Perfect Powder Diffraction Data Automatically:

Dynamic Beam Optimization

Welcome to today's webinar from our Bruker AXS office in Karlsruhe, Germany!

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Outline

Dynamic Beam Optimization

- Origins of parasitic scattering in XRPD data
- Minimizing parasitic scattering: Dynamic Beam Optimization

Application examples

Instrument considerations

- Supported instrument configurations
- Instrument upgrades

Wrap-up

- 3 Things to remember
- Question & Answers

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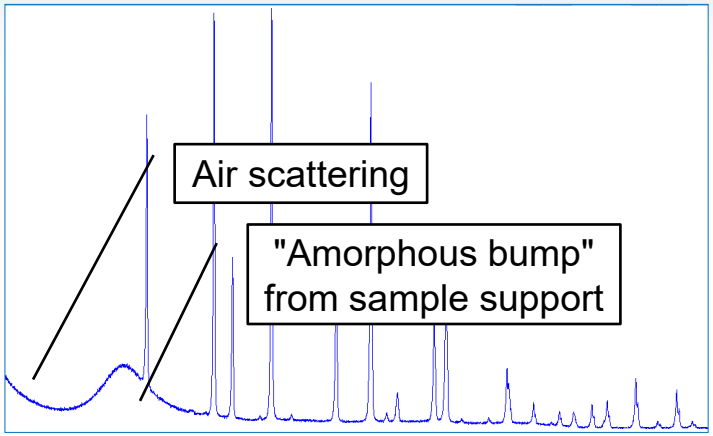
The Source of Parasitic Scattering...
... and its Consequences

- Parasitic Scattering makes data analysis harder and can lead to incorrect results.
- For good data, we want to minimize or completely avoid scattering that does not come from the sample.

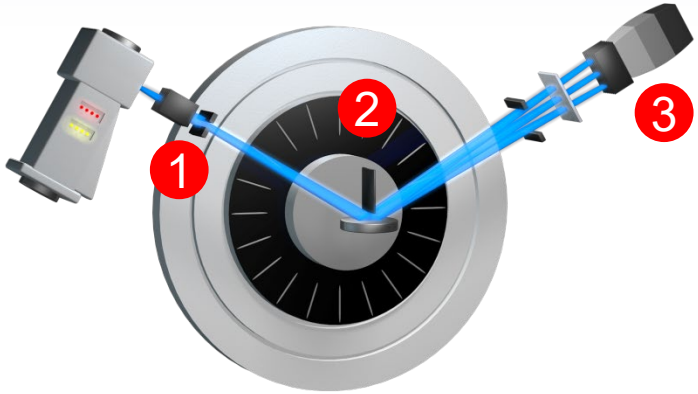


Main contributions	Typical phenomena	Analytical Consequences
Beam overflow	(Broad) peaks from sample holder	Incorrect relative intensities; Incorrect amorphous content
Air-scattering	High background at low and medium angles	Miss low angle peaks, wrong phase-ID; worse LOD* and LOQ**
Direct beam spill	Very high background at very low angles	Difficult to differentiate between SAXS and parasitic signal
Fluorescence	Background is high	Worse LOD and LOQ

* Limit of Detection ** Limit of Quantification

Parasitic Scattering
Traditional Beam Conditioning

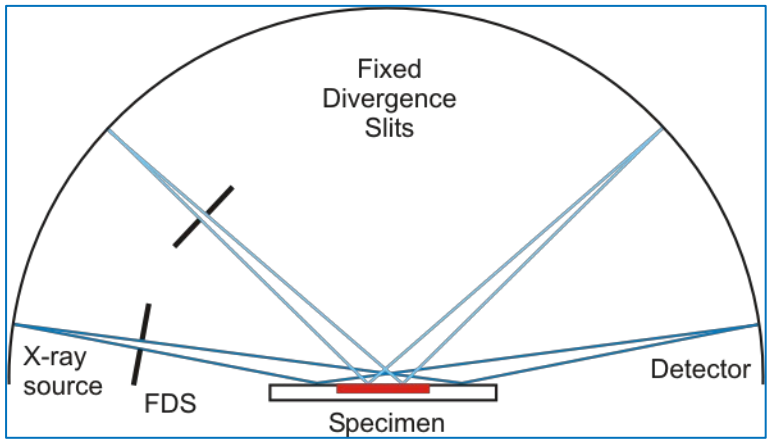
- Beam spillover and air-scattering at low angles can be avoided by choosing appropriate diffractometer settings
- But optimizing the set-up for low angles can lead to detrimental negative effects at higher angles
- In reality, we often live with a compromise



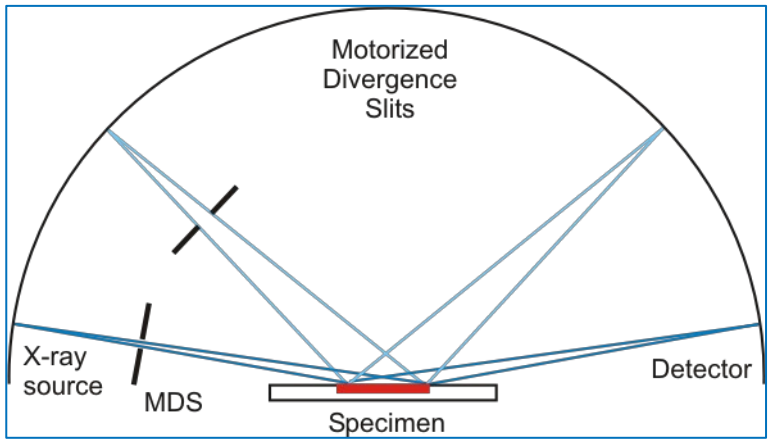
Component	Low angles
1. Divergence slit size	Avoid beam-spill → **Small** slit
2. Knife edge position	Shield air scattering → Knife **close** to sample
3. Detector opening	Shield air scattering → **Small** window

Dynamic Beam Optimization
Fixed vs. Motorized Divergence Slits

- Fixed Divergence Slits (FDS):
  - Beam spillover at low angles 2θ, therefore scattering from the sample support and intensity losses at small angles 2θ
- Motorized Divergence Slits (MDS):
  - Fixed illuminated specimen area, therefore NO beam spillover and NO scattering from the sample support; higher intensity



