

NEW POSSIBILITIES FOR X-RAY DIFFRACTOMETRY

Bernd Hasse – Incoatec, Geesth

Incoatec Microfocus Source – $I\mu S$

The source

- Point focus sealed tube
- Cu, Mo, Cr, or Ag anode
- High brilliance
- Low power: 30 W
- Air-cooled
- Tube change as easy as for conventional sealed-tubes
- 3 Years warranty

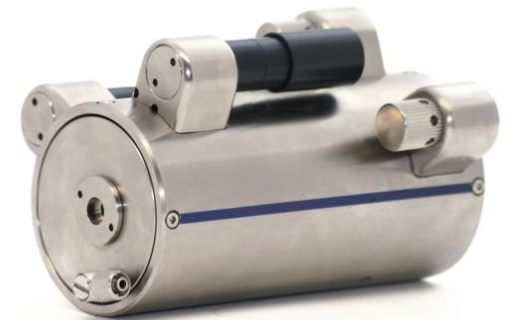
Bright sealed tube for ultimate convenience



Incoatecs Microfocus Source – μS

The optics and housing

- New family of 2D beam shaping
Montel Optics: **The Quazar Optics**
- 2D focusing or collimating or hybrid (f+c)
- Patented housing with optional motors
- Stable positioning



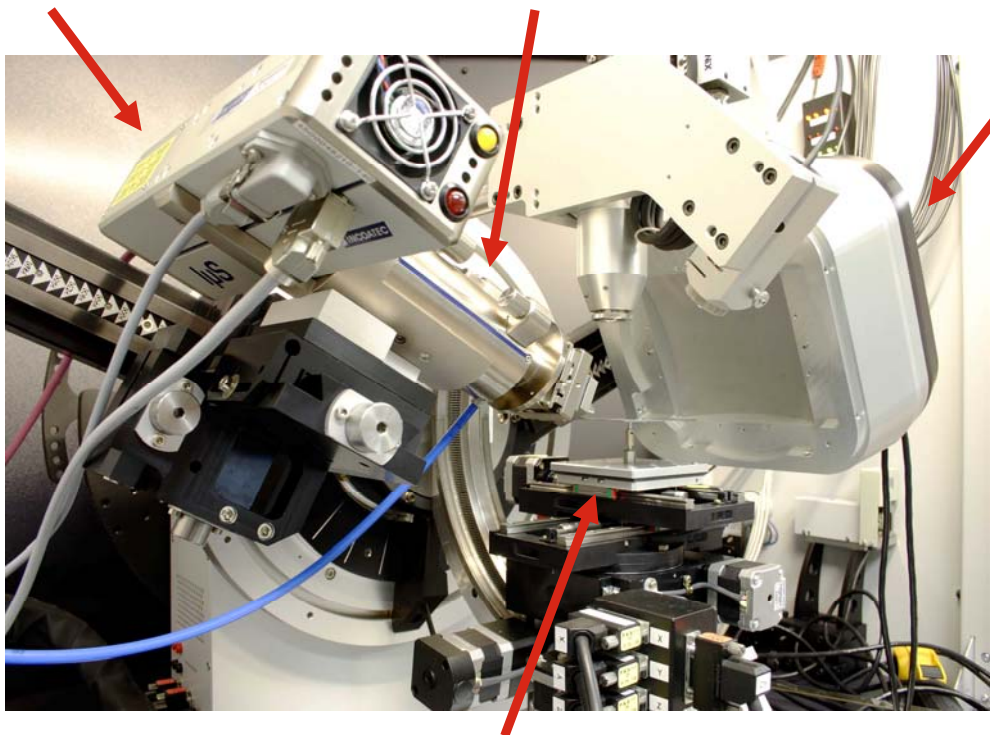
Optimized Optics in Incoatec's new very stable and easy-to-align housing

New Equipment for Diffractometry

Microfocus Source
 μS

Optics with
Housing

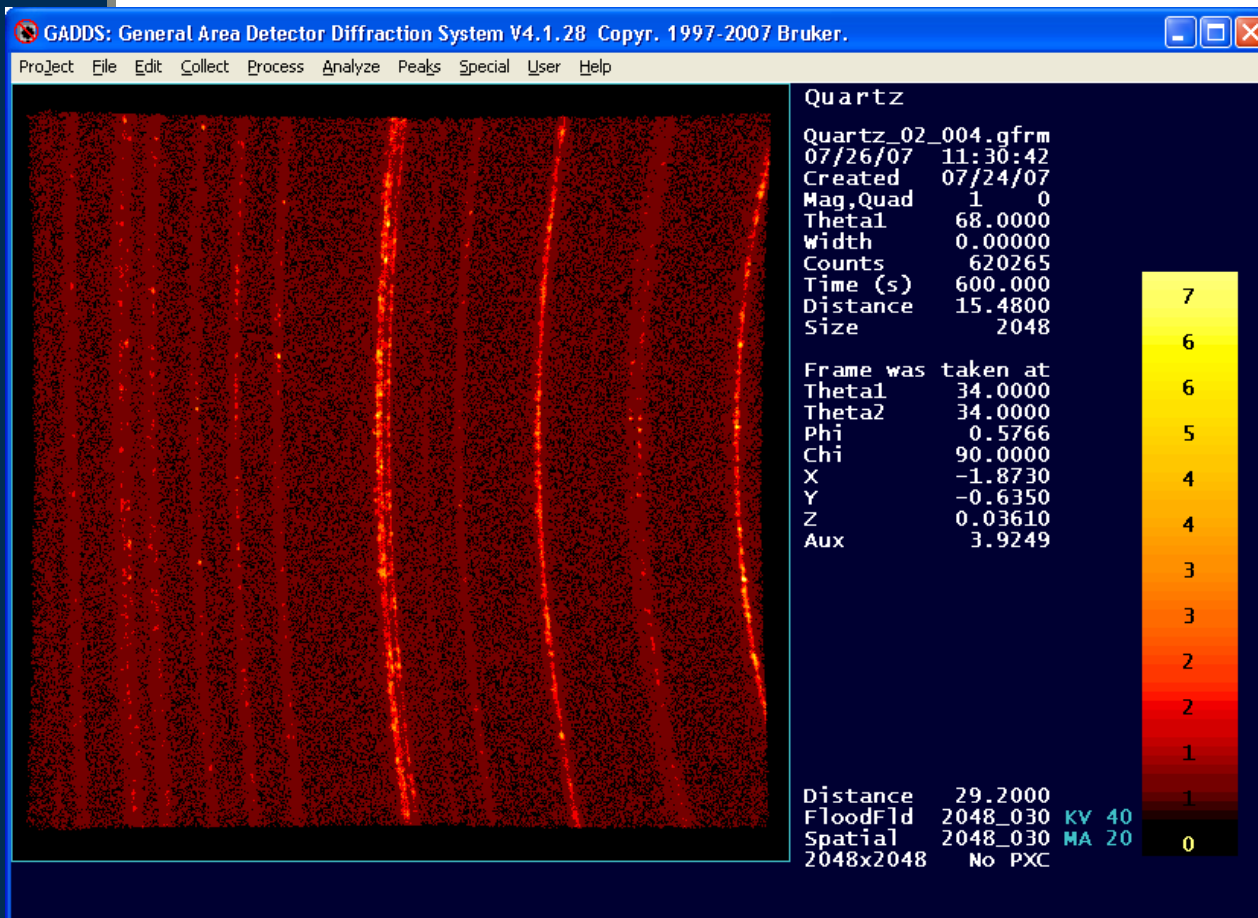
2D detector
(VÅNTEC 2000)



