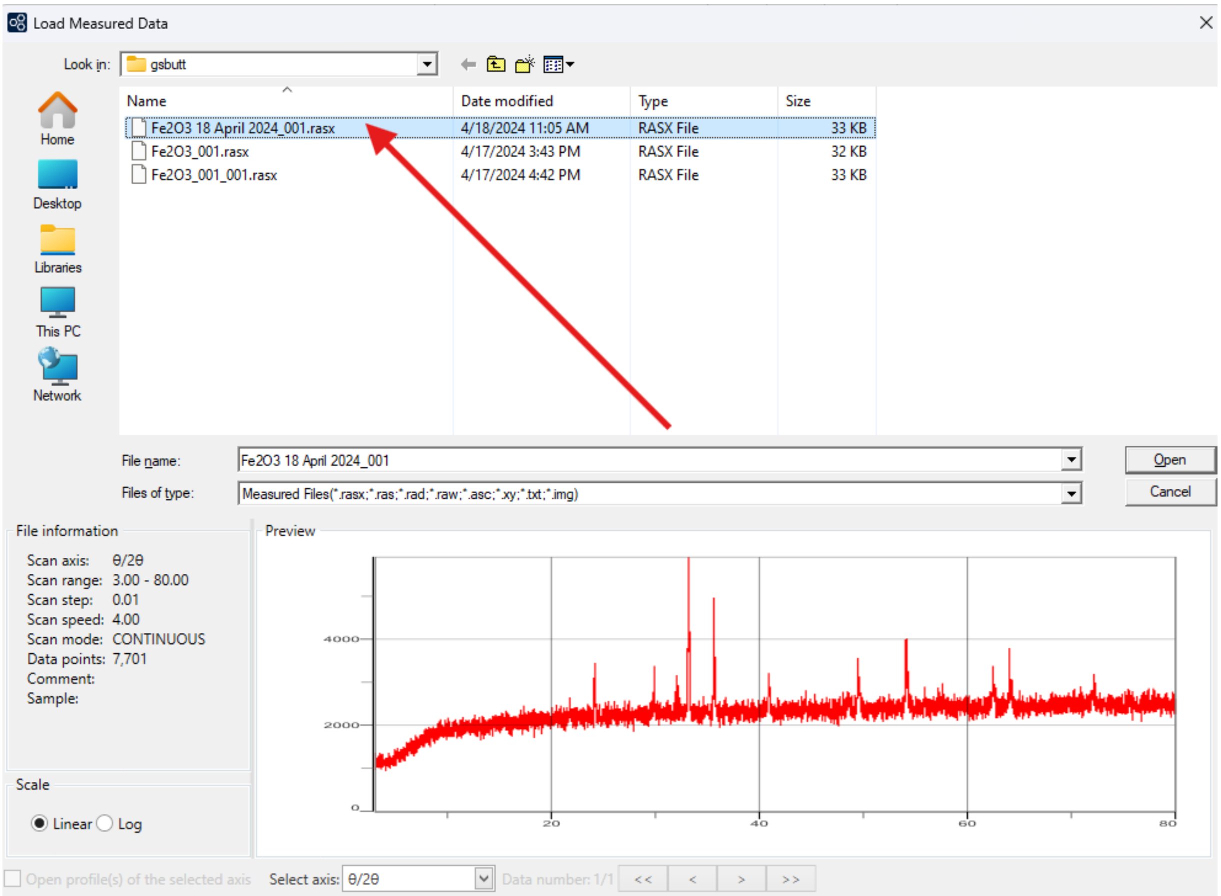


10. Data file of the format .rasx will be saved.

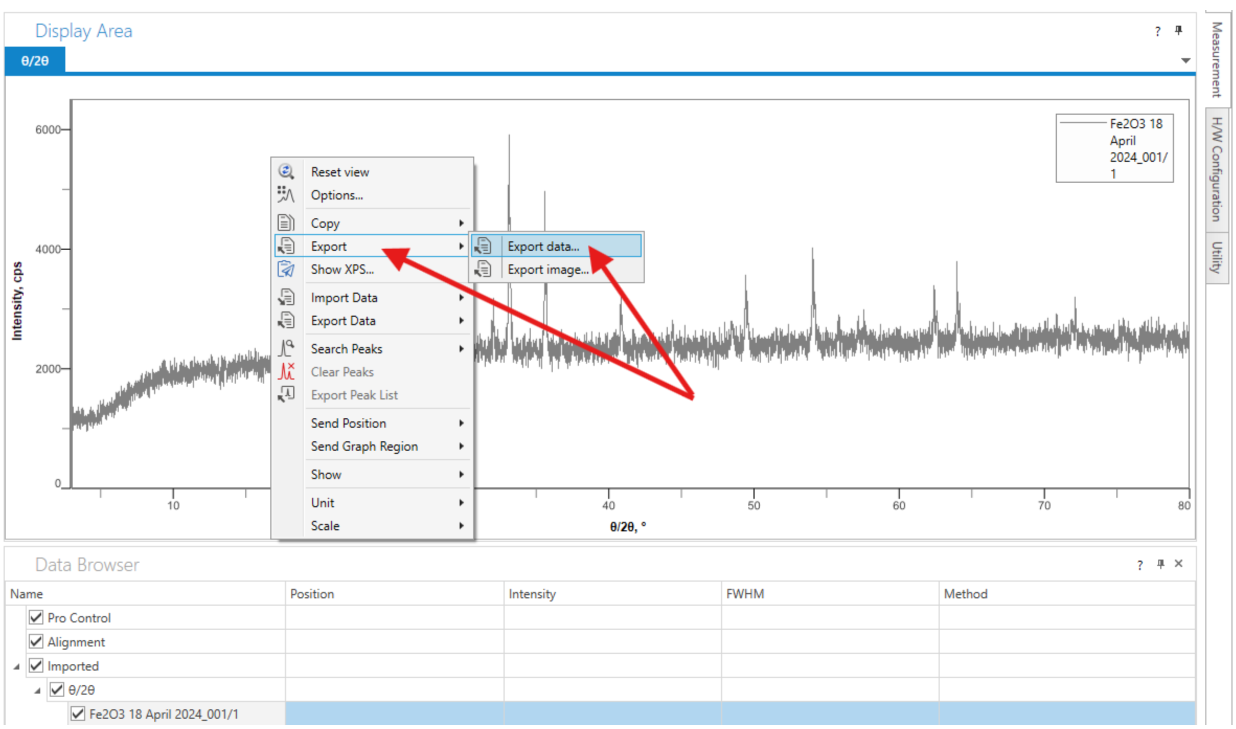


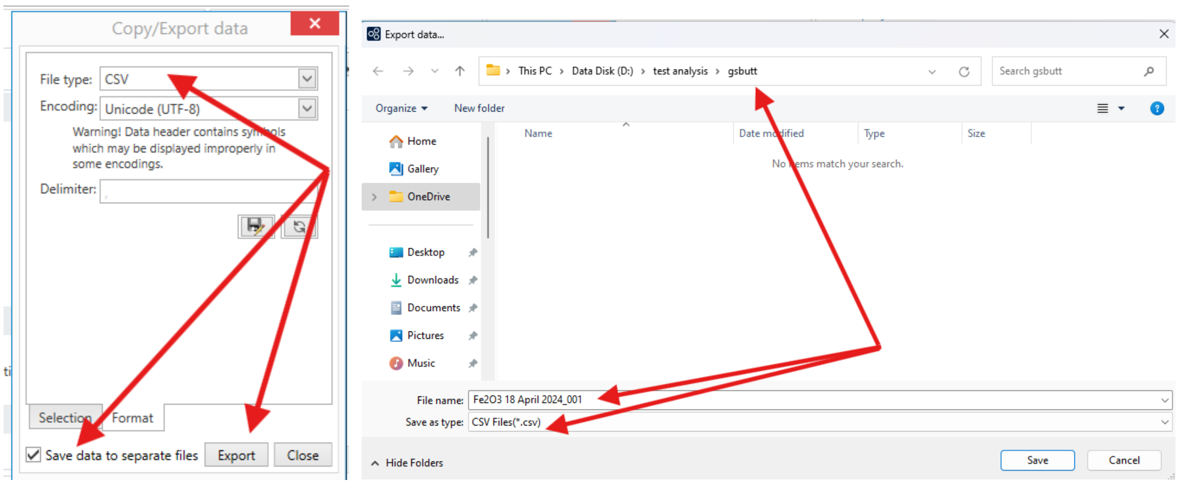
11. To load the .rasx data file, press File → Load Data → File System and then select file from the location.