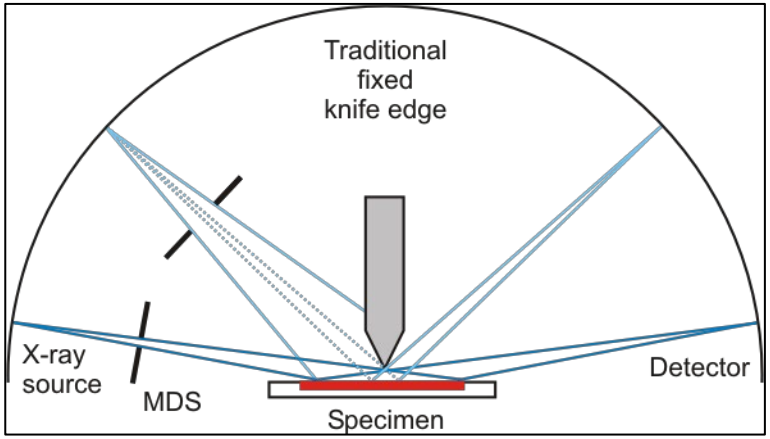
Beam spillover
at low angles 2θ



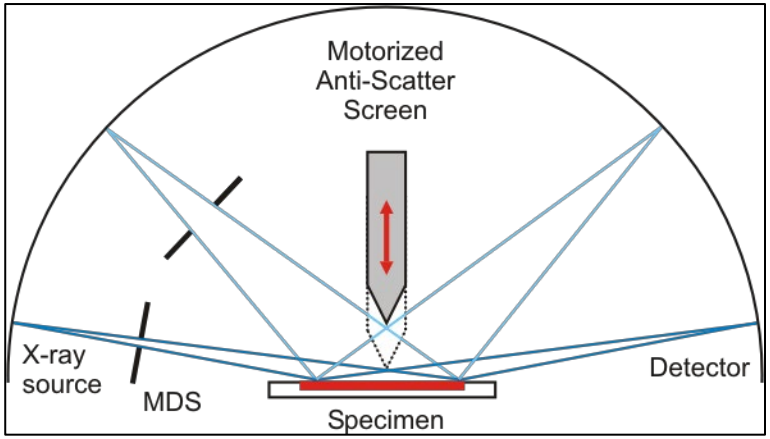
No beam spillover,
no scatter from the sample support

Dynamic Beam Optimization
Fixed vs. Motorized Anti-Scatter Screen

- Traditional fixed knife edge
  - Placed as close to the beam as possible, risk of beam cropping
- Motorized Anti-Scatter Screen
  - Fully software controlled retraction of the knife, thus air scattering is almost entirely eliminated without ANY cropping of the beam
  - The Motorized Anti-Scatter Screen is THE component providing the greatest improvement on data quality



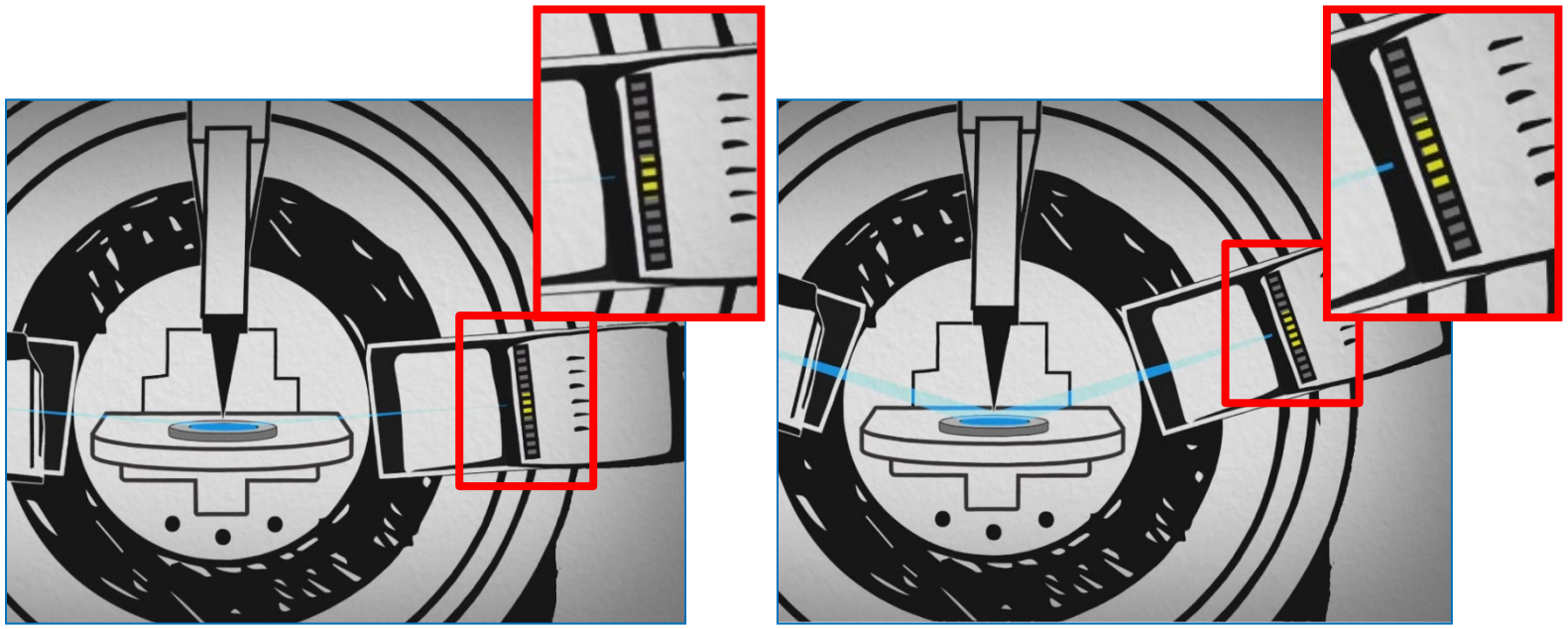
The beam is increasingly cropped as 2θ increases