Sample stage

**Bruker D8 GADDS
with μS^{TM}**

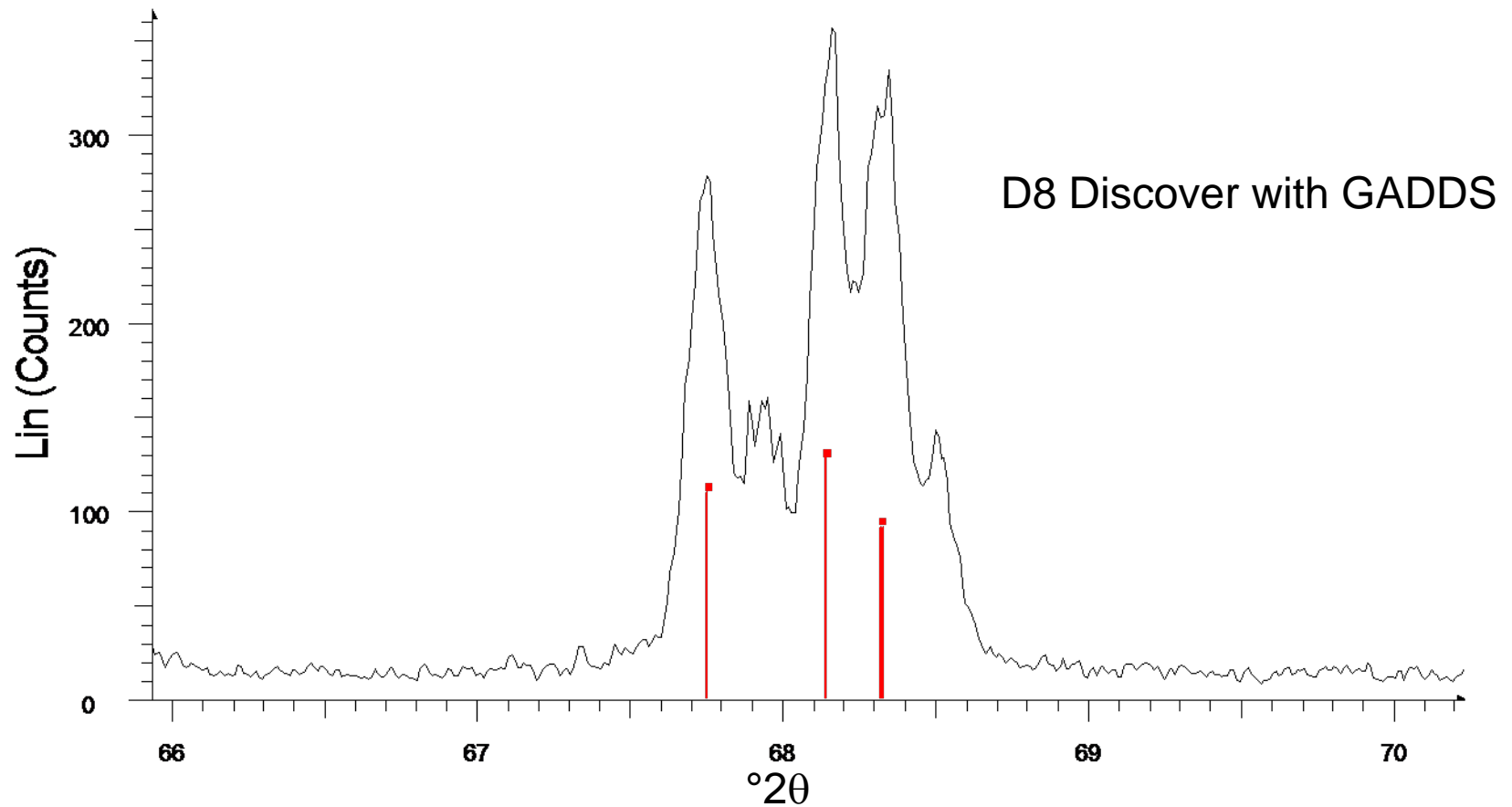
I μ S™ & VANTEC-2000



- symmetrical reflection
- 600 sec collection time
- sample to detector 15 cm

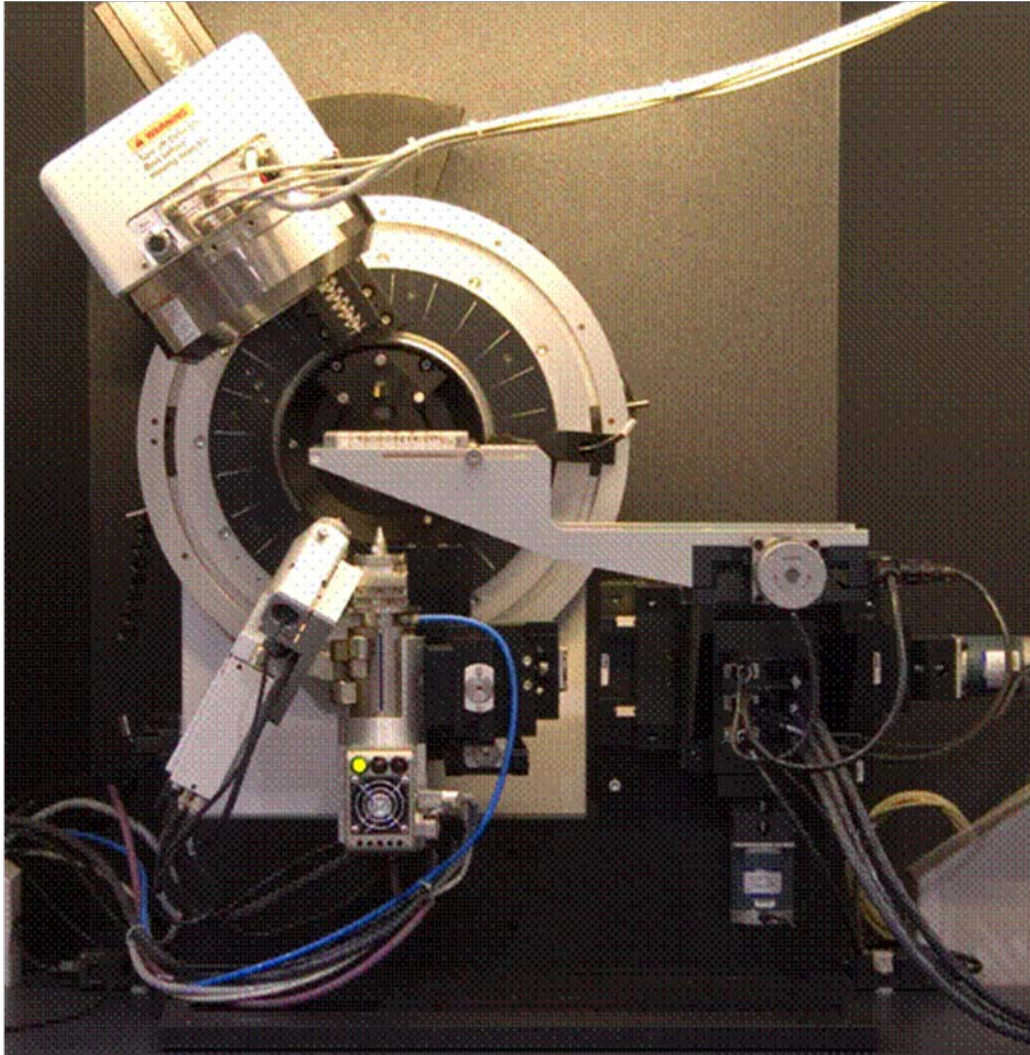
Sample: quartz powder

μS^{TM} & VANTEC-2000



The five fingers of quartz

$I\mu S^{\text{TM}}$ for XRD with focusing optics



- measurements in transmission geometry
- with Bruker D8 GADDS and VÅNTEC 2000

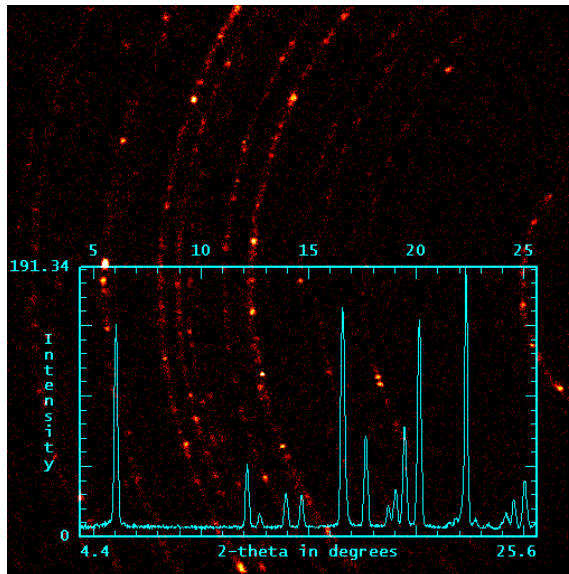
