Role of Sample Holders, Kβ Radiation, Slit Optics, in XRD Measurements

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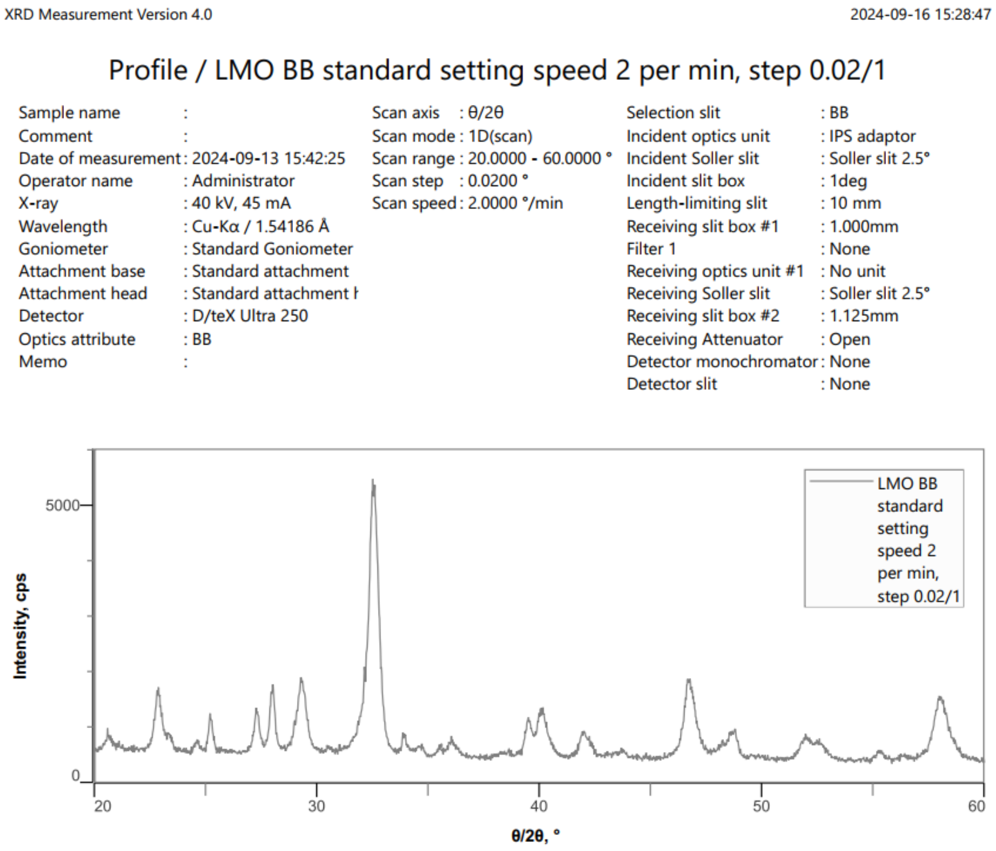
Introduction

In earlier studies, the researcher relied on XRD measurements acquired without a Kβ filter, as the resulting higher intensities were well suited to the experimental objectives at that time. In recent measurements, however, several unexpected peaks became evident during advanced analytical procedures such as Rietveld refinement, prompting the need for clarification regarding their origin. In response to this evolving analytical requirement, a focused investigation was undertaken to determine the source of these additional peaks and to ensure that the measurement conditions remained scientifically reliable.

The goal of this work is to accurately identify the origin of the observed extra peaks and to establish an optimized measurement configuration that meets the updated analytical needs. Instead of challenging prior measurement practices, this work systematically inspects the instrument optics, sample holders, and characteristic X-ray radiation components, enabling a comprehensive re-evaluation of the measurement setup. This approach enables confirmation of the root cause of the additional peaks and refinement of the configuration to ensure consistent, reliable, and reproducible XRD data for future research that support both qualitative and quantitative analyses.

Initially, XRD scans acquired without the Cu Kβ filter (BB optics) were accepted and preferred, as they produced higher count rates and stronger signal evident from the taller peaks compared with the Cu Kβ–filtered scan. By contrast, the Cu Kβ–filtered measurement lowers overall intensity.


XRD Measurement Version 4.0

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Comment :
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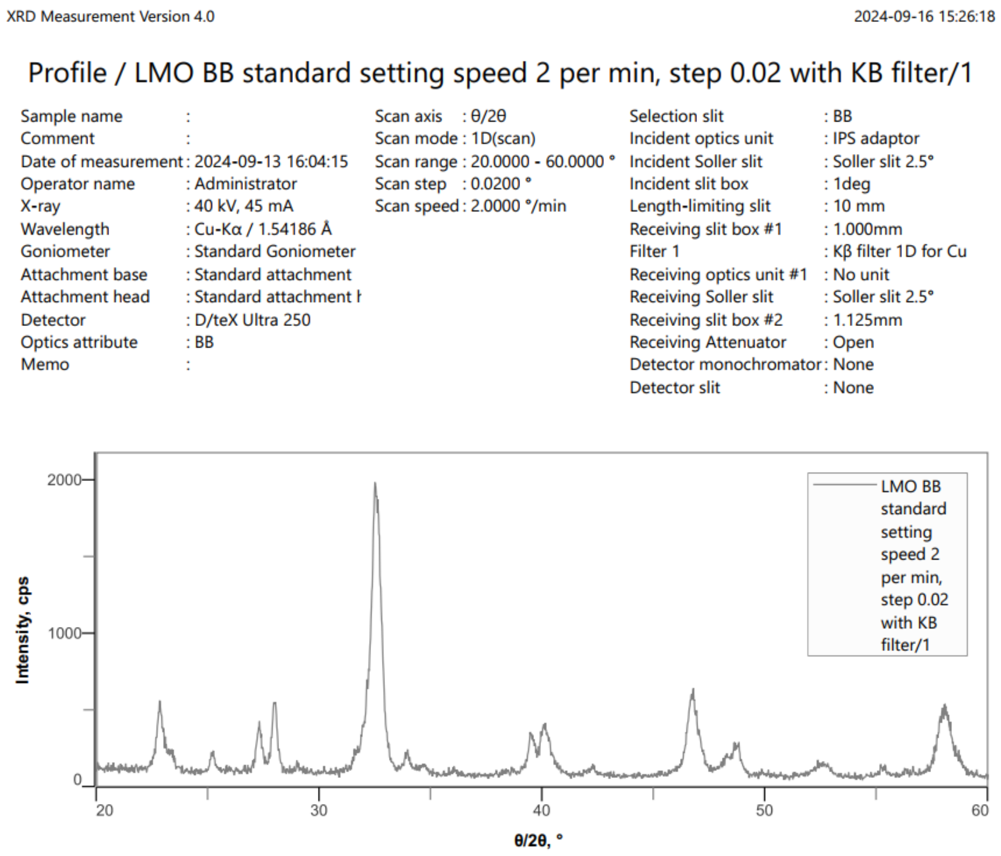
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Standard attachment
Attachment head : Standard attachment
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : 1D(scan)
Scan range : 20.0000 - 60.0000 °
Scan step : 0.0200 °
Scan speed : 2.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



XRD Measurement Version 4.0

2024-09-16 15:26:18

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Date of measurement : 2024-09-13 16:04:15
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Standard attachment
Attachment head : Standard attachment
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : 1D(scan)
Scan range : 20.0000 - 60.0000 °
Scan step : 0.0200 °
Scan speed : 2.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

Role of Sample Holders in XRD measurements

Glass sample holders

Glass holders, being amorphous, provide a clean background and require only a modest powder volume to achieve sufficient sample thickness. When tested alongside zero-background holders at equal powder depths, both produced artifact-free patterns. These comparisons show that, as long as the powder layer is properly leveled and sufficiently thick, none of the holders contribute detectable peaks. The minimum powder volume required for a reliable analysis.

1. Smaller holder dimensions (length × width × depth) 20 mm × 20 mm × 0.2 mm = 80 mm^3
2. Larger holder dimensions (length × width × depth) 20 mm × 20 mm × 0.5 mm =200 mm^3