No beam cropping

Dynamic Beam Optimization
Variable Active Detector Window

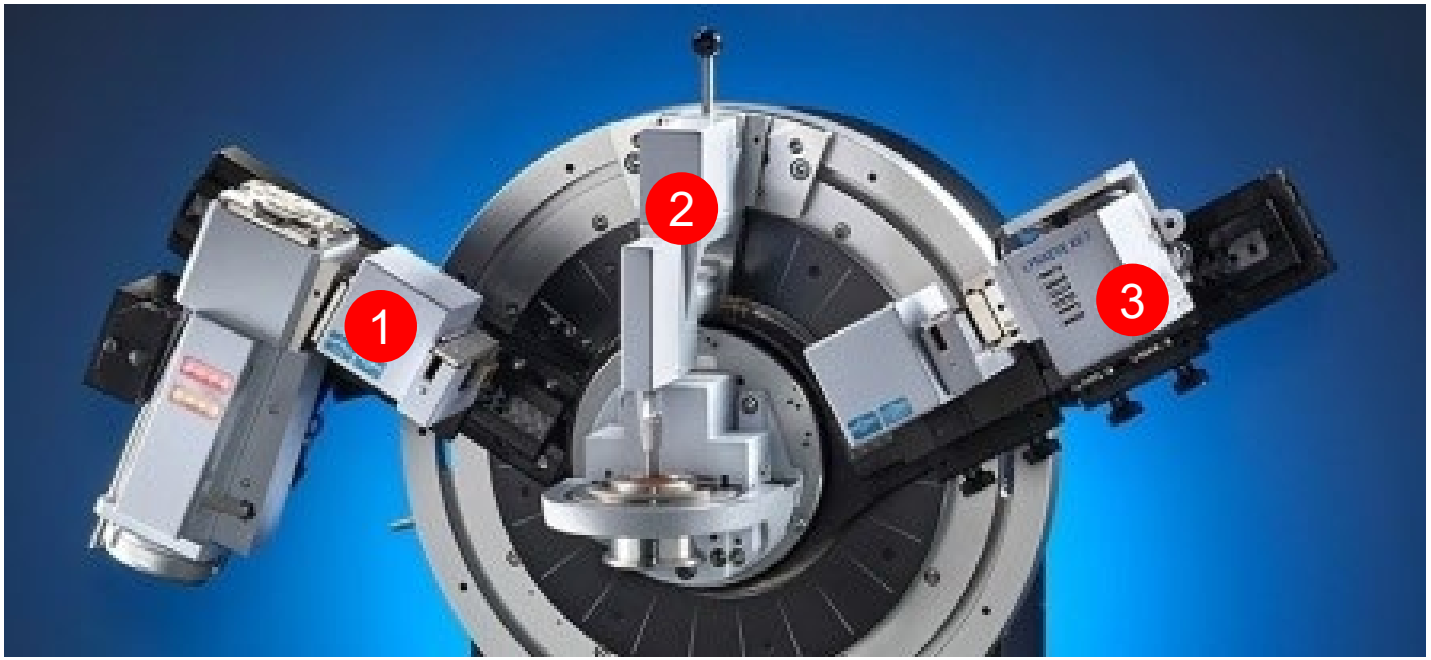
- Fully software controlled switching-on of individual strips to "open" the active detector window as a function of 2θ, to further minimize air scattering
- Effective for data acquisition at angles <5° 2θ.
  - ⇨ Reliable data acquisition starting at angles as low as ~ 0.3° 2θ