μ S (Cu-K α) with focusing optics vs. classical setup

Ibuprofen / measured in transmission geometry / Sample-Detector distance 290 mm

Sealed Tube – 1D collimating optics

- 0.3 mm collimator

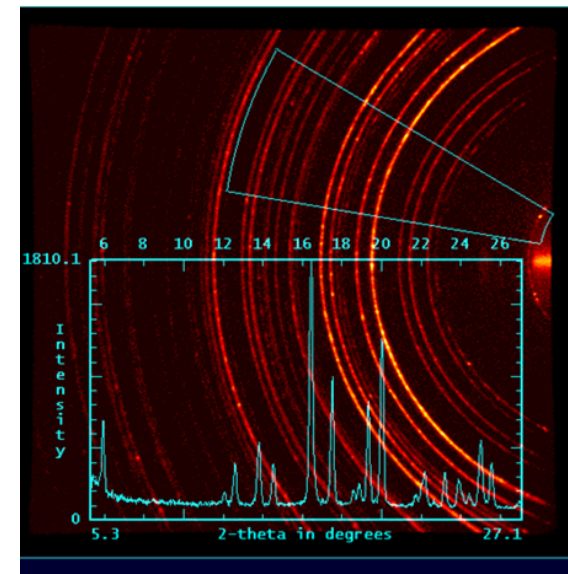
120 sec collection time



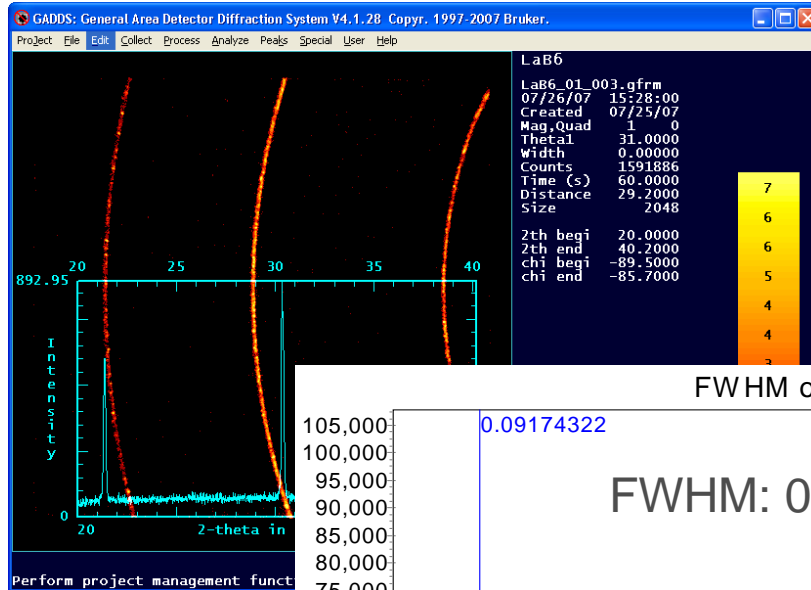
μ S – 2D focussing optics

- Spotsize 230 μ m
- 0.3 mm snout
- small slice for integration to obtain better resolution (poor detector calibration)