Aluminum sample holders

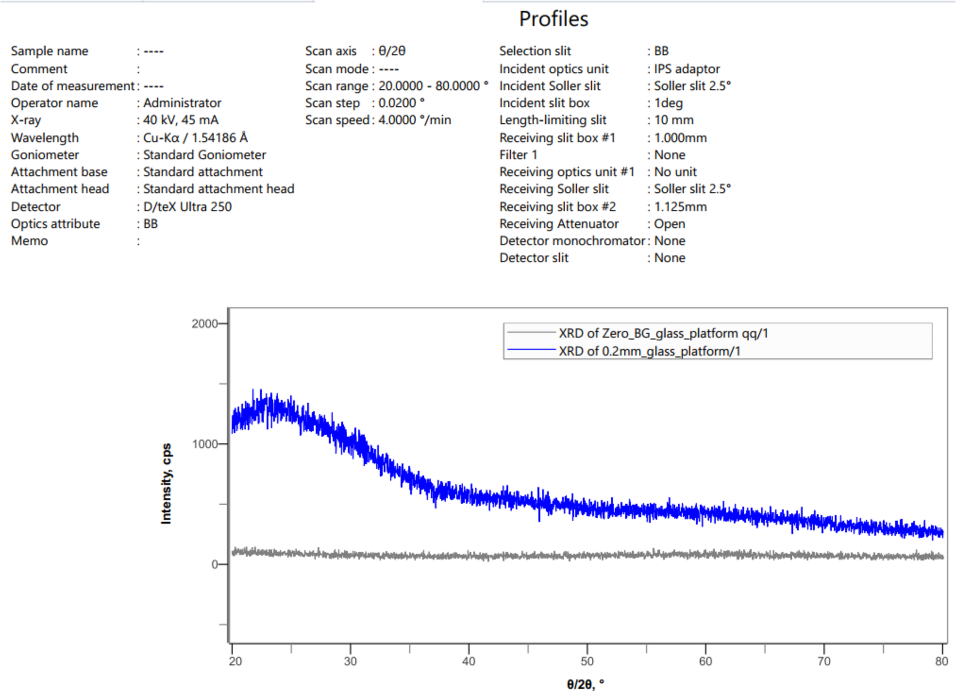
Aluminum autosampler holders, designed for the SmartLab SE geometry, can be used in either the front or inverted orientation depending on powder volume. Measurements performed using multiple autosampler holders with the same powder depth showed highly consistent results, confirming that these holders do not introduce variations in peak positions or intensities. The Automatic Sample Changer (ASC) accommodate two holder to hold the quantity

1. Inverted-side holder dimensions (radius × depth) = 12 mm × 0.2 mm = 227 mm^3
2. Front Side holder dimensions (radius × depth) = 12 mm × 2 mm = 905 mm^3

Overall, the agreement between aluminum, glass, and zero-background holders verifies that the SmartLab SE produces reliable and reproducible diffraction data when appropriate sample-preparation practices are followed.

XRD Measurements of **zero background, glass holders**

A comparison of zero-background and glass holders, showed no detectable substrate peaks. Zero-background holders offer the cleanest baseline, while glass holders provide an amorphous background. The similarity between the two measurements verifies that holder-related effects are not responsible for the unexpected peaks observed earlier, allowing the investigation to shift toward radiation and optical components as the more likely sources.



Profiles

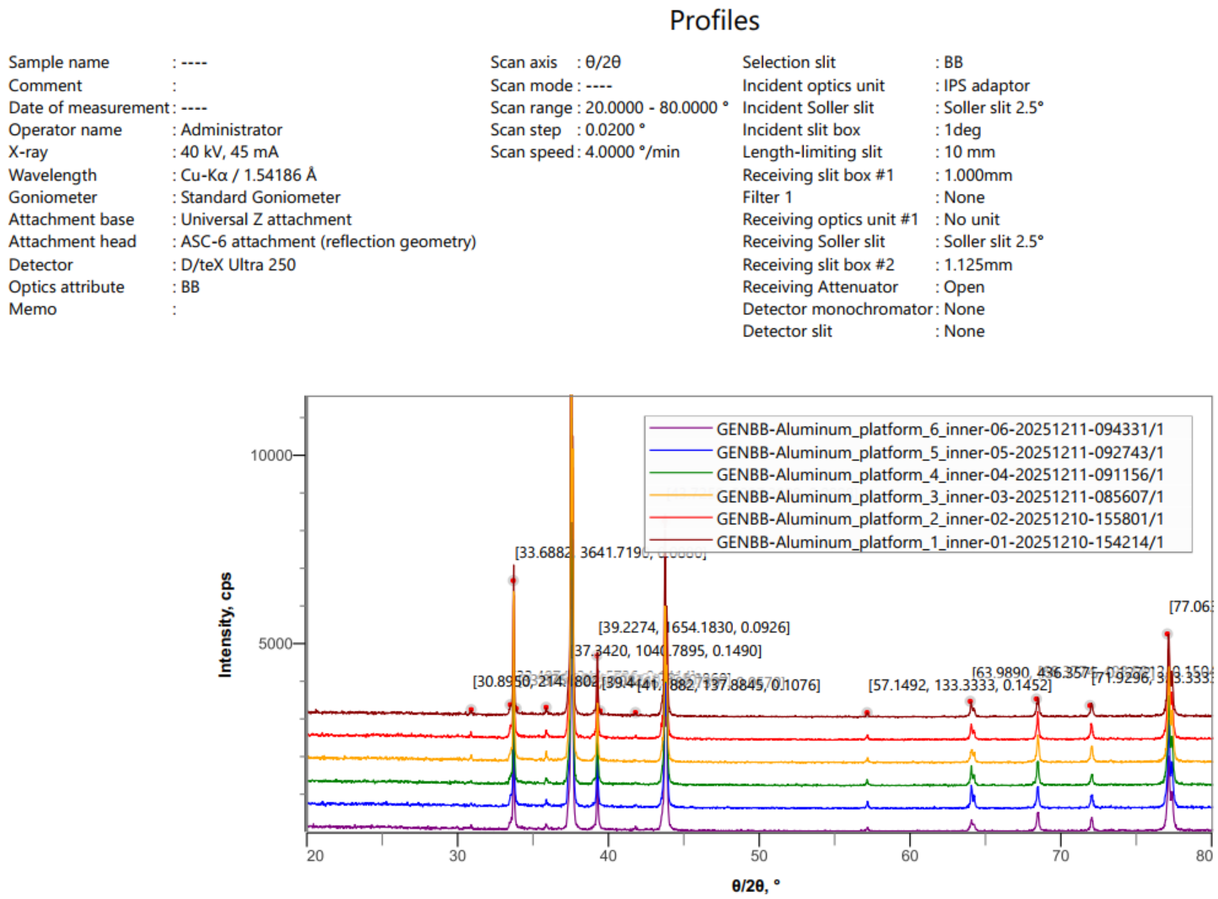
Sample name : ----
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Standard attachment
Attachment head : Standard attachment head
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

XRD Measurements of AL autosampler holders

XRD measurements performed on six empty aluminum autosampler holders, each with a depth of **2.0 mm**, showed highly consistent background patterns with no significant variation in intensity or profile. Importantly, the background features from the holders do not coincide with any sample peaks, confirming that the holders are not responsible for the additional peaks observed earlier. When a sample is present, the powder layer fully absorbs the incident beam, effectively suppressing any potential holder-related contributions.



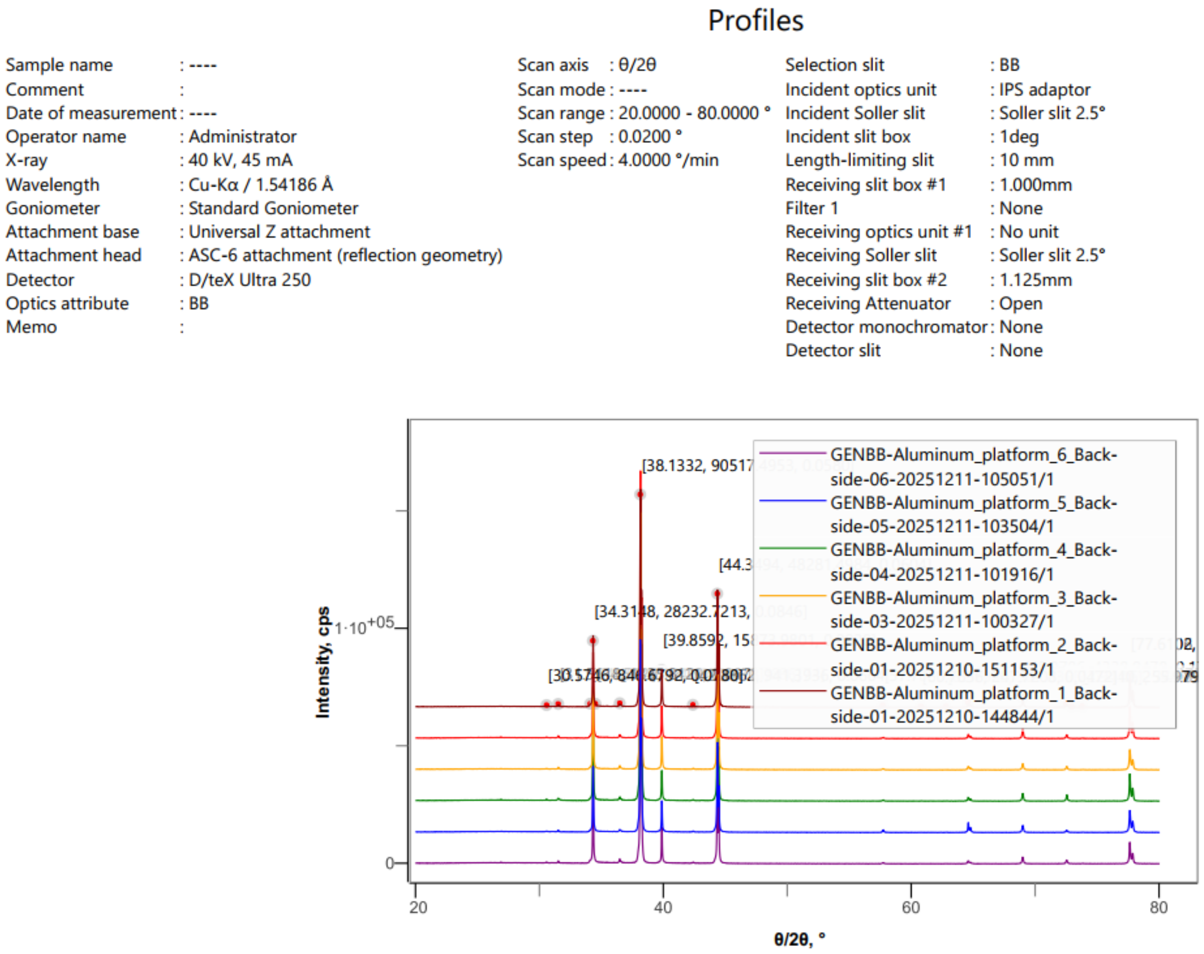
Profiles

Sample name : ----
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Universal Z attachment
Attachment head : ASC-6 attachment (reflection geometry)
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

Similar observations are noted for the six empty autosampler aluminum holders, each configured with a sample depth of **0.2 mm**.