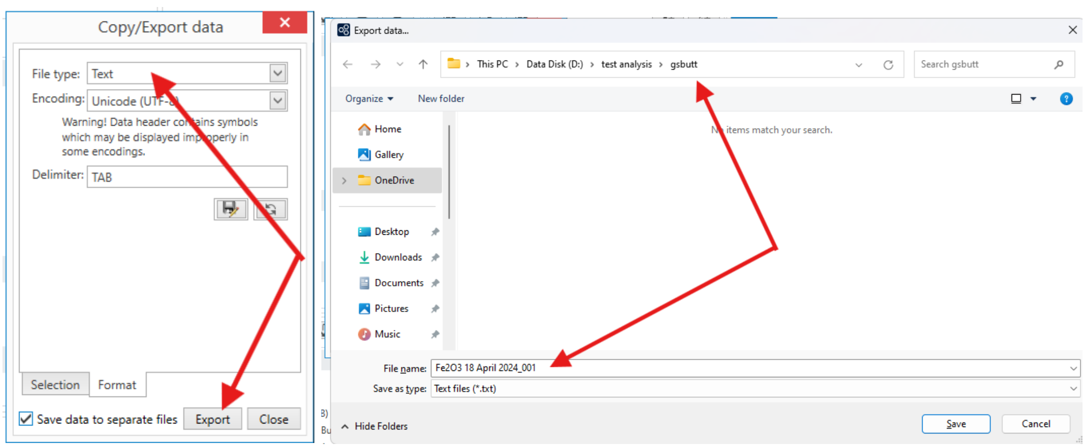


12. Exporting data as .csv

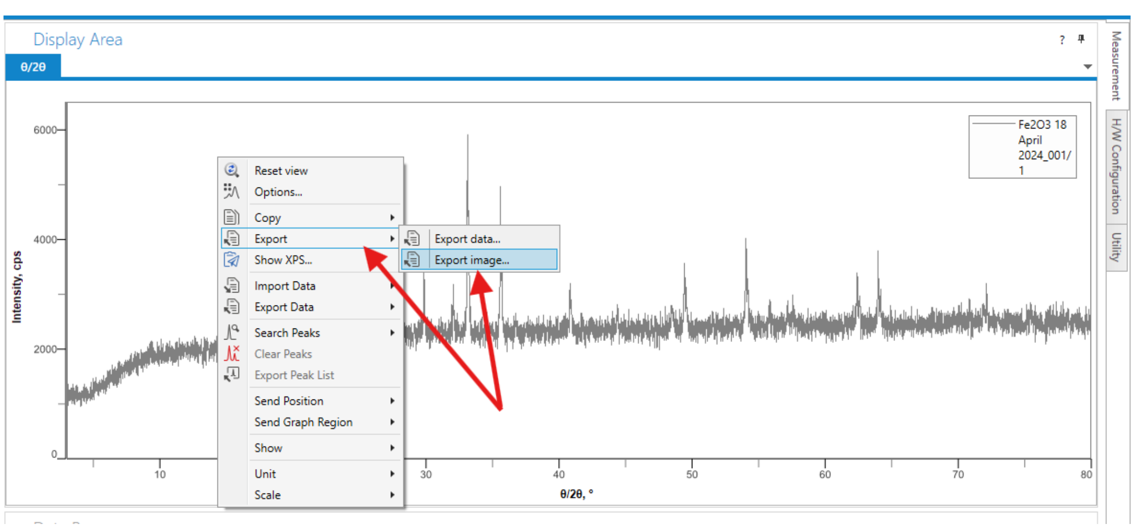


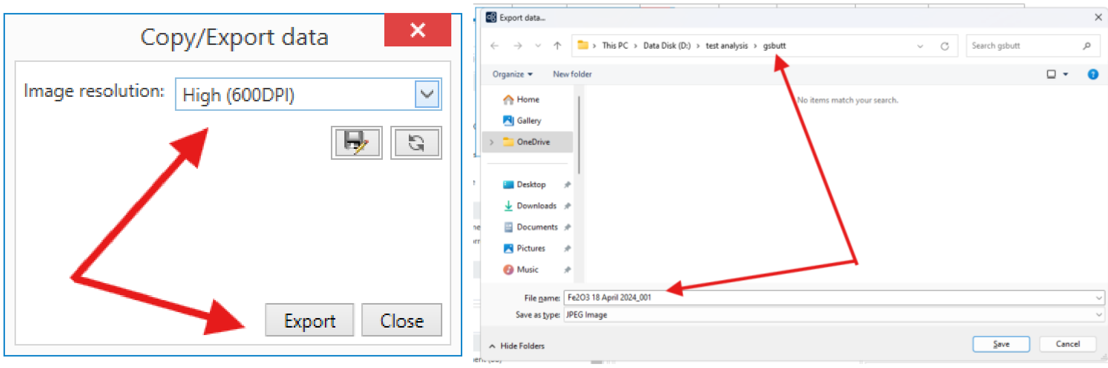


13. Exporting data as text

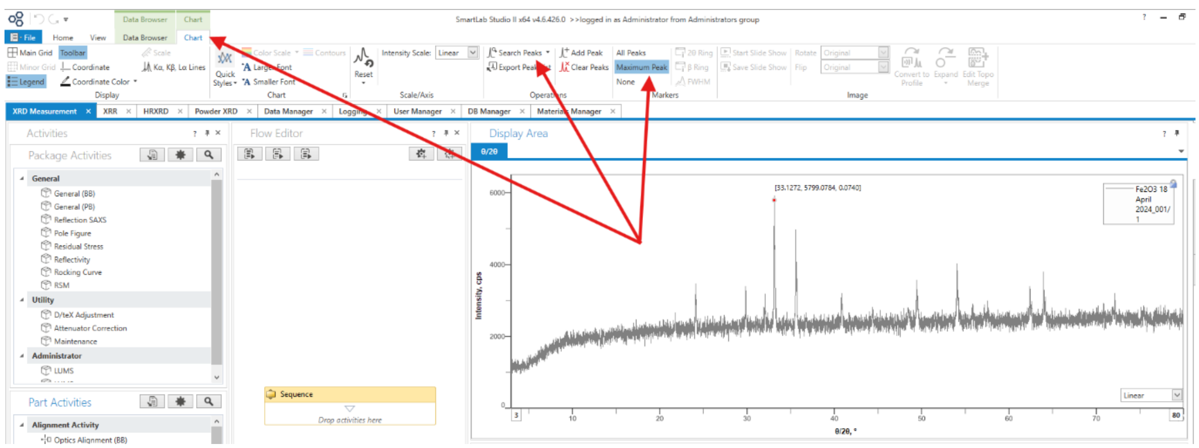


14. Exporting chart image

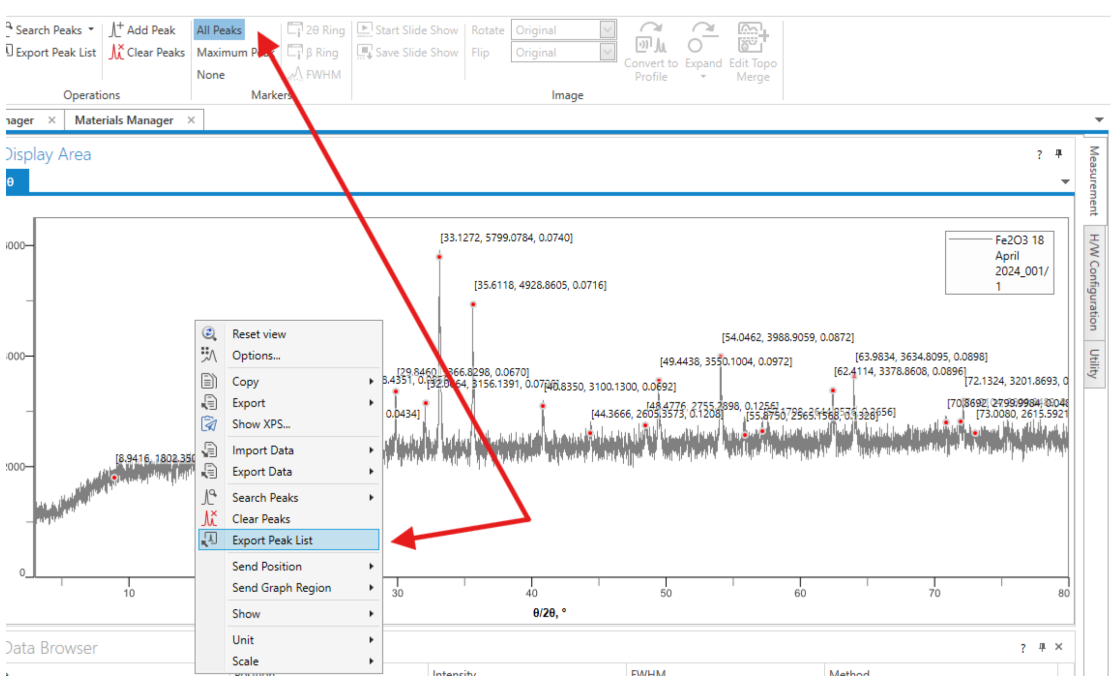