15 sec collection time

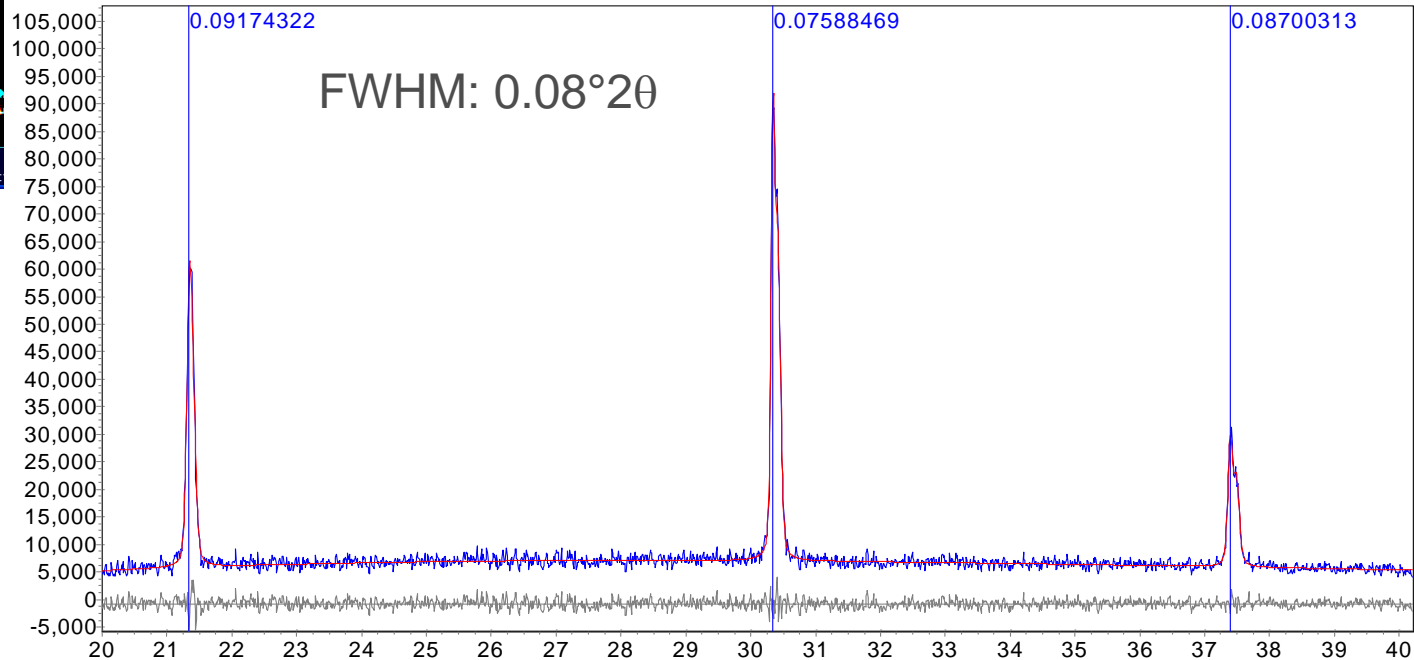


μ S™ focusing onto the detector: LaB₆

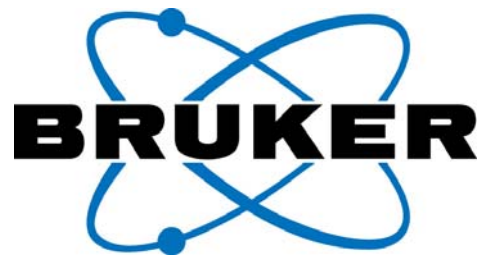
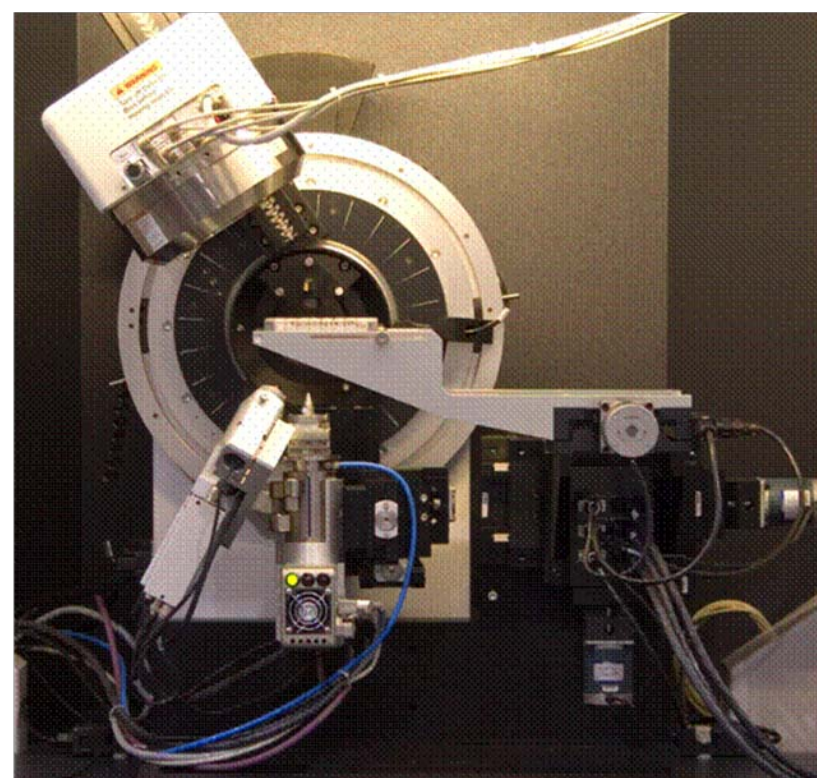
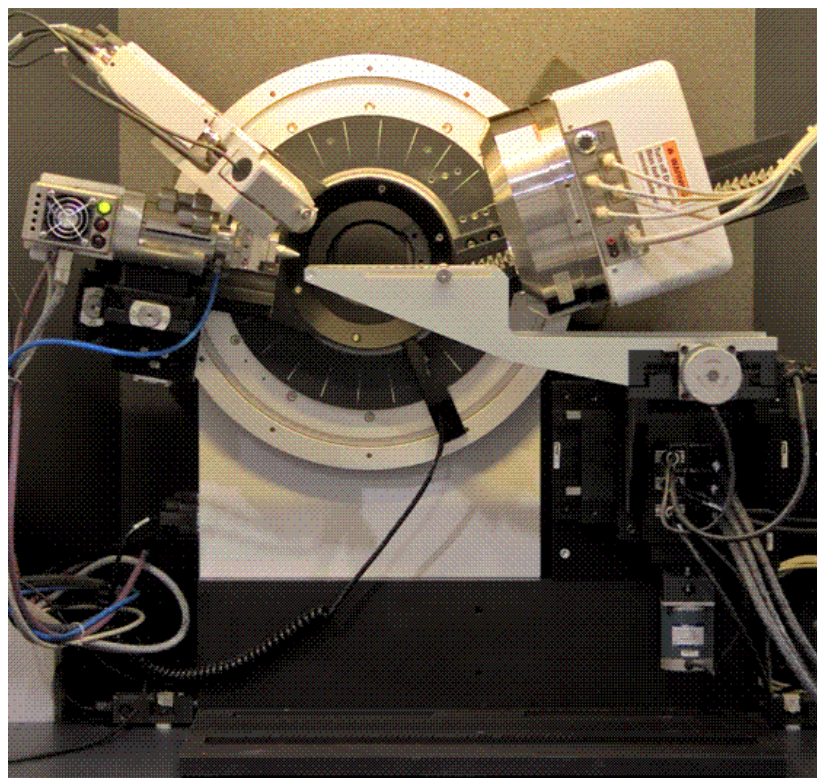