Profiles

Sample name : ----
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Universal Z attachment
Attachment head : ASC-6 attachment (reflection geometry)
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

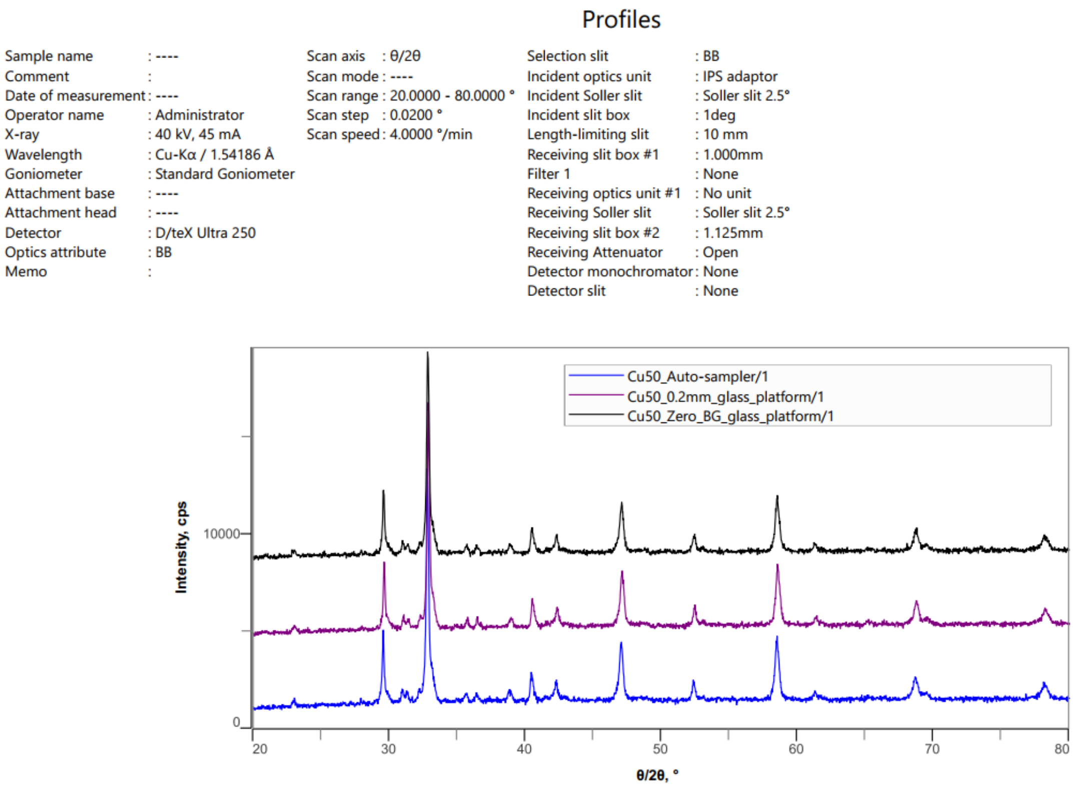
Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

Inference: The glass and zero-background sample holders produced no diffraction peaks. In contrast, the Aluminium holders produced peaks, but none matched the sample's diffraction peaks.

XRD Measurements of the sample on AL autosampler, glass and zero-background holders

To verify whether any holder contributes detectable peaks when a sample is present, the same powder was measured on the aluminium autosampler holder, the glass holder, and the zero-background holder, each prepared with an identical powder depth of 0.2 mm. All three measurements produced nearly identical diffraction patterns with no additional peaks originating from the holders. This confirms that a properly levelled and sufficiently thick powder layer effectively absorbs the incident beam, suppressing any holder-related background. These results further validate that the unexpected peaks observed earlier were not caused by the sample holders and confirm that the SmartLab SE provides high-quality measurements in which the collected patterns accurately represent the sample alone.

XRD measurement results for the Cu-50 sample measured on the Al autosampler holder, glass holder, and zero-background holder, each with a depth of 0.2 mm, showing no detectable peaks originating from any holder.



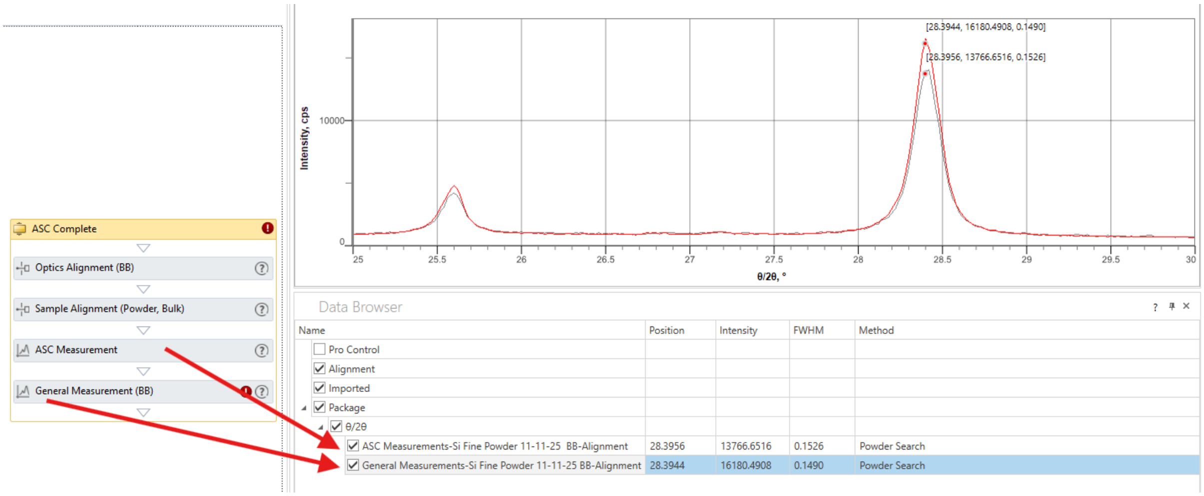
Profiles

Sample name : ----
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : ----
Attachment head : ----
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

It is simply a comparison of XRD measurements of the same sample using Aluminium and glass sample holders, demonstrating that both holders yield results of comparable quality.



Name	Position	Intensity	FWHM	Method
☐ Pro Control				
☑ Alignment				
☑ Imported				
☑ Package				
☑ θ/2θ				
☑ ASC Measurements-Si Fine Powder 11-11-25 BB-Alignment	28.3956	13766.6516	0.1526	Powder Search
☑ General Measurements-Si Fine Powder 11-11-25 BB-Alignment	28.3944	16180.4908	0.1490	Powder Search

Inference**:** No diffraction peaks originate from any holder; in particular, none arise from the aluminium holder. All observed peaks are intrinsic to the sample itself.

Role of Sample Height and Holder Depth in supressing the Peaks from holder

The powder volume used was approximately 100 mm³. Using a smaller quantity may degrade measurement quality, depending on the sample's absorption/scattering characteristics and the measurement objective (e.g., phase identification vs. Rietveld refinement).

The autosampler holder is a crystalline material and therefore produces diffraction peaks, whereas the glass holder is amorphous and does not. This has been correctly verified through the empty-holder test. To prevent X-ray penetration into the holder and suppress background peaks, all samples measured on the autosampler must be prepared with a compacted powder thickness of at least 0.5 mm, which is a universal requirement for all sample types. This thickness stops X-rays from reaching the holder, eliminates substrate-related peaks, ensures a clean baseline suitable for Rietveld refinement and Search/Match analysis, and provides reliable, reproducible results for both commercial and research measurements.

The penetration depth (δ) is given by $\delta = \frac{1}{\mu \sin \theta}$, where μ is the linear absorption coefficient (cm^{-1}), making it dependent on both angle and material. Therefore, a minimum safe powder thickness $t \geq 0.5$ mm should be maintained.