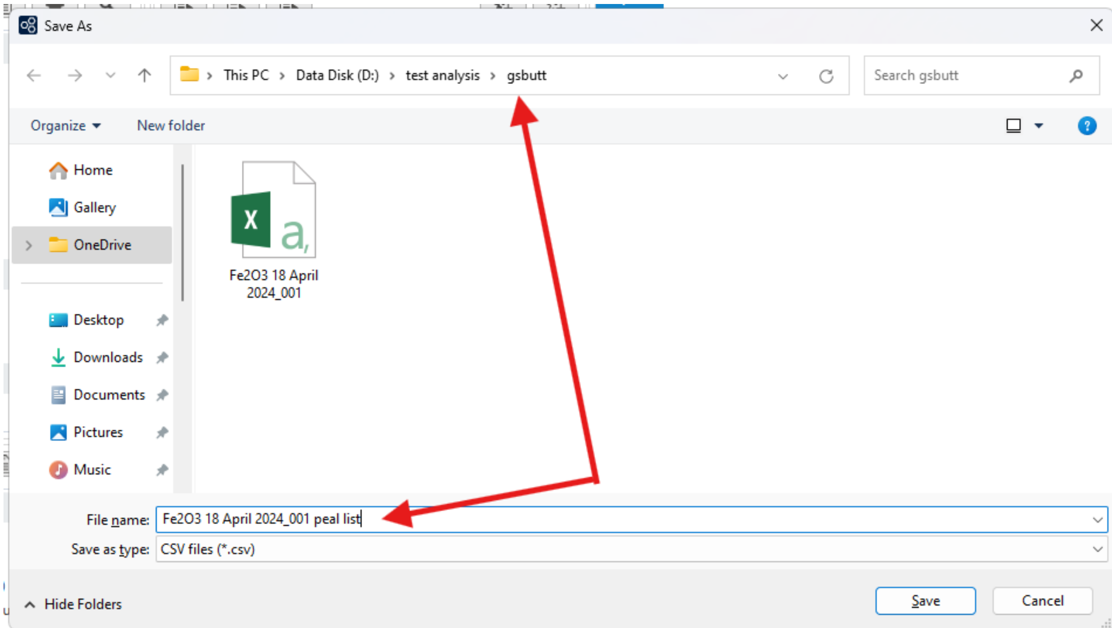


15. Peak Search (maximum/all peaks)

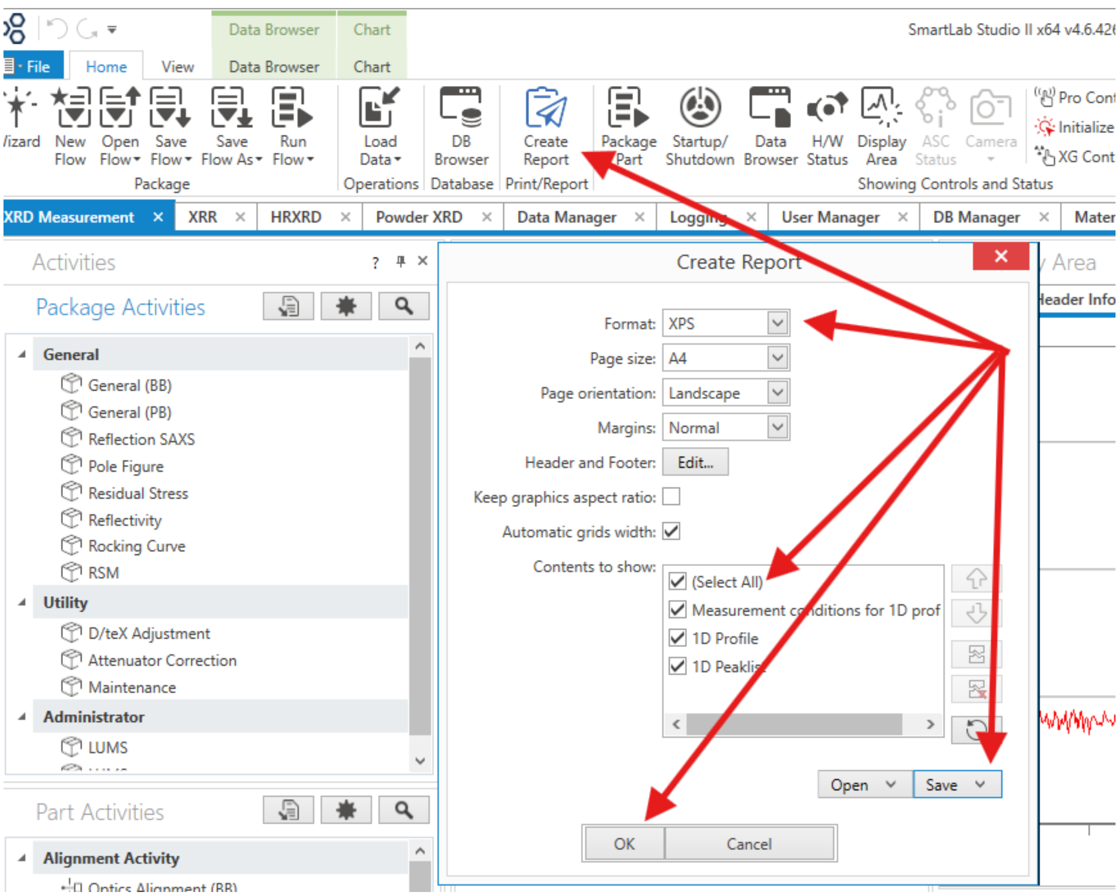


16. Export peak list (Axis, Position, Intensity, FWHM).



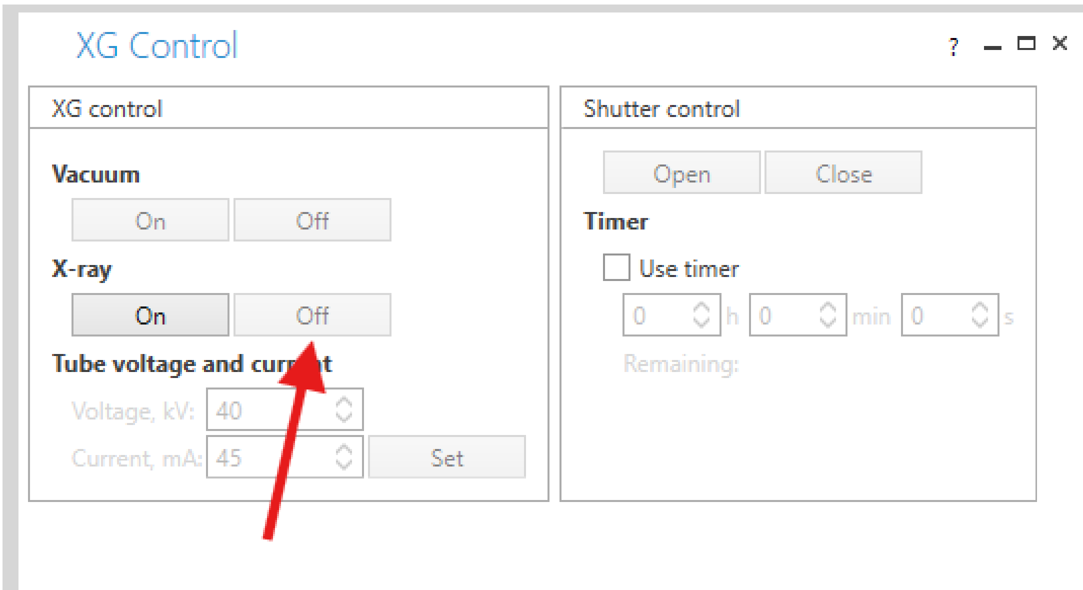


17. Create Report

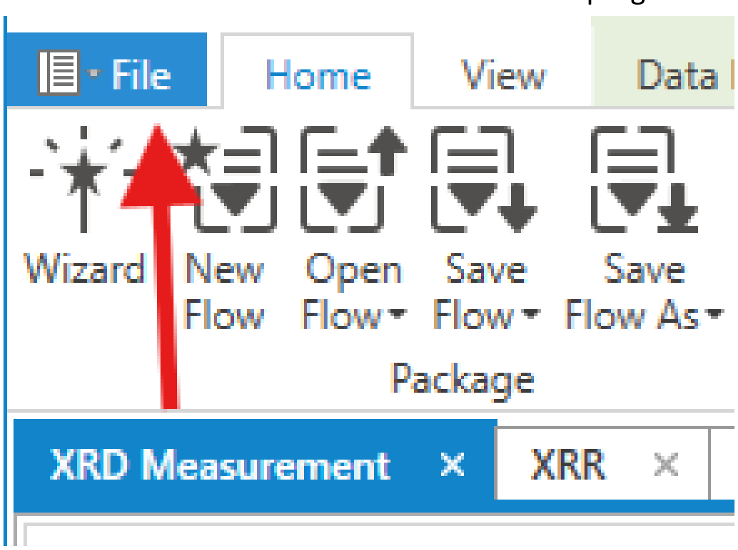


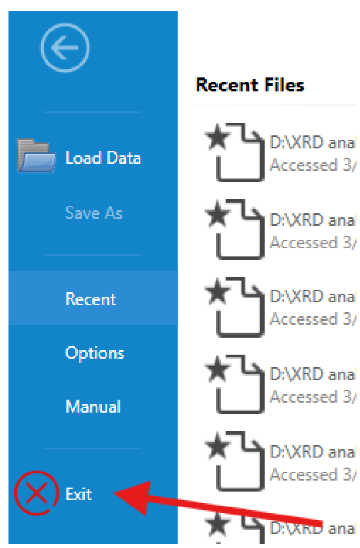
Turning OFF SmartLab SE

1. Confirm that measurements are completed. Press XG Control (Home XG control) and press OFF wait till chiller Turn OFF (5 min approximately). When the generator turns OFF, the X-RAYS ON lamp and the X-RAYS ON indicator will turn OFF.