60 seconds,
292 mm sample-detector

FWHM of LaB6 sample fitted with PV function

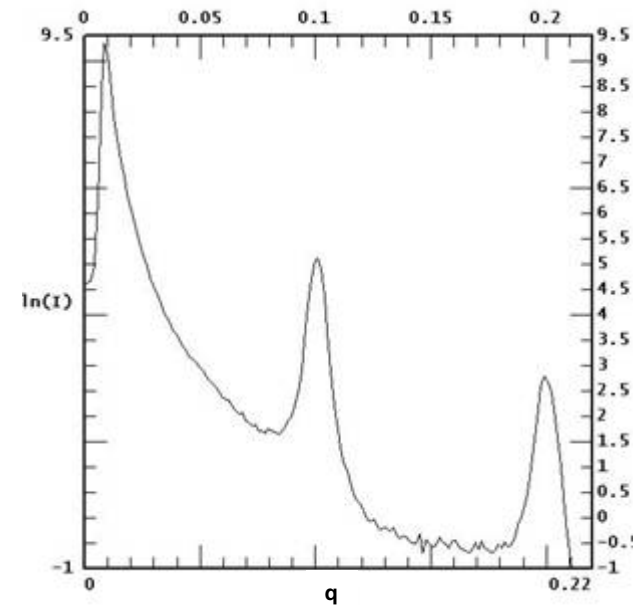
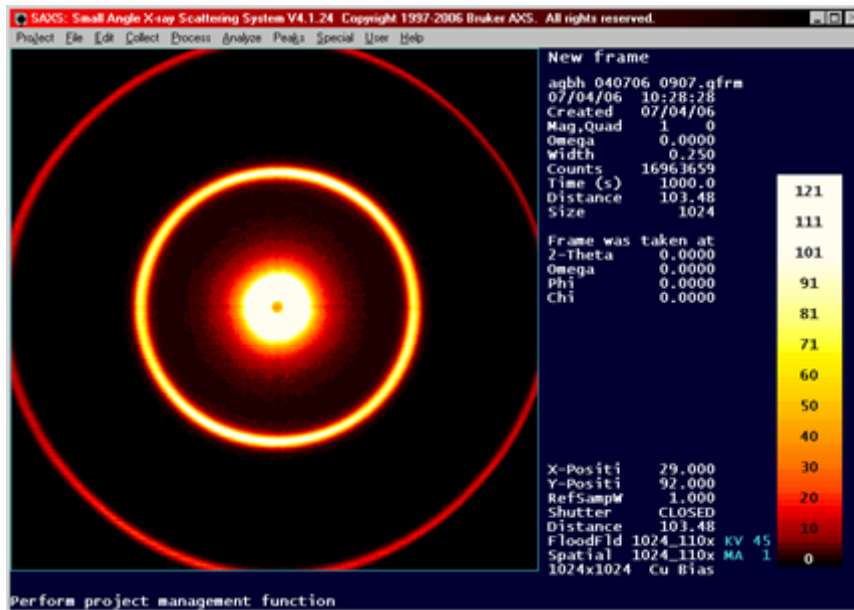


D8 DISCOVER with GADDS HTS ($1\mu\text{S}$): Combinatorial Screening Reflection & Transmission



μ S (Cu-K α) with collimating optics for SAXS measurements

Silver behanate ($\text{H}_3\text{C}-(\text{CH}_2)_{20}-\text{COOAg}$) / Bruker AXS NANOSTAR / 3-Pinhole-geometry / 1000 s exposure time / 1035 mm detector distance

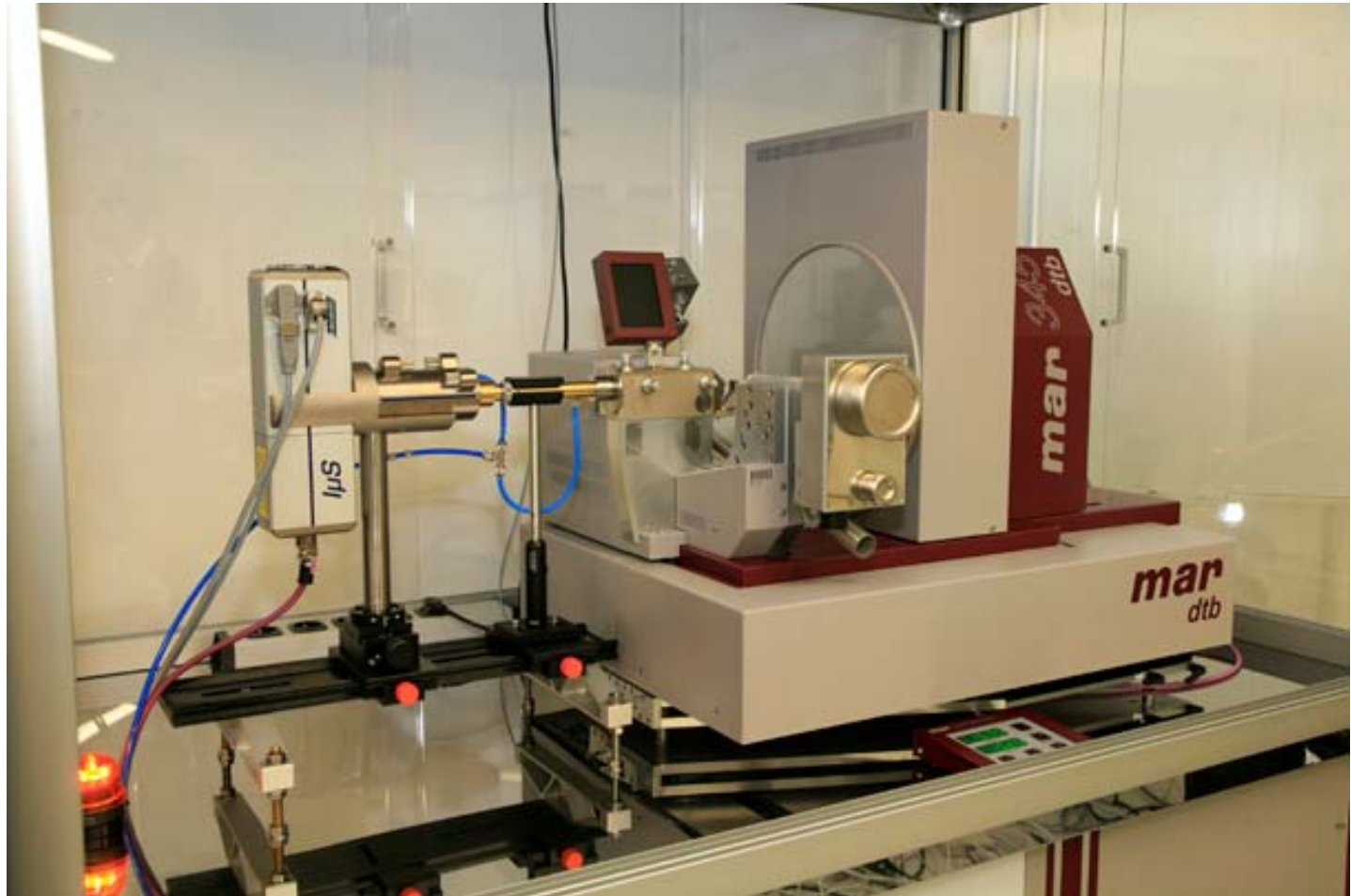


Total Intensity: **17.000 cps** (μ S @ 30W)