Dynamic Beam Optimization

Fully <u>software controlled synchronization</u> of

1. Motorized Divergence Slits,
2. a Motorized Anti-Scatter Screen,
3. and a Variable Active Detector Window



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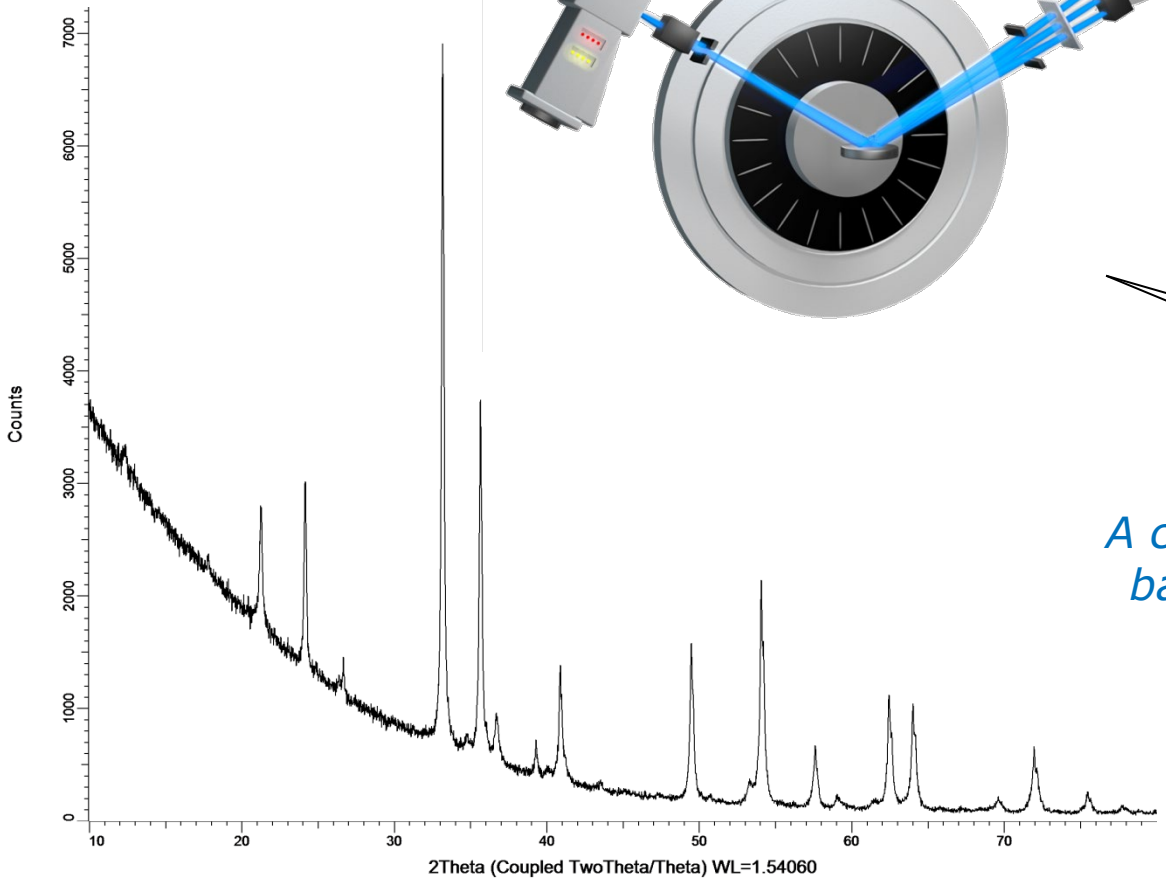
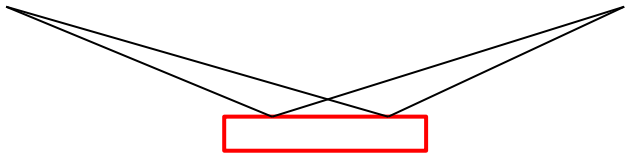
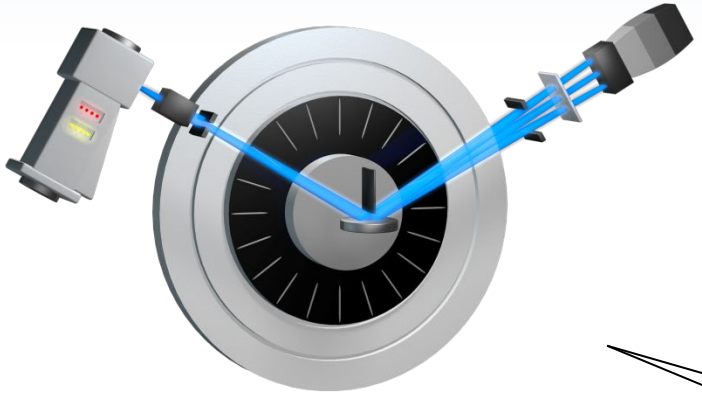
Background Optimization
High background – air scattering

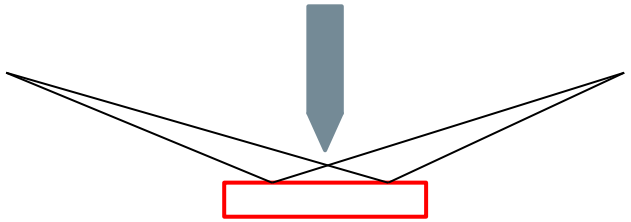




A common source of elevated background comes from air scattering…

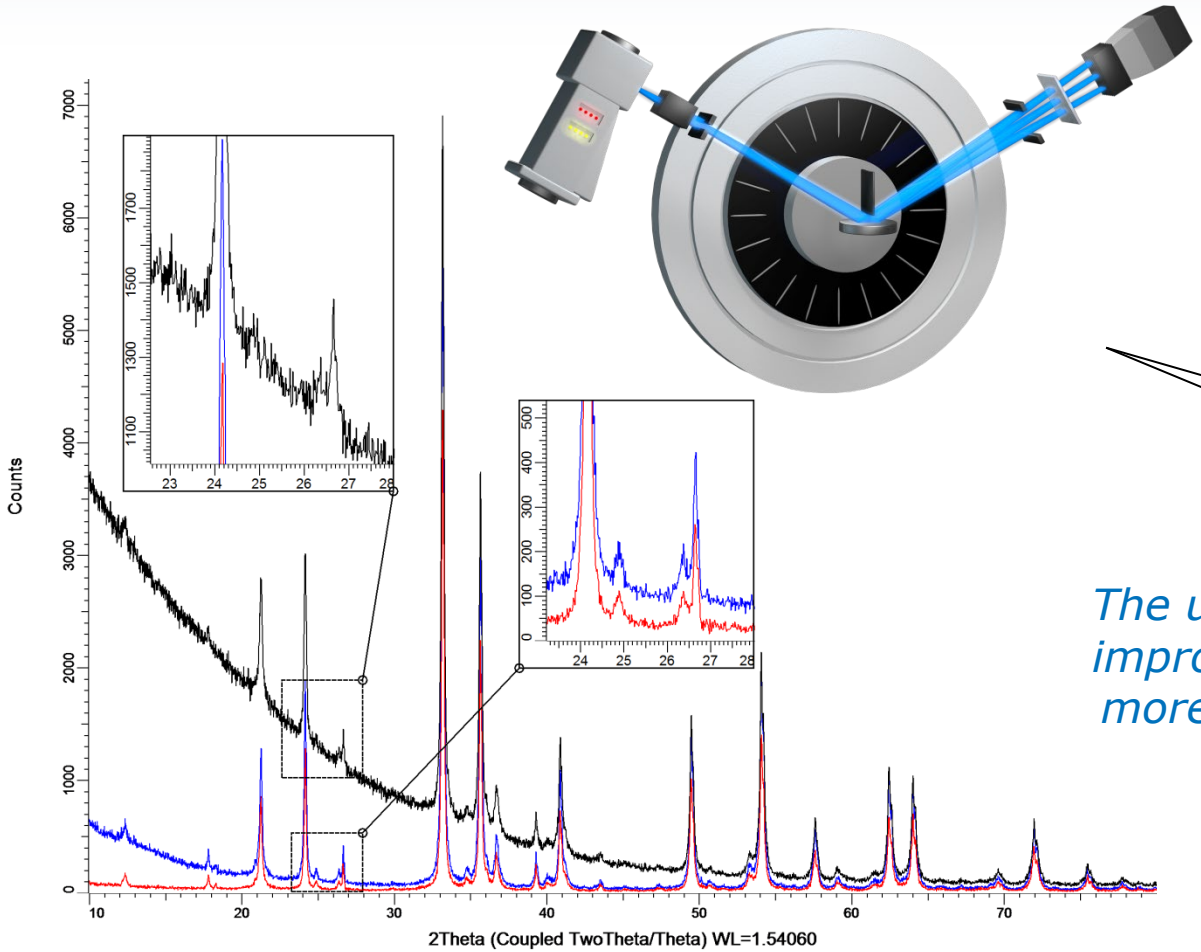
Background Optimization
High background – air scattering