2. Shut down SmartLab Studio II program. Press File and then Exit.

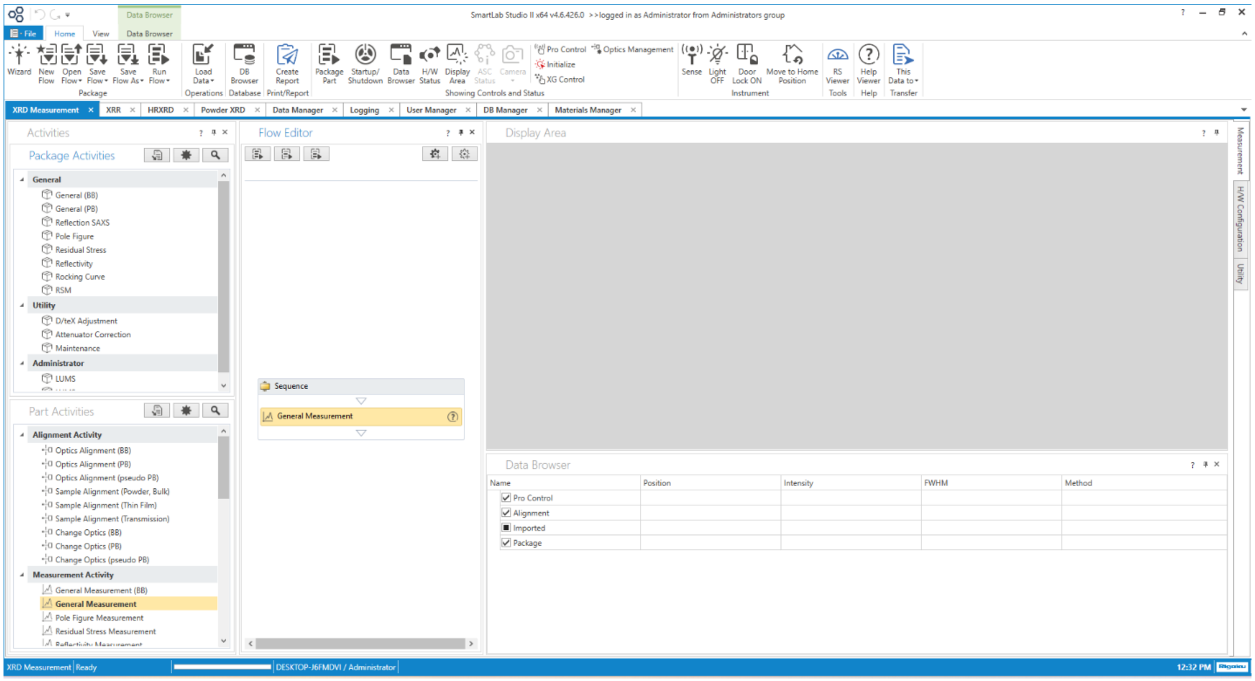


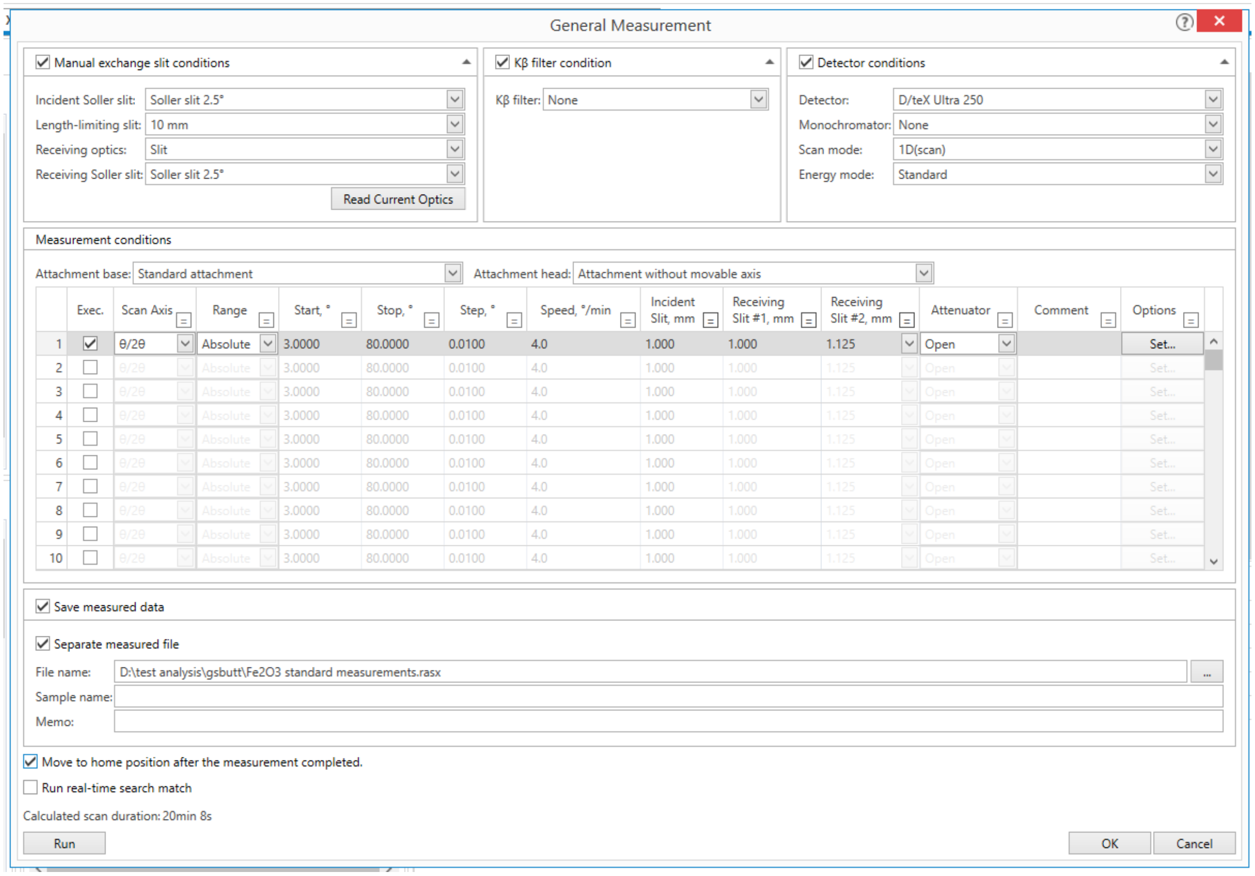


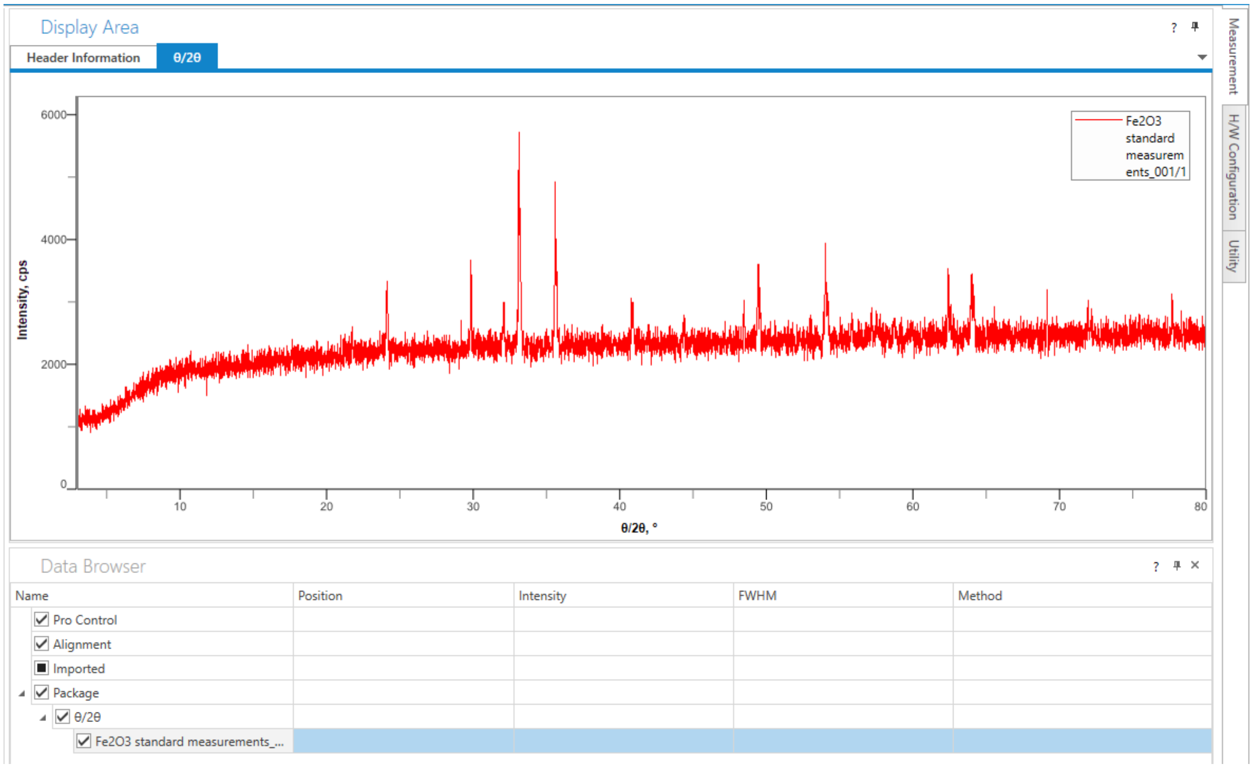
3. Switch OFF XRD instrument by rotating power key counterclockwise position.

4. Confirm that the power on indicator is turned off.
5. Turn OFF the breaker counterclockwise position, on the rear side of the XRD instrument.

Fe2O3 Powder Measurements





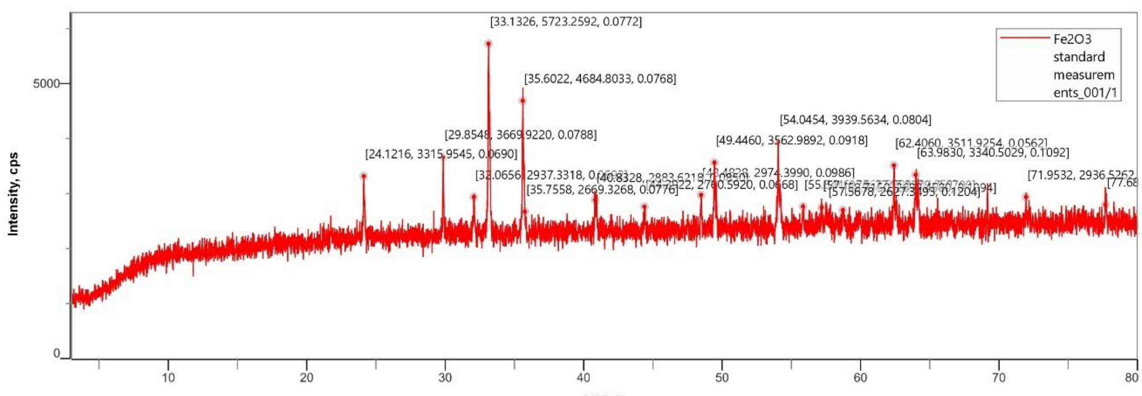


XRD Measurement Version 4.0 2024-04-26 13:10:29

Profile / Fe2O3 standard measurements_001/1

Sample name	:	Scan axis	: θ/2θ
Comment	:	Scan mode	: 1D(scan)
Date of measurement	: 2024-04-26 12:33:00	Scan range	: 3.0000 - 80.0000 °
Operator name	: Administrator	Scan step	: 0.0100 °
X-ray	: 40 kV, 45 mA	Scan speed	: 4.0000 °/min
Wavelength	: Cu-Kα / 1.54186 Å		
Goniometer	: Standard Goniometer		
Attachment base	: Standard attachment		
Attachment head	: Standard attachment head		
Detector	: D/teX Ultra 250		
Optics attribute	: BB		
Memo	:		

Selection slit	: BB
Incident optics unit	: IPS adaptor
Incident Soller slit	: Soller slit 2.5°
Incident slit box	: 1.000mm
Length-limiting slit	: 10 mm
Receiving slit box #1	: 1.000mm
Filter 1	: None
Receiving optics unit #1	: PSA open
Receiving Soller slit	: Soller slit 2.5°
Receiving slit box #2	: 1.125mm
Receiving Attenuator	: Open
Detector monochromator	: None
Detector slit	: None



Peak list

No.	Name	θ/2θ, °	Intensity, cps	FWHM, °	Method
1	Fe2O3 standard measurements_...	33.1326	5723.2592	0.0772	Powder Search
2	Fe2O3 standard measurements_...	35.6022	4684.8033	0.0768	Powder Search