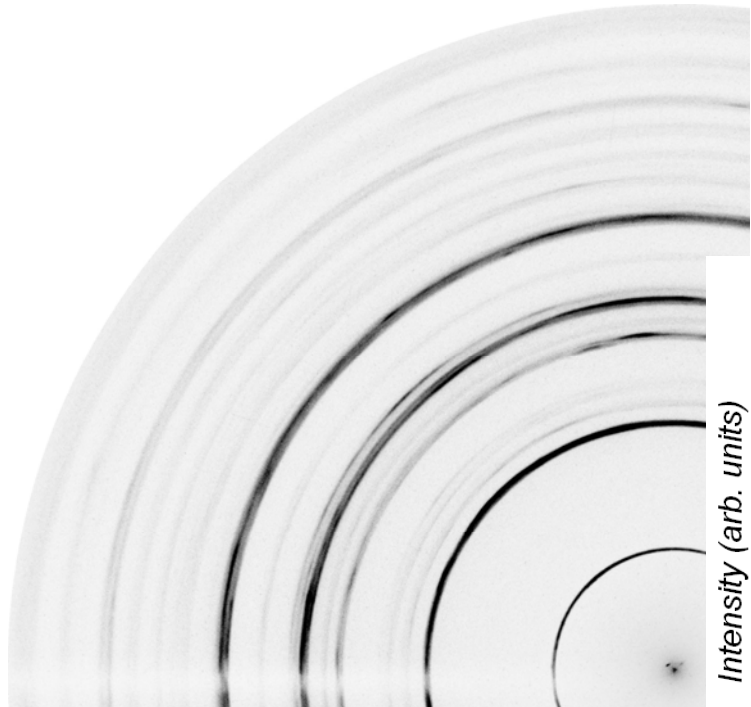
For comparison:

Sealed tube with cc-Göbel mirrors @ 1.4 kW: **3.300 cps**

I μ S with a marDTB

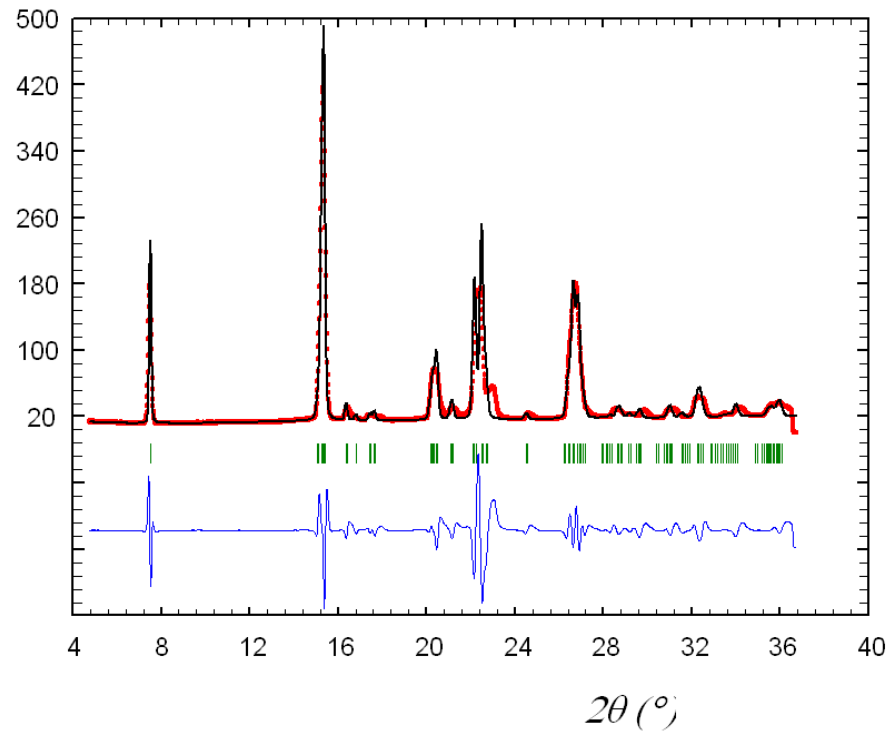


ASS with Cu-K α

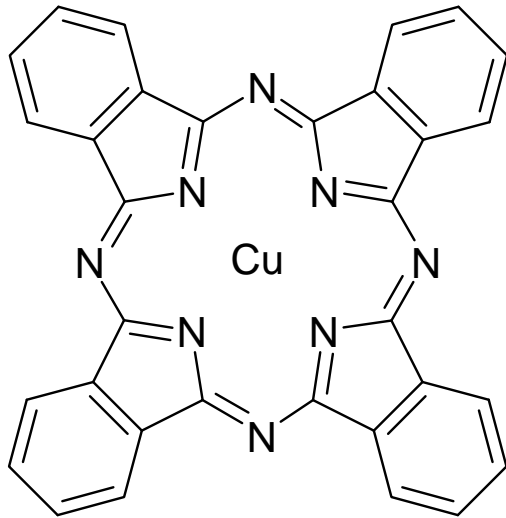


Whole tablet
 Exposure time: 300 s
 Focusing onto the detector

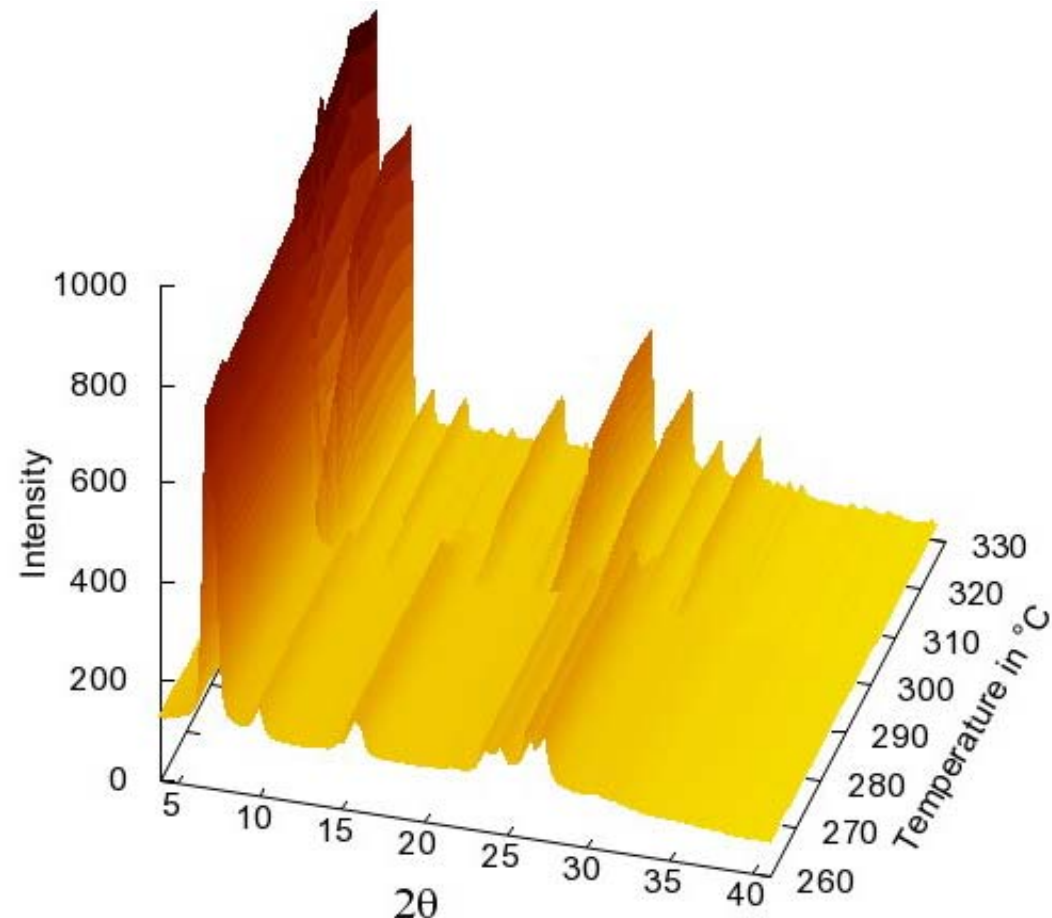
Intensity (arb. units)



Temperature-dependent Phase Transition in Copper phthalocyanine



- Heating from 30 °C to 350 °C with a rate of 0.75 K/min
- Exposure time per frame: 240 s
- Operation mode of mar345: 100 μm x 100 μm @ 240 mm



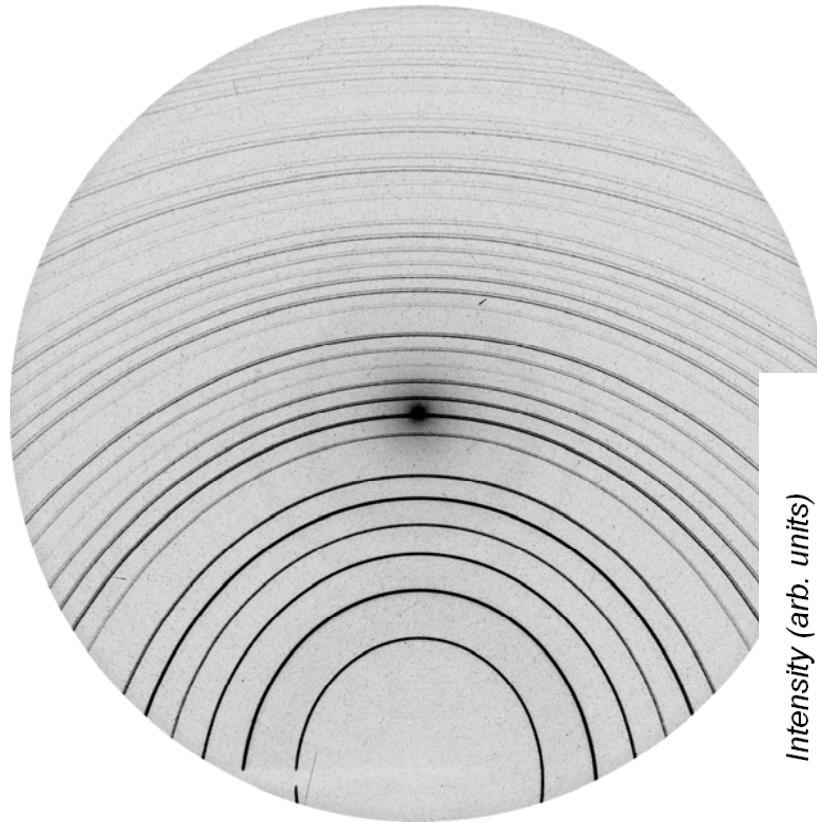
Results

- Reaction Temperature:
 - $T_R = 295 \text{ }^\circ\text{C}$ (Lab data)
 - $T_R = 275 \text{ }^\circ\text{C}$ (Synchrotron data)
- Activation Energy:
 - $E_A = 254 \text{ kJ}\cdot\text{mol}^{-1}$ (Lab data)
 - $E_A = 245 \text{ kJ}\cdot\text{mol}^{-1}$ (Synchrotron data)

Calculated with the non-isothermal Avrami-theorie

 - $E_A = 241 \text{ kJ}\cdot\text{mol}^{-1}$ (isothermal investigation)