Practical Thickness Requirement for Powder in Holders

Sample Type	Minimum Safe Powder Thickness
Light oxides	0.3–0.5 mm
Medium oxides	0.15–0.3 mm
Metals / heavy phases	0.05–0.15 mm

Effect of Holder Depth on 2θ Offset

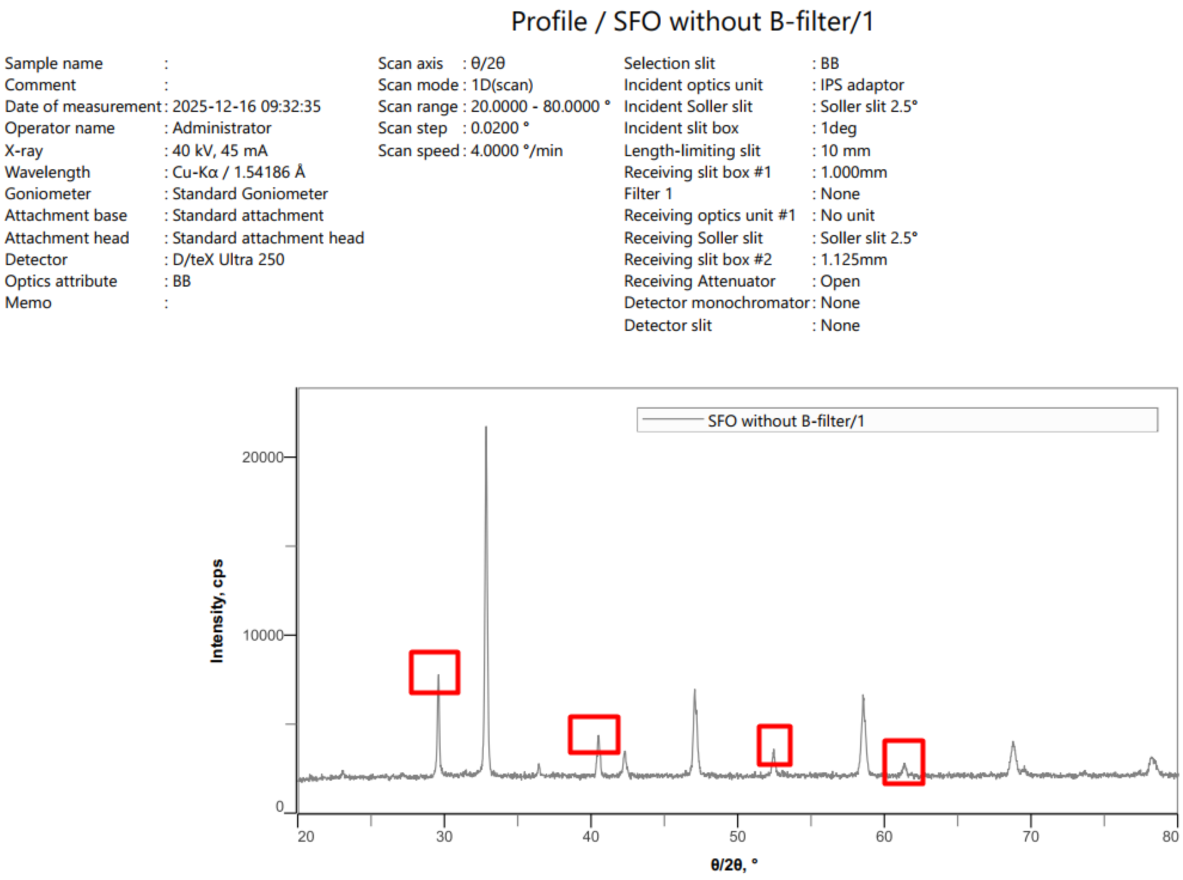
The 0.63° shift between the two peaks is observed Al autosampler holders of depths 2.0 mm and the 0.2 mm. The powder surface is adjusted to the same height in both holders.

Sample Holder of Auto Sampler		
Depth 0.2mm	Depth 2mm	Offset
34.31	33.68	0.63
38.13	37.51	0.62
39.85	39.22	0.63
44.34	43.72	0.62
64.58	63.98	0.60
68.97	68.37	0.60
77.61	77.06	0.55

Inference**:** No diffraction peaks originating from the sample holder are observed when the sample thickness exceeds the X-ray penetration depth for the given material.

Identification of Cu Kβ Contributions with SmartLab Studio II

In the first measurement of the SFO sample taken without a K-beta filter, several additional peaks were observed and are highlighted in red in the XRD pattern. The researcher suspected that these peaks might have originated from the sample holders used during measurement. To investigate this, the sample was measured on the autosampler holder, the glass holder, and the zero-background holder. All tests showed that none of these holders produced detectable diffraction peaks, demonstrating that the holders were not responsible for the extra peaks seen in the initial pattern.



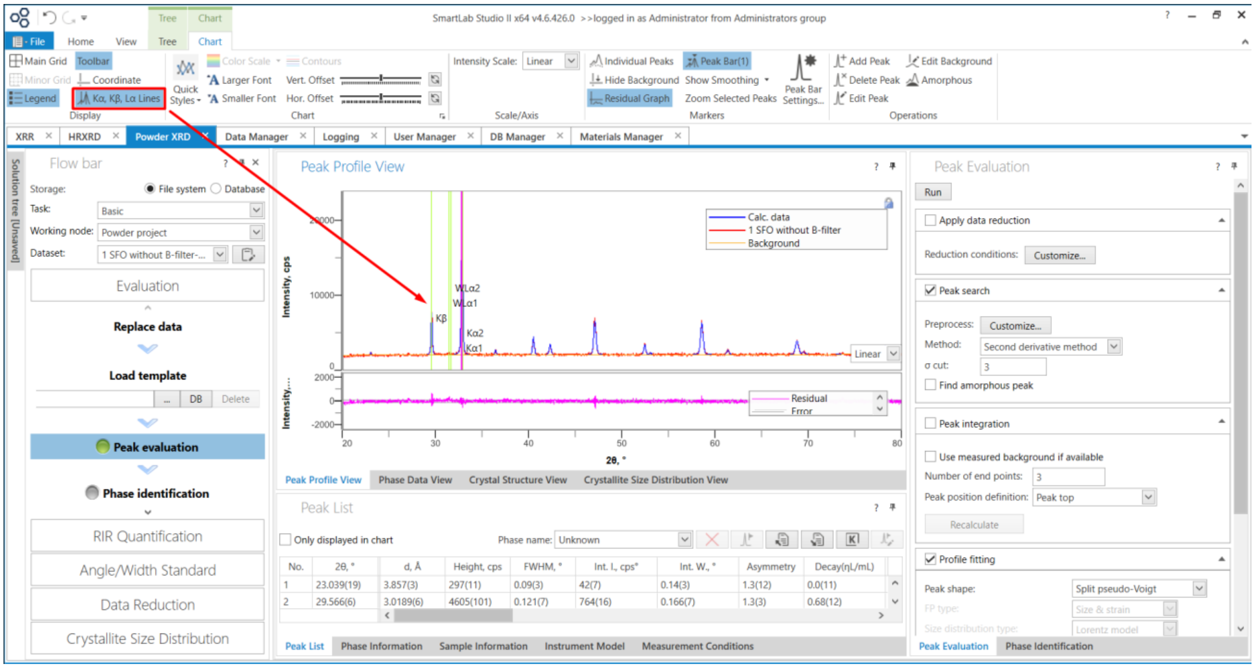
Profile / SFO without B-filter/1

Sample name :
Comment :
Date of measurement : 2025-12-16 09:32:35
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : 1D(scan)
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : None
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None

Despite ruling out the sample holders as a possible source, the researcher continued to observe additional peaks during their fitting analysis. Consequently, a second-phase investigation was carried out using SmartLab Studio II. The software's peak evaluation tools were employed to overlay the observed peaks with the characteristic radiation lines of the Cu X-ray source. This analysis clearly demonstrated that the extra peaks correspond to Cu Kβ radiation, which is present when no Kβ filter is used. The investigation therefore confirmed that the additional peaks are not sample-related but arise due to the intrinsic Kβ component of the X-ray source.



SmartLab Studio II x64 v4.6.426.0 >>logged in as Administrator from Administrators group

Intensity Scale: Linear

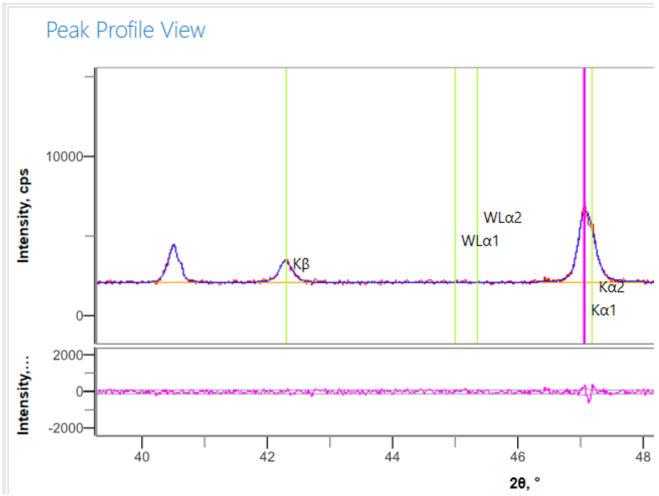
Storage: ● File system ○ Database
Task: Basic
Working node: Powder project
Dataset: 1 SFO without B-filter-...