3	Fe2O3 standard measurements_...	54.0454	3939.5634	0.0804	Powder Search
4	Fe2O3 standard measurements_...	29.8548	3669.9220	0.0788	Powder Search
5	Fe2O3 standard measurements_...	49.4460	3562.9892	0.0918	Powder Search
6	Fe2O3 standard measurements_...	62.4060	3511.9254	0.0562	Powder Search
7	Fe2O3 standard measurements_...	63.9830	3340.5029	0.1092	Powder Search
8	Fe2O3 standard measurements_...	24.1216	3315.9545	0.0690	Powder Search
9	Fe2O3 standard measurements_...	48.4828	2974.3990	0.0986	Powder Search
10	Fe2O3 standard measurements_...	32.0656	2937.3318	0.0692	Powder Search
11	Fe2O3 standard measurements_...	71.9532	2936.5262	0.0366	Powder Search
12	Fe2O3 standard measurements_...	40.8328	2883.6218	0.0850	Powder Search
13	Fe2O3 standard measurements_...	77.6830	2799.1880	0.0988	Powder Search
14	Fe2O3 standard measurements_...	55.8446	2762.2339	0.1052	Powder Search
15	Fe2O3 standard measurements_...	44.3922	2760.5920	0.0668	Powder Search
16	Fe2O3 standard measurements_...	57.1976	2747.2378	0.0700	Powder Search
17	Fe2O3 standard measurements_...	58.7150	2699.4580	0.1094	Powder Search
18	Fe2O3 standard measurements_...	35.7558	2669.3268	0.0776	Powder Search
19	Fe2O3 standard measurements_...	57.5678	2627.3493	0.1204	Powder Search

SmartLab SE Workshop
Concept Refresh

SmartLab SE Workshop Concept Refresh

Basic XRD principle followed by SmartLab SE parts and sample requirements. Prepared for quick workshop revision. The language is intentionally simple and practical.

1. Basic Principle of XRD: Why Diffraction Happens

X-ray diffraction happens when X-rays interact with the regular arrangement of atoms in a crystal.