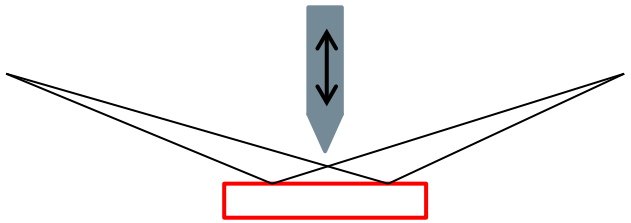




By placing a fixed knife 5mm above the sample, the background level improves dramatically

Background Optimization
High background – air scattering

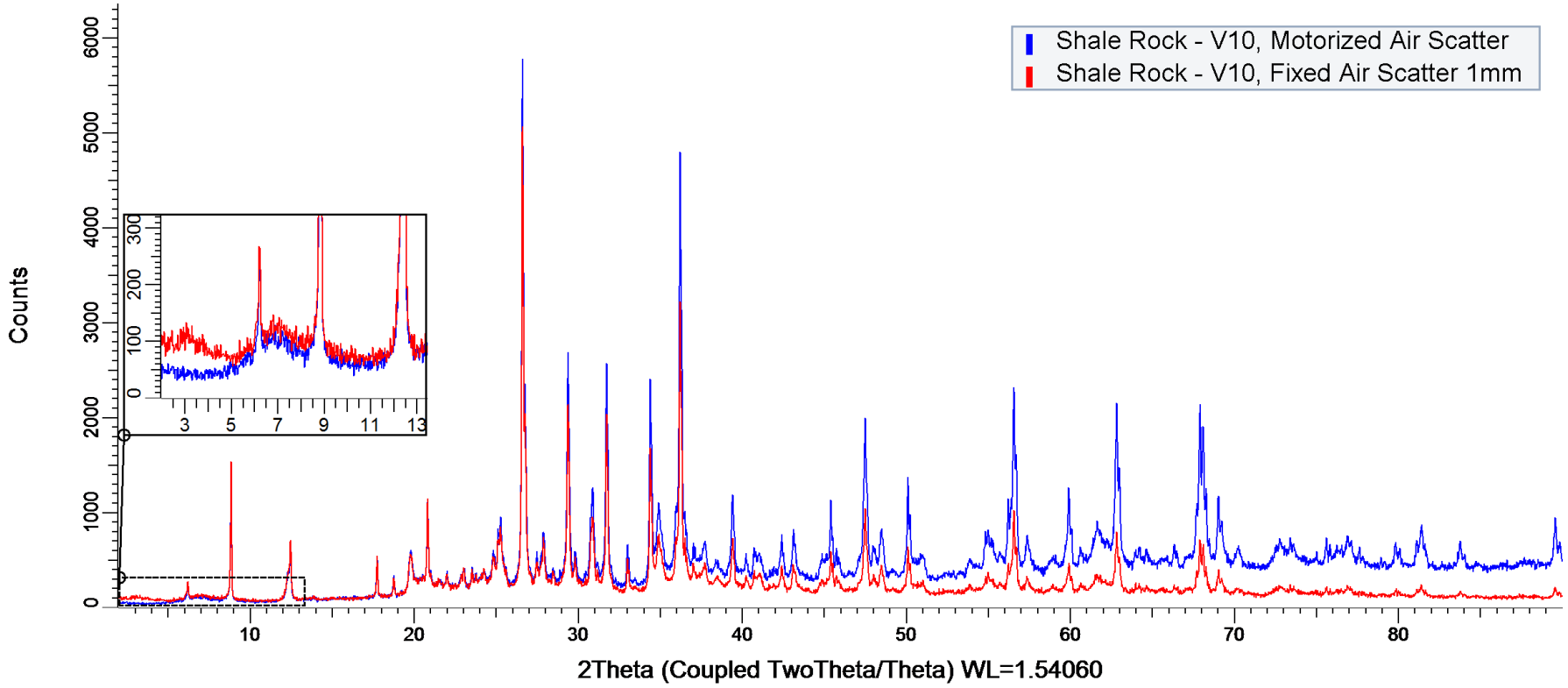




The usage of a motorized knife improves the background even more, especially at low angles

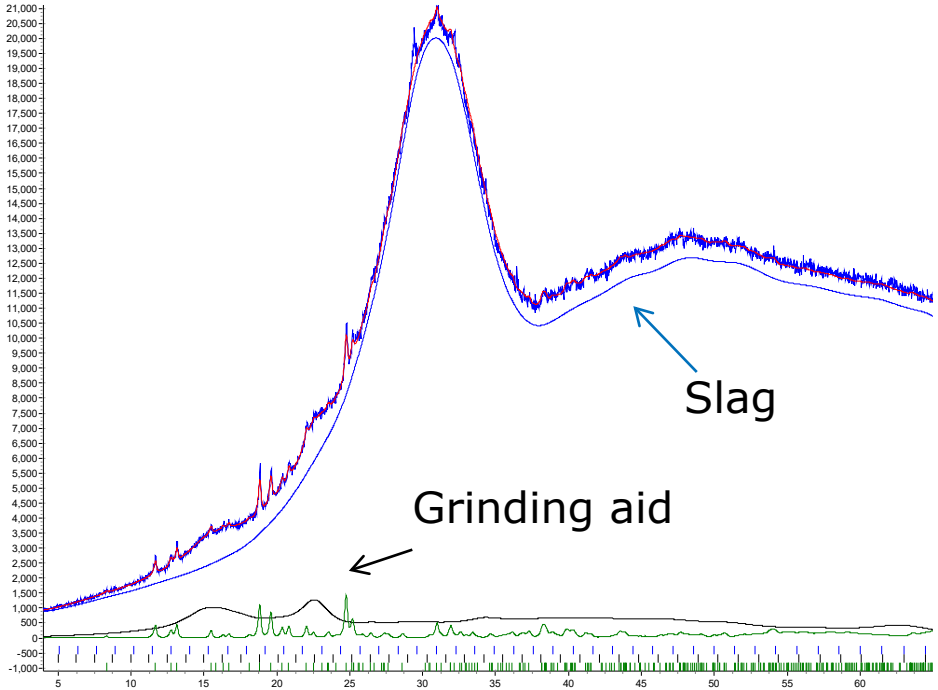
Quantitative Mineralogy
Shale Rock

- Comparison of fixed slit and motorized slit
- With MASS: improved low angle background, and no cropping of beam at high angles