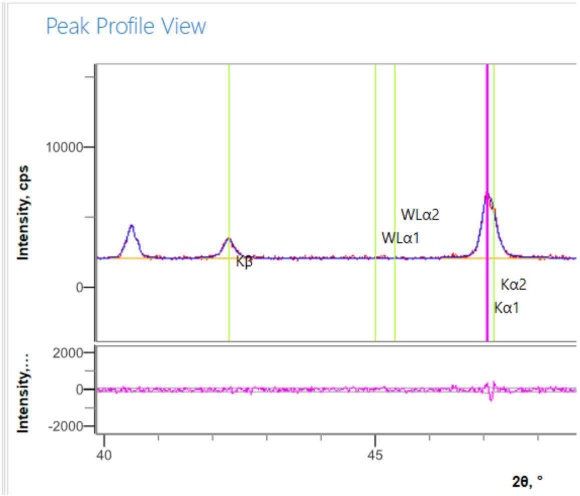
☐ Only displayed in chart
Phase name: Unknown

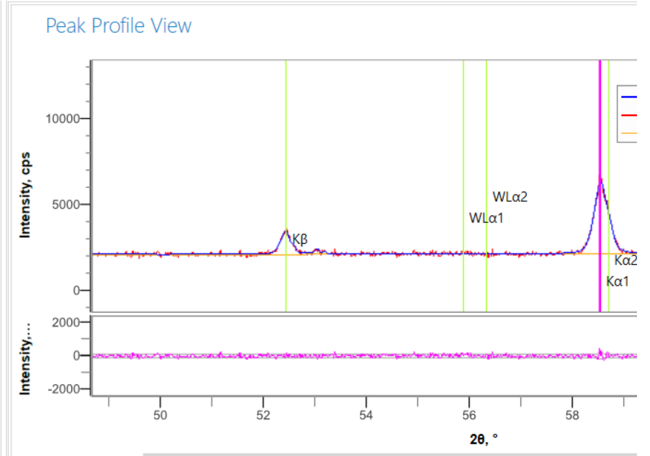
No.	2θ, °	d, Å	Height, cps	FWHM, °	Int. I., cps°	Int. W., °	Asymmetry	Decay(ηL/mL)
1	23.039(19)	3.857(3)	297(11)	0.09(3)	42(7)	0.14(3)	1.3(12)	0.0(11)
2	29.566(6)	3.0189(6)	4605(101)	0.121(7)	764(16)	0.166(7)	1.3(3)	0.68(12)

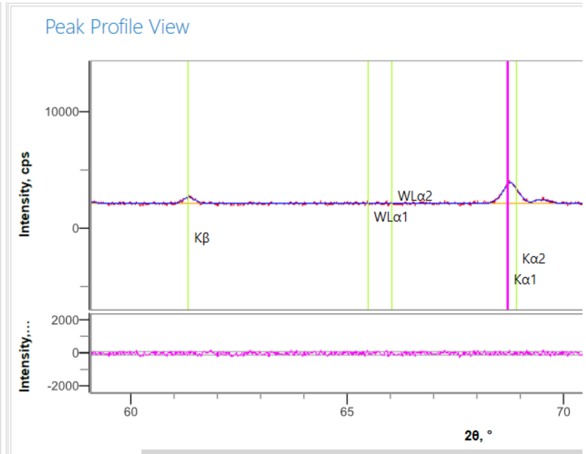
☐ Apply data reduction
Reduction conditions: Customize...
☑ Peak search
Preprocess: Customize...
Method: Second derivative method
σ cut: 3
☐ Find amorphous peak
☐ Peak integration
☐ Use measured background if available
Number of end points: 3
Peak position definition: Peak top
☑ Profile fitting
Peak shape: Split pseudo-Voigt
FP type: Size & strain
Size distribution type: Lorentz model

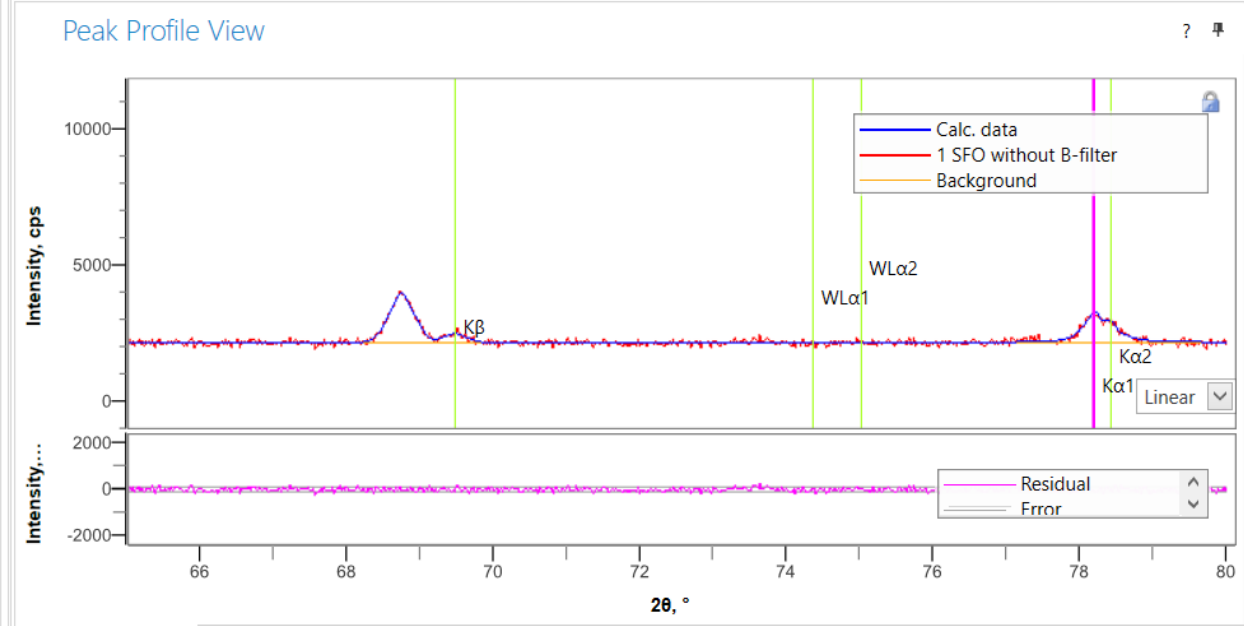
All five suspected peaks were examined individually in SmartLab Studio II using the peak-profile view, as shown in the images above. For each peak, the software overlaid the characteristic Cu radiation lines (Kα1, Kα2, and Kβ). In every case the extra peaks aligned exactly with the Cu Kβ position, confirming that they were not related to the sample. Because the measurement was performed without a K-beta filter, the Kβ component appeared as weak secondary peaks beside the main Kα reflections. This analysis demonstrates that all four additional peaks originated solely from Cu Kβ radiation, not from the sample or the sample holders.











Impact of Cu Kβ Radiation on XRD Analyses

The additional peaks caused by Cu Kβ radiation are a natural part of the Cu X-ray tube spectrum when a K-beta filter or monochromator is not used. These Kβ peaks generally do not interfere with basic analyses such as *phase identification* or *qualitative comparison*, because modern databases and software can easily distinguish Kβ positions from true sample reflections. However, they become problematic in quantitative and precision-based analyses, where the presence of mixed radiation can distort peak shapes, skew background modeling, and reduce the accuracy of refined parameters. For such cases especially *Rietveld refinement*, *crystallite size/strain analysis*, *quantitative phase analysis*, and *structure refinement* the Kβ component must be removed using a K-beta filter, a monochromator, or software based Kβ stripping to ensure the dataset contains only Cu Kα radiation. In summary, Kβ peaks are acceptable for quick qualitative work, but must be eliminated for any high-accuracy or quantitative XRD analysis.

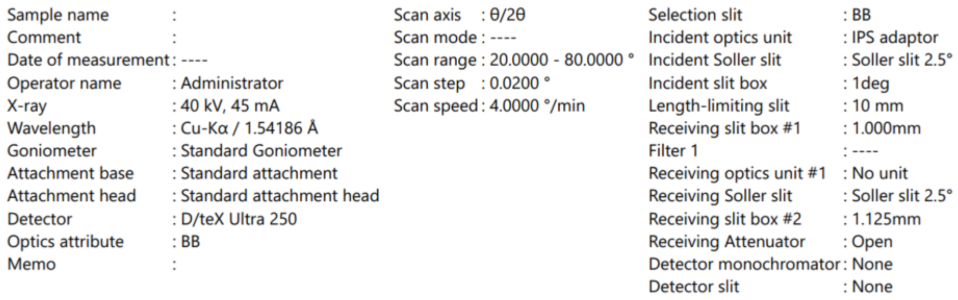
Inference**:** All observed additional peaks are conclusively attributed to intrinsic Cu Kβ radiation arising from measurements performed without a Kβ filter, and are not related to the sample or sample holders.