Because the wavelength of X-rays is close to the spacing between atomic planes, X-rays can scatter and interfere with each other.

If the scattered X-rays interfere constructively, a diffraction peak appears. If they interfere destructively, no peak or very weak intensity is observed.

Workshop line: XRD works because X-rays scatter from crystal planes and produce strong peaks only when the scattered waves add constructively.

2. Laue Condition

The Laue condition explains diffraction in terms of wave interference from atoms in a crystal lattice.

It says that diffraction occurs only when scattered waves from different atoms remain in phase and produce constructive interference.

In simple words, the crystal acts like a 3D diffraction grating. Only certain directions satisfy the condition for constructive interference.

Workshop line: Laue condition says that diffraction occurs only in those directions where X-rays scattered from the crystal lattice interfere constructively.

3. Bragg's Law

Bragg's law gives a simple relation between X-ray wavelength, crystal plane spacing, and diffraction angle:

$$n\lambda = 2d \sin\theta$$

Where n is diffraction order, λ is X-ray wavelength, d is spacing between crystal planes, and θ is the Bragg angle.

Bragg's law says that diffraction occurs when the path difference between X-rays reflected from parallel crystal planes equals an integer multiple of wavelength.

Workshop line: Bragg's law tells us at which angle a crystal plane with spacing d will give a diffraction peak for a given X-ray wavelength.

4. Physical Meaning of Bragg's Law

When X-rays reflect from two neighboring crystal planes, one ray travels an extra distance compared with the other ray.

This extra distance is called the path difference. For constructive interference, the path difference must be equal to a whole number of wavelengths.

Path difference = $n\lambda$

Path difference = $2d \sin\theta$

Therefore: $n\lambda = 2d \sin\theta$

Workshop line: A peak appears when the extra distance travelled by one X-ray is equal to a whole number of wavelengths.

5. Relation Between Laue and Bragg Laws

Laue law and Bragg's law explain the same diffraction phenomenon in two different ways.

Laue condition explains diffraction using wave vectors and 3D crystal periodicity.

Bragg's law explains diffraction using reflection from parallel atomic planes.

Bragg's law is easier for basic XRD interpretation because it directly relates peak position to lattice spacing.

Workshop line: Laue and Bragg laws describe the same diffraction condition. Laue uses crystal lattice wave interference, while Bragg uses reflection from atomic planes.

6. Why Peak Position Depends on d, λ, and θ

From Bragg's law, if wavelength λ, lattice spacing d, or angle θ changes, the diffraction condition changes.

A uniform change usually causes a peak shift. A distribution or spread in λ, d, or θ usually causes peak broadening.

Change in d due to stress, strain, temperature, or composition can cause peak shift or broadening.

Spread in θ due to beam divergence, rough sample, or poor alignment can cause peak broadening.

Multiple wavelengths such as Cu $K\alpha_1$ and Cu $K\alpha_2$ can cause peak splitting or broadening.

Workshop line: Any small change or spread in wavelength, lattice spacing, or diffraction angle affects XRD peak position and peak shape.

7. Simple Summary of Laue and Bragg Laws

Laue condition tells us when scattered X-rays from a crystal interfere constructively.

Bragg's law gives the practical equation $n\lambda = 2d \sin\theta$ used to calculate diffraction peak positions.

In XRD, peak position and peak shape are controlled by wavelength, lattice spacing, diffraction angle, instrument optics, and sample quality.

Workshop line: Laue gives the interference condition; Bragg's law gives the practical peak-position equation.

8. Cu X-ray Gun

The Cu X-ray gun produces X-rays by accelerating electrons from the cathode to a copper target/anode.

When high-energy electrons hit the Cu target, characteristic Cu radiation is produced, mainly Cu Kα and Cu Kβ, along with continuous Bremsstrahlung radiation.

For most Cu XRD work, the useful wavelength is Cu Kα, approximately 1.5406 Å. This wavelength is fixed by the electronic energy levels of copper.

Workshop line: The Cu X-ray gun is the X-ray source. It produces Cu Kα radiation used for diffraction measurements.

9. Effect of Voltage and Current Settings

Voltage (kV) controls the kinetic energy of electrons hitting the Cu target. Higher voltage increases electron impact energy and the maximum energy of the continuous X-ray spectrum.

Current (mA) controls the number of electrons hitting the target per second. Higher current mainly increases X-ray intensity/counts.

In Cu XRD, increasing voltage does not change the Cu Kα wavelength. It mainly increases tube output and continuous background contribution.

Increasing current gives stronger peaks and shorter scan time, but also increases tube heating and tube load.

For 40 kV and 45 mA, tube power is $40 \times 45 = 1800$ W = 1.8 kW.

Workshop line: Voltage mainly controls X-ray energy/output condition; current mainly controls intensity. Cu Kα wavelength remains fixed.

10. CBO: Cross Beam Optics

CBO means Cross Beam Optics. It allows SmartLab SE to switch between Bragg-Brentano focusing beam condition and parallel beam condition.

CBO prepares the incident beam condition, but the complete optics and goniometer geometry together produce the final beam behavior.

In BB mode, CBO enables a divergent/focusing beam condition. In PB mode, CBO helps produce a low-divergence nearly parallel beam.

Workshop line: CBO prepares the beam condition for BB or PB mode; the full optical setup creates the final measurement geometry.

11. Bragg-Brentano Geometry

Bragg-Brentano geometry uses a divergent incident beam and parafocusing geometry.

If the sample is flat and at correct height, the diffracted rays are focused toward the detector slit.

BB gives high intensity and fast routine powder XRD scans, but it is sensitive to sample height error, roughness, and non-flat surfaces.

Workshop line: BB = divergent beam + focusing toward detector + high intensity for flat powder samples.

12. Parallel Beam Geometry

Parallel beam geometry uses X-rays that are nearly parallel to each other. The rays are not parallel to the sample surface; they hit the sample at the selected incident angle.

PB has low divergence, so it is less sensitive to sample height error, roughness, uneven surface, thin films, or irregular samples.

PB usually gives lower intensity than BB, so longer scan time may be required.

Workshop line: PB = nearly parallel beam + low divergence + better tolerance for rough, thin, or irregular samples.

13. Soller Slit

A Soller slit controls axial divergence. Axial divergence is the sideways/lengthwise spreading of X-rays out of the main diffraction plane.

The Soller slit contains many thin parallel plates that block off-axis rays and allow only rays within a limited angle to pass.

A 2.5° Soller slit means the allowed axial divergence is about 2.5 degrees.

It improves peak shape, reduces peak asymmetry, and lowers unwanted background, especially at low 2θ angles.

Workshop line: Soller slit controls beam direction in the axial direction; it reduces off-plane rays and improves peak shape.

14. Incident and Receiving Slits: General Idea

Incident slits are before the sample. They control the incoming X-ray beam size, divergence, and footprint.

Receiving slits are after the sample. They control which diffracted X-rays are accepted by the detector.

Smaller slits improve angular control and resolution, but reduce intensity. Larger slits increase intensity, but may increase background, peak broadening, or small apparent shifts.

Workshop line: Incident slits control the beam before the sample; receiving slits control the diffracted beam after the sample.