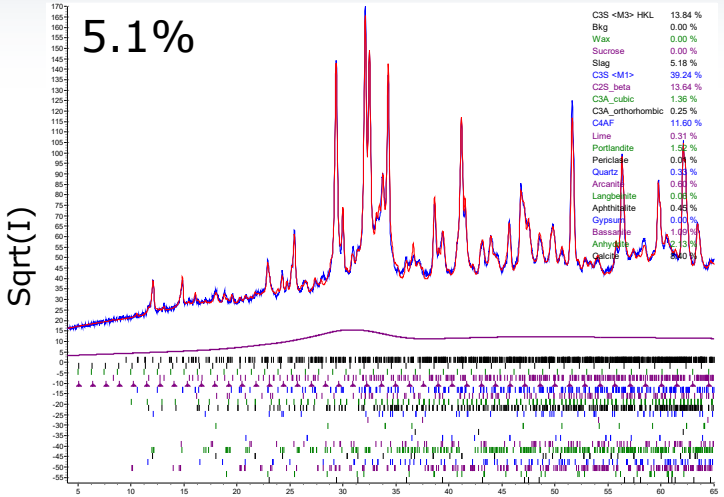


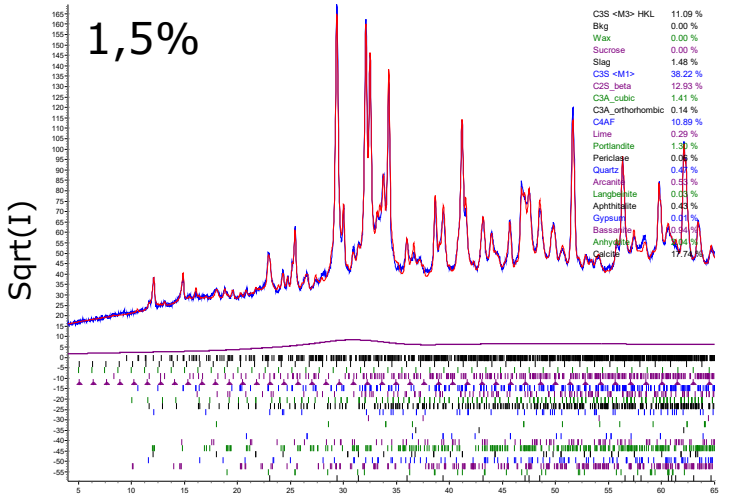
Quantification of amorphous content
5% and 1.5% Slag in Cement

- Quantification of amorphous components is challenging
- DBO ensures that signal is coming only from sample