Table: Suitability of XRD Analyses in the Presence of Cu Kβ Peaks

Type of XRD Analysis	**Effect of Cu Kβ Peaks**	**Recommended Action**
Phase Identification (Search/Match)	Minimal effect; software distinguishes Kβ easily.	Kβ removal *not required*.
Qualitative Comparison Between Samples	Negligible impact on pattern interpretation.	Kβ removal *not required*.
Fingerprint Matching with Standard Patterns	Acceptable; Kβ peaks appear as small secondaries.	Optional cleanup.
Rietveld Refinement	Distorts peak shape and fitting accuracy.	Kβ must be removed.
Crystallite Size & Microstrain Analysis	Affects FWHM and peak-profile parameters.	Kβ must be removed.
Structure Refinement & Lattice Parameters	Mixed radiation reduces precision.	Kβ must be removed.
Quantitative Phase Analysis (QPA)	Peak intensities become unreliable.	Kβ must be removed.
Texture & Residual Stress Analysis	Reduced precision due to profile distortion.	Kβ removal recommended.

Elimination of Kβ Radiation

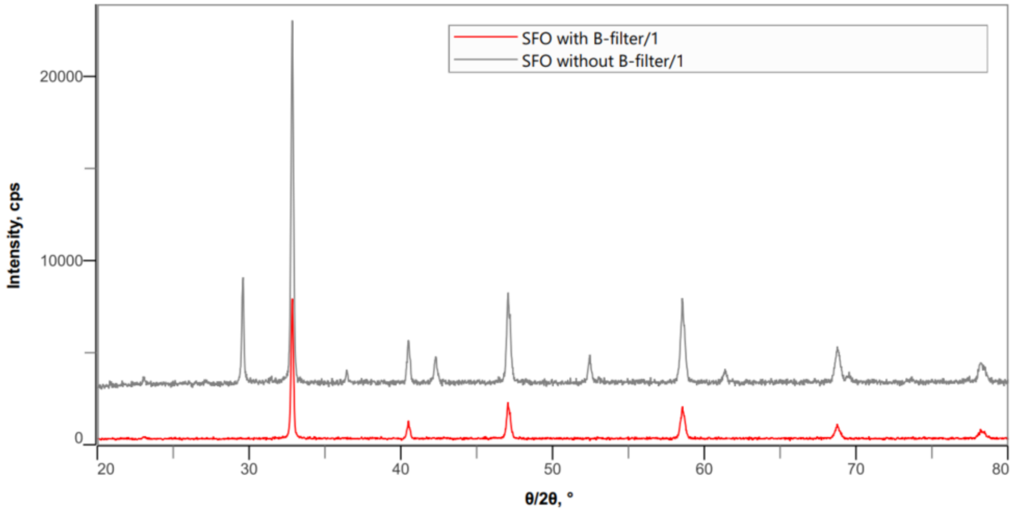
After confirming that the additional peaks originated from Cu Kβ radiation, the sample was re-measured on the SmartLab SE using the K-beta filter. As shown in the comparison plot, applying the Kβ filter completely eliminated all the unwanted secondary Kβ peaks, leaving only the pure Cu Kα radiation in the final diffraction pattern. This produced a much cleaner and more accurate dataset, free from mixed-radiation artifacts. With the Kβ component removed, the XRD pattern is now fully suitable for high-precision analyses such as Rietveld refinement, quantitative phase analysis, and crystallite size/strain calculations, ensuring reliable and reproducible results.

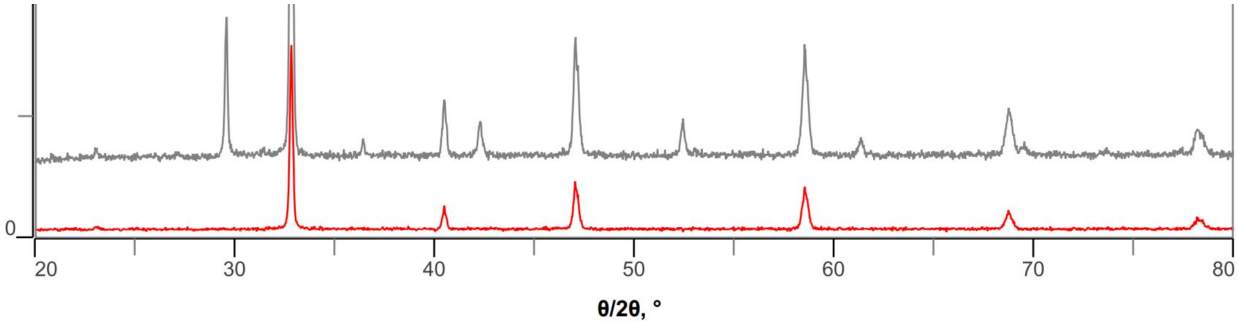


Sample name :
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Standard attachment
Attachment head : Standard attachment head
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 4.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 1.000mm
Filter 1 : ----
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None





Peak-Intensity Reduction After Applying Kβ Filter and the Need for Slit Optimization

When the K-beta filter is applied on the SmartLab SE, the overall peak intensity decreases because the filter selectively absorbs a portion of the Cu radiation, allowing only the pure Kα component to reach the detector. While this removes all unwanted Kβ peaks and produces a cleaner diffraction pattern, the loss of total photon flux naturally lowers the intensity of the Kα peaks, as observed in the measurement results. Since the researchers require higher peak intensities for their analysis, the investigation is being continued by optimizing the **incident slit** and **receiving slit** settings. By adjusting combinations of Soller slits, divergence slits, and receiving slits, the photon flux reaching the sample and detector can be increased while still maintaining controlled resolution. This approach allows the instrument to deliver higher intensities even with the Kβ filter in place, ensuring that the data meets both purity and intensity requirements.

When High Intensity Is Required and When It Is Not: Does Intensity Matter?

Higher peak intensity is particularly important in applications where weak or minor phases must be detected, where very small crystallite-size effects are studied, or where low-concentration impurities need to be identified. Stronger intensities improve the signal-to-noise ratio, enhance the visibility of small peaks, and support more precise quantitative analysis. However, for routine phase identification, general pattern comparison, and qualitative analysis, extremely high intensity is not essential as long as the peaks are clearly visible and free from artifacts such as Kβ radiation. In XRD, intensity matters mainly when sensitivity, detection limits, and quantitative accuracy are critical; otherwise, clean, artifact-free peaks are more valuable than very high counts. Thus, intensity is important but only relative to the analytical objective.

Inference**:** The use of a Kβ filter effectively removes Cu Kβ contributions, yielding a clean Cu Kα diffraction pattern that is fully suitable for high-precision and reliable XRD analyses.

Optimization of Incident and Receiving Slits for Enhancing Peak Intensity

Peak intensity in powder XRD depends strongly on the configuration of the incident and receiving optics, since these components directly control the amount of X-ray flux reaching the sample and the detector. After applying the K-beta filter, a natural reduction in intensity occurs because the filter selectively absorbs part of the Cu spectrum. To recover or enhance peak intensity without sacrificing data quality, the next step involves optimizing the incident slit, Soller slit, and receiving slit combinations on the SmartLab SE. By selecting wider slits or adjusting optics in a controlled manner, it is possible to significantly increase photon flux, improve the signal-to-noise ratio, and enhance peak visibility while maintaining acceptable resolution for the intended analysis. This optimization allows researchers to achieve higher intensities that meet their analytical requirements, even when operating with filtered radiation.

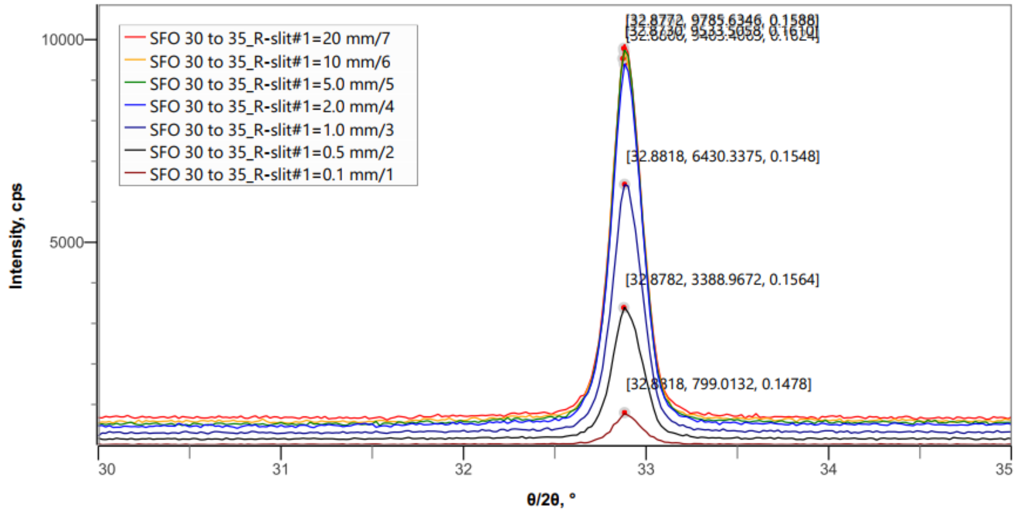
Influence of Receiving Slit #1 Width on XRD Peak Intensity

The XRD measurements indicate that widening Receiving Slit #1 increases peak intensity as the slit width is expanded from 0.1 mm to approximately 2 mm, beyond which no further intensity gain is observed. This behavior suggests that at 2 mm the diffracted beam is fully collected by the optics, resulting in intensity saturation. Further opening of the slit does not increase photon flux and may instead degrade angular resolution without providing any meaningful improvement in signal.