15. Incident Slit Box

The Incident Slit Box is placed in the incident beam path before the sample.

It controls beam size, width, and divergence before the beam reaches the sample.

A smaller slit cuts off outer, more divergent rays. This improves resolution and peak sharpness but lowers intensity.

A larger slit allows more rays to reach the sample. This increases intensity but may increase divergence, background, and broadening.

Workshop line: The Incident Slit Box balances intensity and resolution by controlling the incident beam divergence and size.

16. Length-Limiting Slit

The Length-Limiting Slit controls the length of the X-ray beam footprint on the sample surface.

Common slit sizes are 10 mm, 5 mm, and 2 mm. These values refer to the approximate irradiated length, not the total area alone.

Approximate irradiated area = beam length × beam width.

A 10 mm slit gives a larger illuminated sample length and higher intensity. A 2 mm slit is better for small samples or when avoiding the holder/background area.

Workshop line: Length-limiting slit controls how much sample length is irradiated; larger gives more intensity, smaller reduces holder/background contribution.

17. Receiving Slit Box #1 (RS1)

Receiving Slit Box #1 is placed just after the sample in the diffracted beam path.

It controls the angular acceptance of diffracted rays coming from the sample.

A smaller RS1 opening improves peak sharpness and lowers background, but intensity decreases.

Workshop line: RS1 selects the diffracted rays after the sample and improves angular resolution.

18. Receiving Slit Box #2 (RS2)

Receiving Slit Box #2 is closer to the detector than RS1.

It further cleans the diffracted beam by removing scattered or off-angle rays before detection.

RS2 helps improve background, peak shape, and detector signal quality.

Workshop line: RS2 further cleans/collimates the diffracted beam before the detector.

19. Detector

The detector measures the diffracted X-ray photons at each 2θ angle and produces the XRD pattern: intensity vs 2θ.

D/teX Ultra 250 is a 1D silicon strip detector. It records intensity along a line of strips and is much faster than a single point detector for routine scans.

A 0D detector behaves like a point counter, measuring one angular position at a time. A 1D detector measures a small angular range at the same time.

Some SmartLab SE systems can also use a HyPix-400 2D detector, which records diffraction over an area and can operate in 0D/1D/2D modes depending on configuration.

Workshop line: The detector counts diffracted X-rays and converts them into the final XRD pattern.

20. Beta Filter

A beta filter is used to reduce or remove Kβ radiation from the X-ray beam.

For a Cu source, a Ni filter is commonly used because it strongly absorbs Cu Kβ while allowing much of Cu Kα to pass.

The beta filter improves spectral purity and reduces unwanted peaks/background caused by Kβ radiation.

It does not select a very narrow single Kα1 wavelength; it mainly suppresses Kβ.

Workshop line: Beta filter removes Kβ radiation; for Cu X-rays, Ni filter is commonly used to keep mainly Cu Kα.

21. Ge(220) 2-Bounce Monochromator

A Ge(220) 2-bounce monochromator selects X-rays by diffraction from germanium crystals using the Ge(220) planes.

It improves wavelength purity by allowing a narrow wavelength band to pass. In high-resolution setups, it can help select mainly Cu Kα1.

Compared with a beta filter, a monochromator gives much better spectral purification but reduces intensity more.

It is useful in PB/high-resolution measurements where sharper peaks and better wavelength definition are required.

Workshop line: Monochromator controls wavelength purity; Soller slit controls beam direction/divergence.

22. Sample Requirements to Achieve Best Results

The sample surface should be flat, smooth, and at the correct height, especially in Bragg-Brentano mode.

Powder should be fine, homogeneous, and randomly oriented to reduce preferred orientation effects.

The irradiated area should stay fully on the sample. If the beam hits the holder, background and unwanted peaks may increase.

The sample should be thick enough for normal powder XRD, but not too rough or uneven. For thin, rough, curved, or irregular samples, PB mode is usually better.

Avoid contamination, large grains, cracks, uneven packing, and transparent/loose powder surfaces when possible.

Workshop line: Best XRD data comes from a flat, homogeneous, correctly positioned sample with good particle statistics and minimum holder contribution.

22. PSA Open

PSA means Parallel Slit Analyzer.
It is a receiving-side optical component used to control the angular acceptance of diffracted X-rays before they reach the detector.

When the setting is **PSA Open**, the PSA position is open, so strong angular selection by the parallel slit analyzer is not applied.
This allows more diffracted X-rays to pass, giving higher intensity, but angular resolution and background control may be lower.

Workshop line:

PSA Open means the receiving beam path is open at the Parallel Slit Analyzer position. It gives higher intensity but less angular selection.

23. Receiving Soller Slit 2.5°

The **Receiving Soller Slit 2.5°** controls **axial divergence** of the diffracted X-ray beam on the receiving side.
Axial divergence means sideways or lengthwise spreading of X-rays out of the main diffraction plane.

It contains thin parallel plates that block off-axis rays and allow only rays within about **2.5° axial divergence** to pass.
This improves peak shape, reduces asymmetry, and helps lower background.

Workshop line:

The receiving Soller slit 2.5° reduces axial divergence of diffracted X-rays and improves peak shape and background control.

24. ROD Adaptor

ROD means Receiving Optical Device adaptor.
It is a receiving-side adaptor used to mount and position receiving optical devices in the diffracted beam path.

The ROD adaptor itself does not create diffraction and does not mainly select wavelength.
Its main function is to support correct placement and alignment of receiving-side optics.

Workshop line:

The ROD adaptor is the Receiving Optical Device adaptor. It helps mount and position receiving-side optics correctly in the diffracted beam path.

25. RPS Adaptor

RPS means Receiving Parallel Slit adaptor.
It is part of the receiving-side optics and is used to mount or select the receiving parallel slit condition.

The receiving parallel slit controls the direction and angular acceptance of diffracted X-rays before they reach the detector.
A narrower parallel slit condition improves resolution and background control, while a more open condition gives higher intensity.

Workshop line:

The RPS adaptor is the Receiving Parallel Slit adaptor. It supports receiving-side parallel slit optics and helps control resolution, background, and intensity.

26. Quick Comparison Table

Part / Mode	Main Control	Main Effect
Laue condition	Constructive interference in crystal lattice	Defines allowed diffraction directions
Bragg's law	λ, d, and θ relation	Calculates peak position
Voltage	Electron energy	Higher tube output; higher continuous spectrum limit
Current	Number of electrons	Higher intensity/counts
CBO	Beam condition	Switches/enables BB or PB optical condition
BB	Divergent beam geometry	High intensity; needs flat sample
PB	Low-divergence beam	Better for rough/thin/irregular samples
Soller slit	Axial divergence	Less asymmetry and cleaner peak shape
Incident slit	Incident beam divergence/size	Intensity-resolution balance
Length-limiting slit	Beam footprint length	Controls irradiated sample length
RS1/RS2	Diffracted beam acceptance	Better resolution/background control
Beta filter	Kβ wavelength removal	Cleaner Cu Kα-dominant beam
Monochromator	Wavelength purity	Higher spectral resolution, lower intensity
Detector	Photon counting	Produces intensity vs 2θ pattern