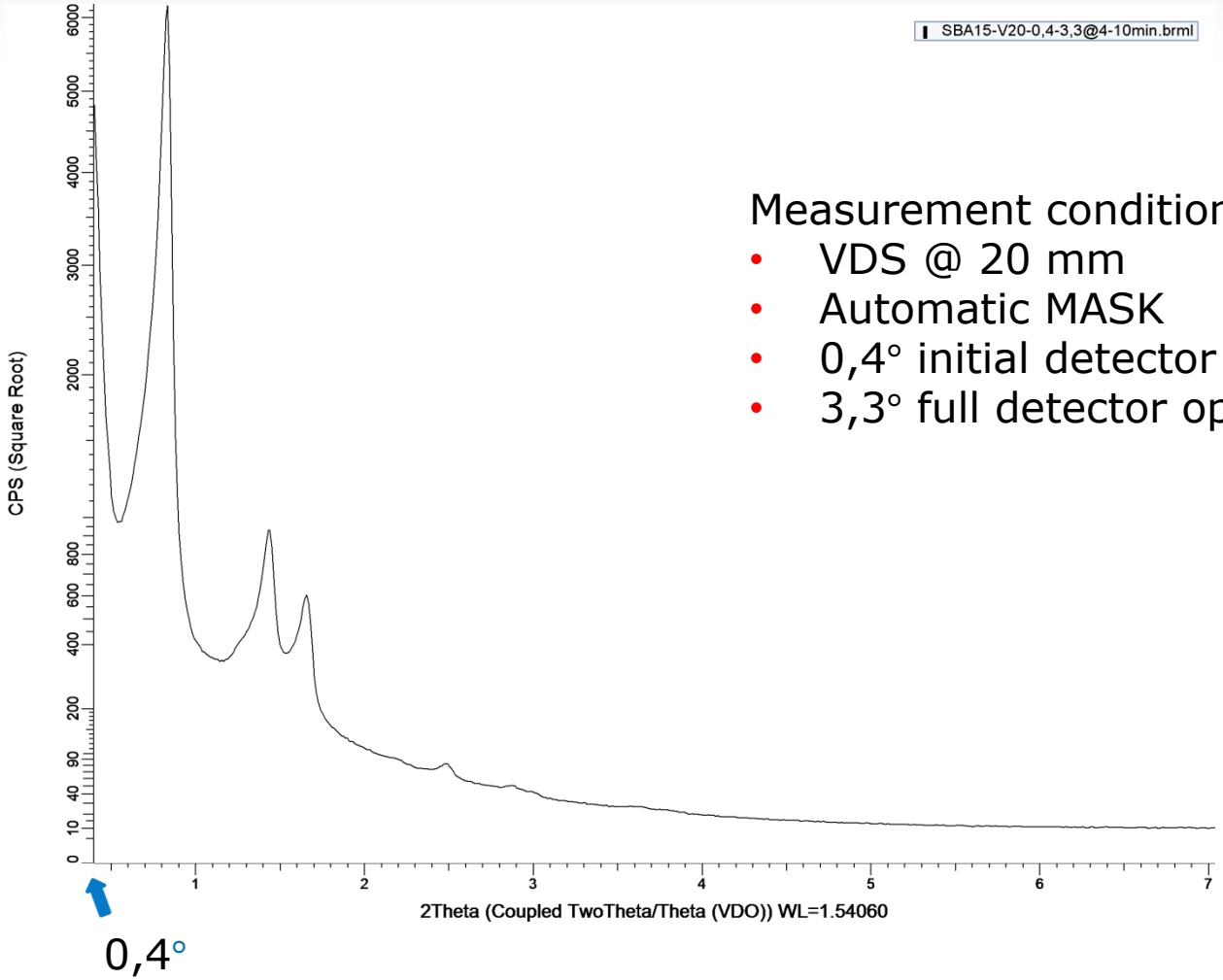


Fit of slag sample in DIFFRAC.TOPAS





Very low angle measurement using VDO
SAXS signal in SBA-15



Measurement conditions

- VDS @ 20 mm
- Automatic MASK
- 0,4° initial detector opening
- 3,3° full detector opening @ 2θ=4°

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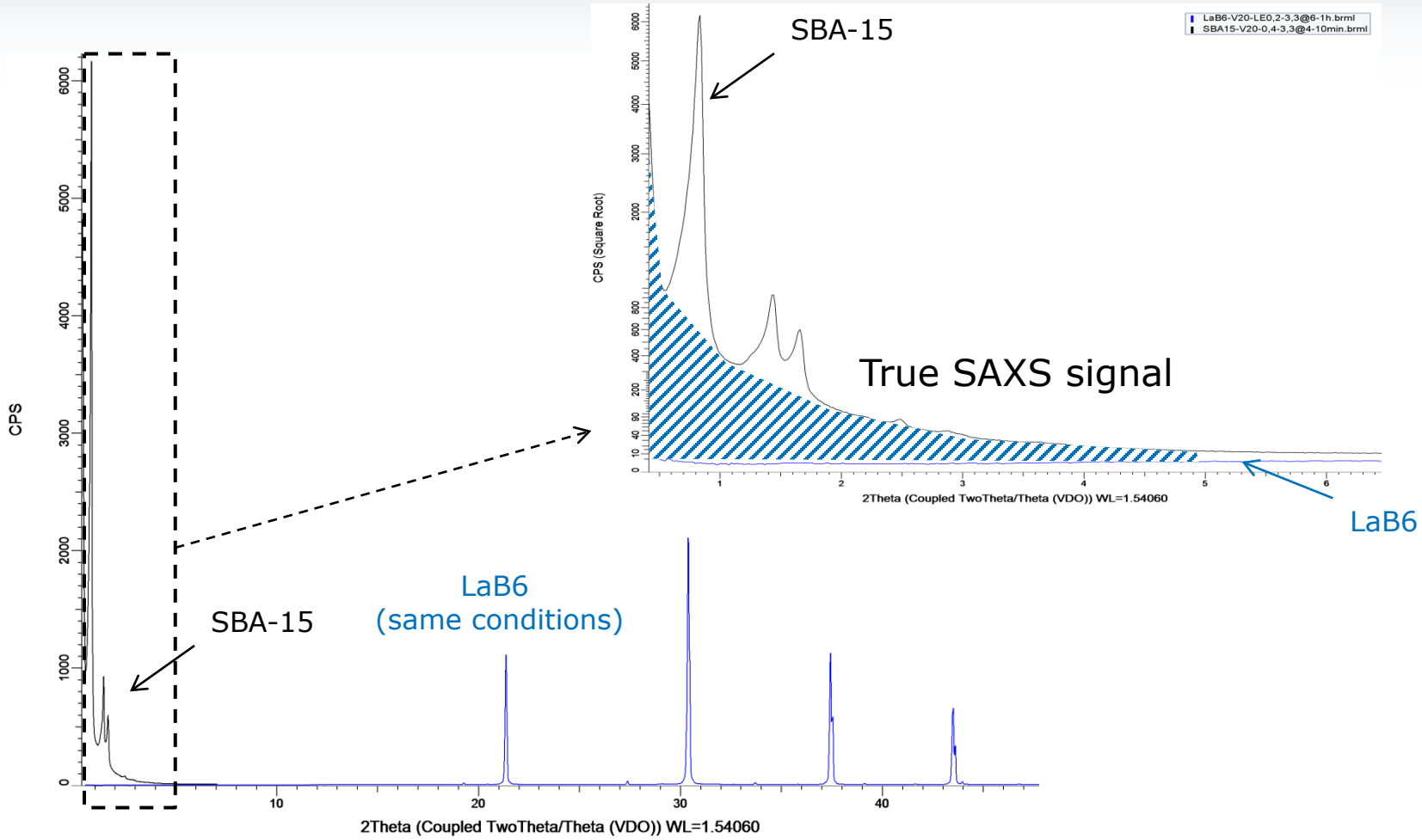
SAXS signal in SBA-15



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Dynamic Beam Optimization
Supported Instrument Configurations

Supported instruments:

- D8 ENDEAVOR, D8 ADVANCE, D8 DISCOVER

Dynamic beam components:

- Motorized Divergence Slits
- Motorized Anti-Scatter Screen
- Variable Active Detector Window

⇨ Fully software controlled / synchronized, no user-intervention required

⇨ Not all 3 components are required. All dynamic beam components may also be operated independently (unsynchronized) or in any dual combination (synchronized) - at the user's discretion

Dynamic Beam Optimization
Upgrades

Dynamic beam components:

- Motorized Divergence Slits
  - Most instruments are already equipped with motorized slits
  - Upgrade recommended when beam spillover is an issue or to reduce measurement time
- Motorized Anti-Scatter Screen
  - The core component of Dynamic Beam Optimization with the highest benefit in terms of data quality
  - "Must have" to effectively minimize air scattering, specifically when using Motorized Divergence Slits
- Variable Active Detector Window
  - Recommended for data acquisition at angles <5° 2θ only
  - Always included with the SSD160-2, LYNXEYE-2, LYNXEYE XE, LYNXEYE XE-T and EIGER2 R 500K detectors. No upgrade required.
    - Not available for SSD-160 and LYNXEYE detectors

Please contact your local Bruker AXS office
or contact us via info.baxs@bruker.com

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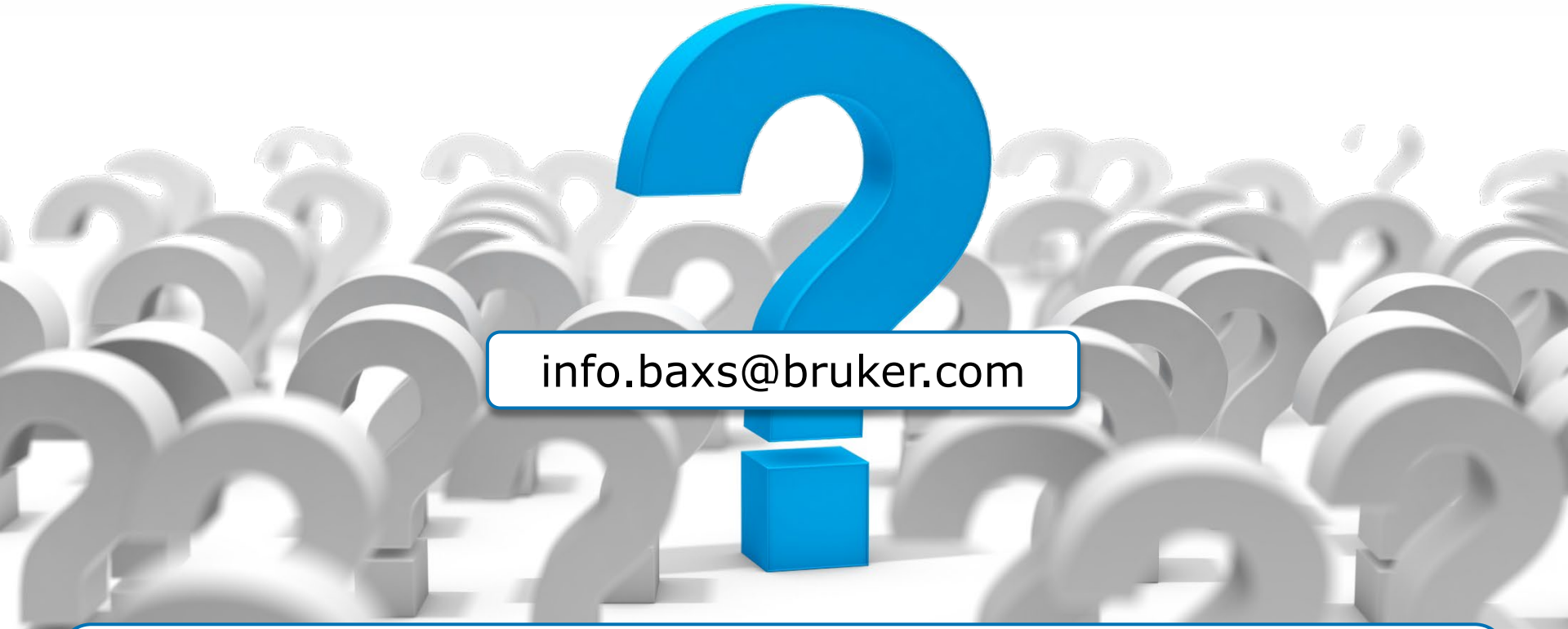
Dynamic Beam Optimization
3 Things to Remember

1. Dynamic beam optimization allows to acquire data virtually free of air, instrument, and sample support scatter

2. Dynamic beam optimization significantly enhances lower limits of detection and enables accurate quantification of minor crystalline and amorphous phases

3. Dynamic beam optimization delivers unparalleled data quality for materials with low angle peaks (large d-spacings) such as clays, pharmaceuticals, zeolites, and porous framework materials

Any Questions?



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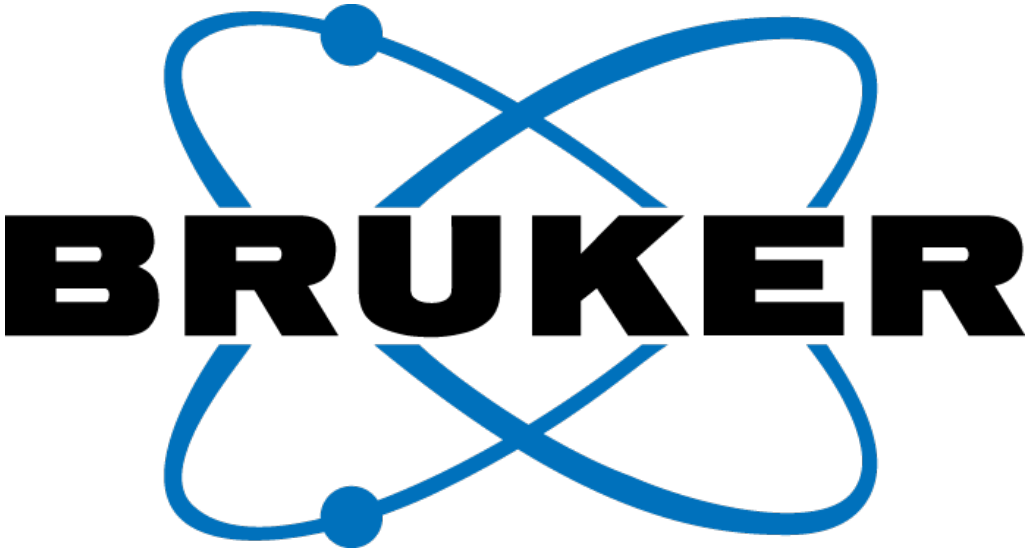
May 25th, 1:30 to 3pm (CET)



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