Sample name :
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 30.0000 - 35.0000 °
Scan step : 0.0200 °
Scan speed : 1.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : ----
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 1.125mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



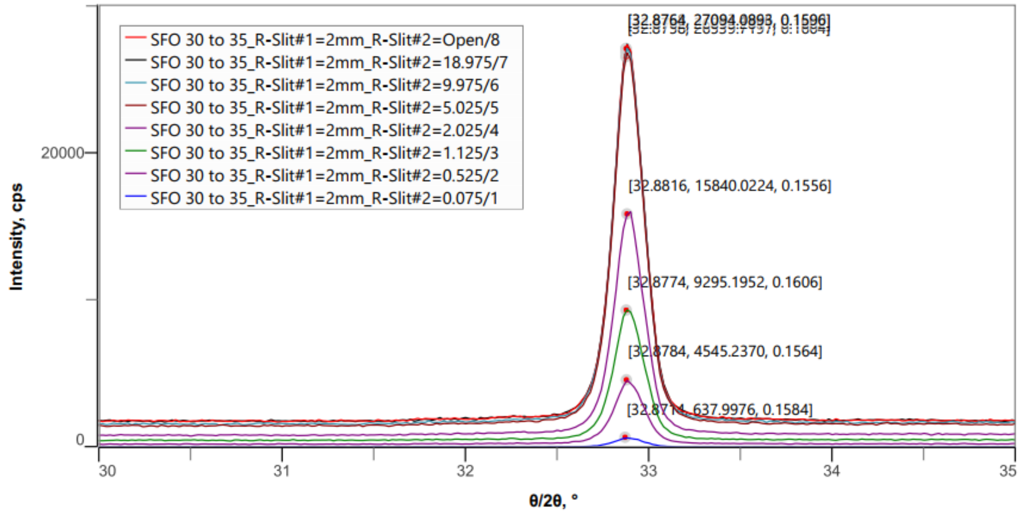
Optimization of Receiving Slit #2 for Maximum XRD Signal

With Receiving Slit #1 fixed at its optimal width of 2 mm, the XRD results show that increasing the width of Receiving Slit #2 produces a steady increase in peak intensity, reaching a maximum at approximately 5.025 mm. This behavior reflects enhanced acceptance of the diffracted beam by the secondary receiving optics as the slit is widened. Beyond this width, no significant additional intensity gain is observed, indicating saturation where the full diffracted beam is already collected. Therefore, a width of 5.025 mm represents the optimal setting for Receiving Slit #2, providing maximum signal without unnecessary resolution degradation.

Sample name :
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 30.0000 - 35.0000 °
Scan step : 0.0200 °
Scan speed : 1.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 2.000mm
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : ----
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



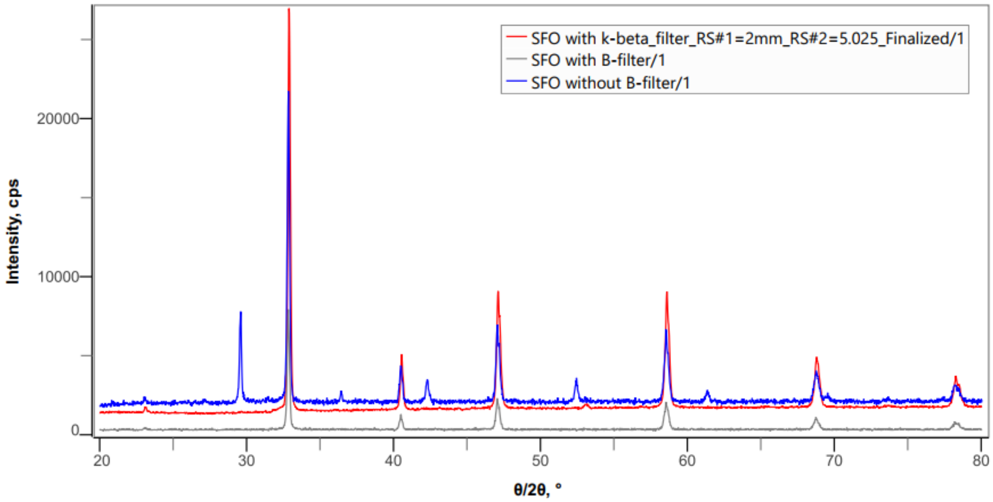
Optimization of XRD Intensity Using Kβ Filtering and Receiving Slits Configuration

This section presents a comparison of XRD measurements of the same sample acquired under three conditions: without a Kβ filter, with a Kβ filter using identical slit settings, and with a Kβ filter combined with optimized settings for Receiving Slit #1 and Receiving Slit #2. The comparison clearly demonstrates that the highest diffraction intensity is achieved when the Kβ filter is used together with the optimized slit configuration, specifically Receiving Slit #1 at 2 mm and Receiving Slit #2 at 5.025 mm. Notably, this optimized filtered setup yields significantly higher peak intensities than the measurement performed without the Kβ filter. The results highlight the synergistic effect of spectral purification and slit optimization, showing that both intensity and resolution can be systematically tuned to meet specific experimental requirements. Consequently, the XRD system can be efficiently configured to deliver high-quality, application-specific data for a wide range of research needs.

Sample name :
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : ----

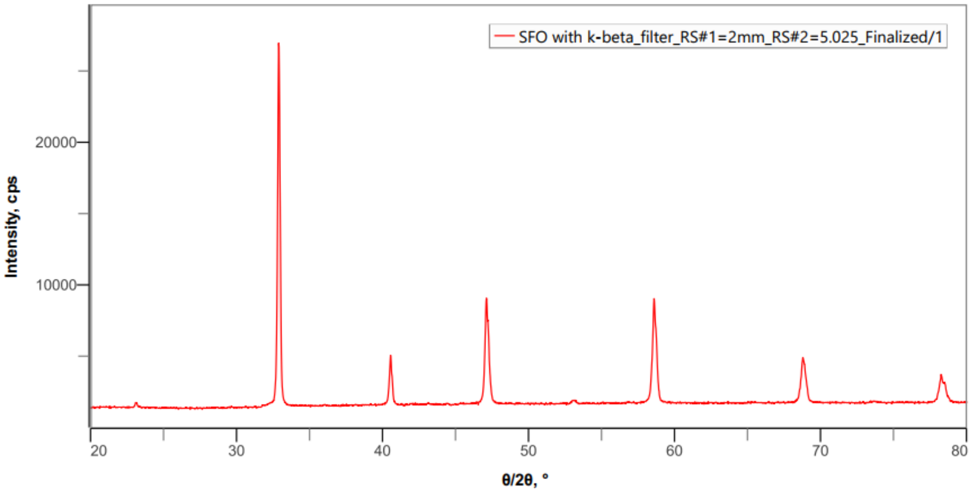
Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : ----
Filter 1 : ----
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : ----
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



Sample name :
Comment :
Date of measurement : 2025-12-17 11:38:15
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : 1D(scan)
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : 1.0000 °/min

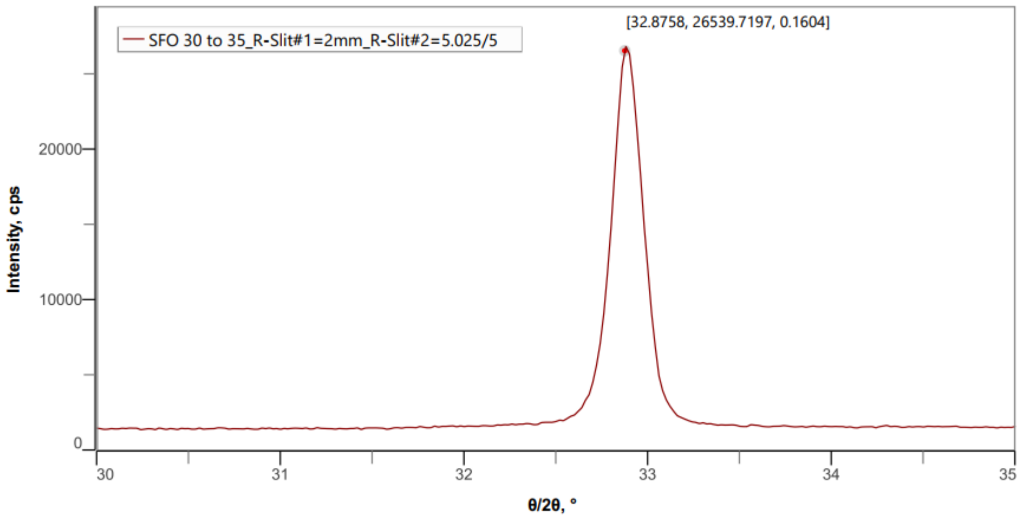
Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 2.000mm
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 5.025mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



Sample name :
Comment :
Date of measurement : 2025-12-17 10:37:05
Operator name : Administrator
X-ray : 40 kV, 45 mA
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 :

Scan axis : θ/2θ
Scan mode : 1D(scan)
Scan range : 30.0000 - 35.0000 °
Scan step : 0.0200 °
Scan speed : 1.0000 °/min

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 2.000mm
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 5.025mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None



Inference**:** Optimal XRD performance is achieved by combining Kβ filtering with carefully optimized receiving slits width, resulting in higher intensity and cleaner diffraction data than unfiltered measurements.

Effect of Scan Speed on XRD Data Quality and Measurement Throughput

A comparison of XRD scans acquired at 1, 2, and 4°/min demonstrates that slower scan speeds yield improved data quality due to longer counting times per step, but this comes at the expense of increased total scan duration, approximately 18 minutes at 4°/min, 36 minutes at 2°/min, and 72 minutes at 1°/min for the 10–80° 2θ range. Importantly, the number and angular positions of the data points are independent of scan speed and are determined solely by the selected step size. Extended measurement times may affect sensitive samples, increase instrument load, and reduce overall laboratory throughput. Therefore, the scan speed must be chosen by balancing the desired data quality against practical constraints on measurement time and system utilization.

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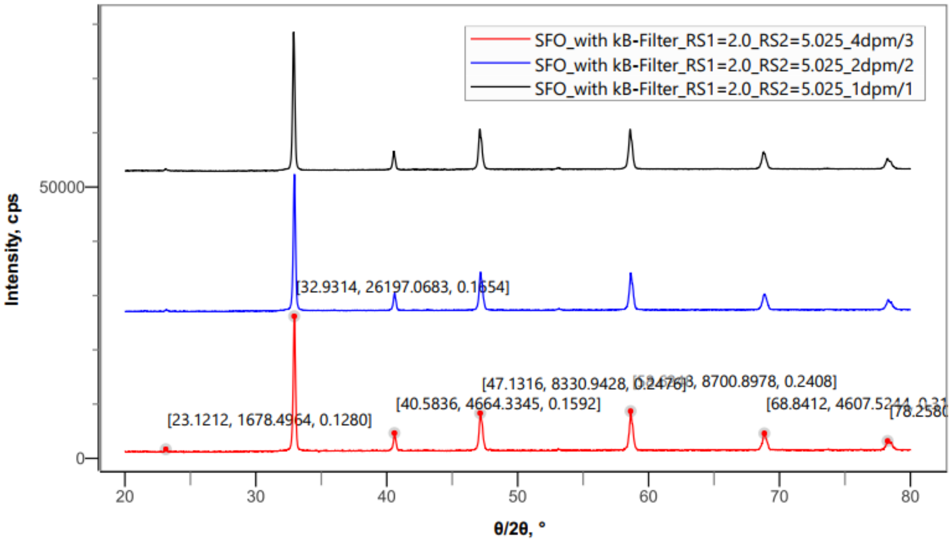
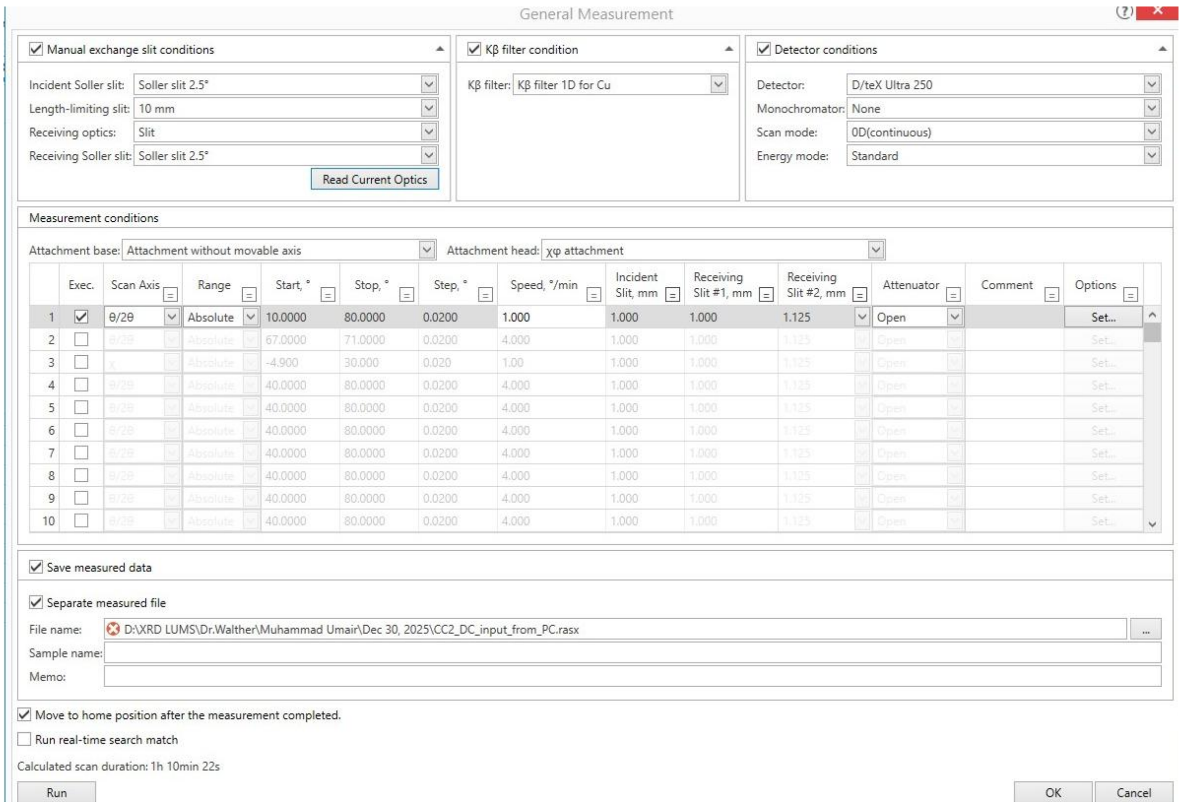
Sample name :
Comment :
Date of measurement : ----
Operator name : Administrator
X-ray : 40 kV, 45 mA
Wavelength : Cu-Kα / 1.54186 Å
Goniometer : Standard Goniometer
Attachment base : Standard attachment
Attachment head : Standard attachment head
Detector : D/teX Ultra 250
Optics attribute : BB
Memo :

Scan axis : θ/2θ
Scan mode : ----
Scan range : 20.0000 - 80.0000 °
Scan step : 0.0200 °
Scan speed : ----

Selection slit : BB
Incident optics unit : IPS adaptor
Incident Soller slit : Soller slit 2.5°
Incident slit box : 1deg
Length-limiting slit : 10 mm
Receiving slit box #1 : 2.000mm
Filter 1 : Kβ filter 1D for Cu
Receiving optics unit #1 : No unit
Receiving Soller slit : Soller slit 2.5°
Receiving slit box #2 : 5.025mm
Receiving Attenuator : Open
Detector monochromator : None
Detector slit : None





☑ Manual exchange slit conditions

Incident Soller slit: Soller slit 2.5°
Length-limiting slit: 10 mm
Receiving optics: Slit
Receiving Soller slit: Soller slit 2.5°

☑ Kβ filter condition

Kβ filter: Kβ filter 1D for Cu

☑ Detector conditions

Detector: D/teX Ultra 250
Monochromator: None
Scan mode: 0D(continuous)
Energy mode: Standard

Measurement conditions

Attachment base: Attachment without movable axis
Attachment head: χφ attachment

	Exec.	Scan Axis	Range	Start, °	Stop, °	Step, °	Speed, °/min	Incident Slit, mm	Receiving Slit #1, mm	Receiving Slit #2, mm	Attenuator	Comment	Options
1	☑	θ/2θ	Absolute	10.0000	80.0000	0.0200	1.000	1.000	1.000	1.125	Open		Set...
2	☐	θ/2θ	Absolute	67.0000	71.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
3	☐	χ	Absolute	-4.900	30.000	0.020	1.00	1.000	1.000	1.125	Open		Set...
4	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
5	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
6	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
7	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
8	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
9	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...
10	☐	θ/2θ	Absolute	40.0000	80.0000	0.0200	4.000	1.000	1.000	1.125	Open		Set...

☑ Save measured data

☑ Separate measured file

File name: D:\XRD LUMS\Dr.Walther\Muhammad Umair\Dec 30, 2025\CC2_DC_input_from_PC.rasx
Sample name:
Memo:

☑ Move to home position after the measurement completed.

☐ Run real-time search match

Calculated scan duration: 1h 10min 22s

Handling of Kβ Radiation (SmartLab SE / SmartLab Studio II)

When operating the SmartLab SE in Bragg–Brentano geometry without a β-filter or monochromator, Kβ diffraction peaks may appear due to the broader spectral acceptance of the optical configuration, while parallel beam optics inherently suppress Kβ contributions. The intensity of Kβ peaks in BB geometry is sample-dependent and influenced by composition, absorption, crystallinity, diffracting volume, background, and secondary effects.

- For **quantitative or high-precision analyses** (PDF-based phase identification, lattice-parameter determination, Rietveld refinement, quantitative phase analysis, residual stress, rocking curves, or reciprocal space mapping), Kβ radiation **must be removed or suppressed** using appropriate optics or software-based Kβ stripping, as Kβ peaks are not included in standard reference databases and may cause misidentification or refinement instability.

- For **qualitative,** exploratory, instructional, or rapid screening measurements, Kβ peaks may be safely ignored because these analyses rely on the top few strongest Kα peaks for phase identification. Users should ensure that Kβ peaks are recognized and do not overlap with critical Kα reflections that are required for correct interpretation.