$I\mu S$ (Mo- $K\alpha$) with focusing optics: LaB_6



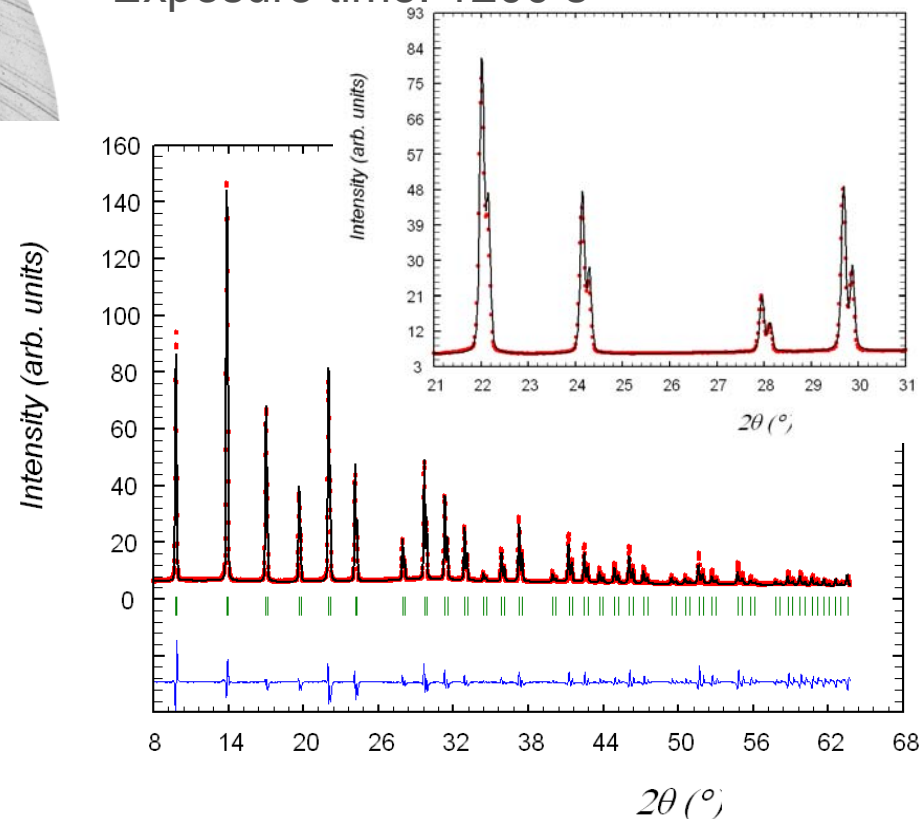
Sample-Detector Distance: 260 mm

2θ : 30°

Slitsize: $0.2 \times 0.2 \text{ mm}^2$

Spot size: $110 \mu\text{m}$

Exposure time: 1200 s



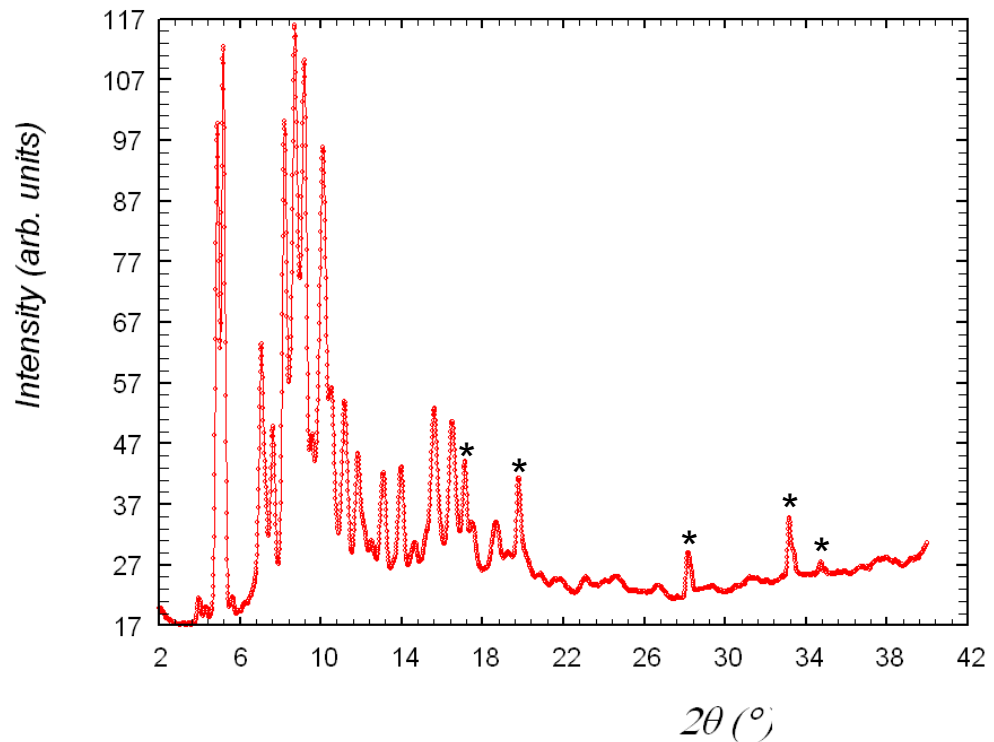
Frame integrated with Powder3DIP
Profile Matching with FullProf

$\langle \text{FWHM} \rangle = 0.13^\circ 2\theta$

Ibuprofen with Mo-K α



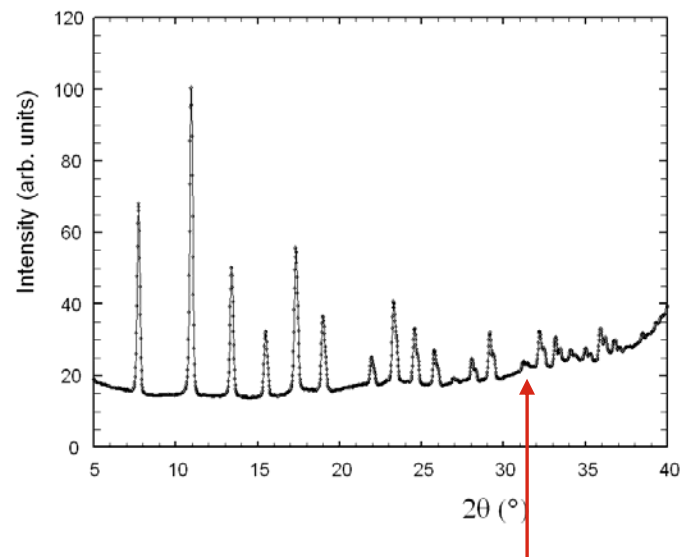
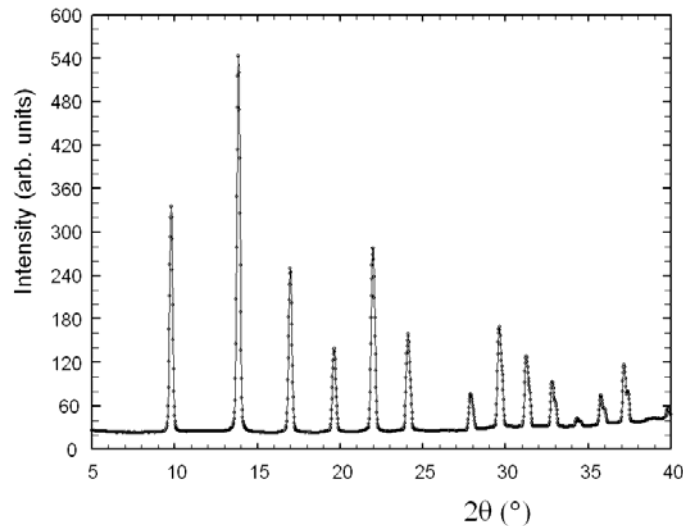
Whole tablet in blister pack
 Exposure time: 120 s
 Focusing onto the detector



Comparison of $I\mu S$ (Mo- $K\alpha$) and $I\mu S$ (Ag- $K\alpha$)

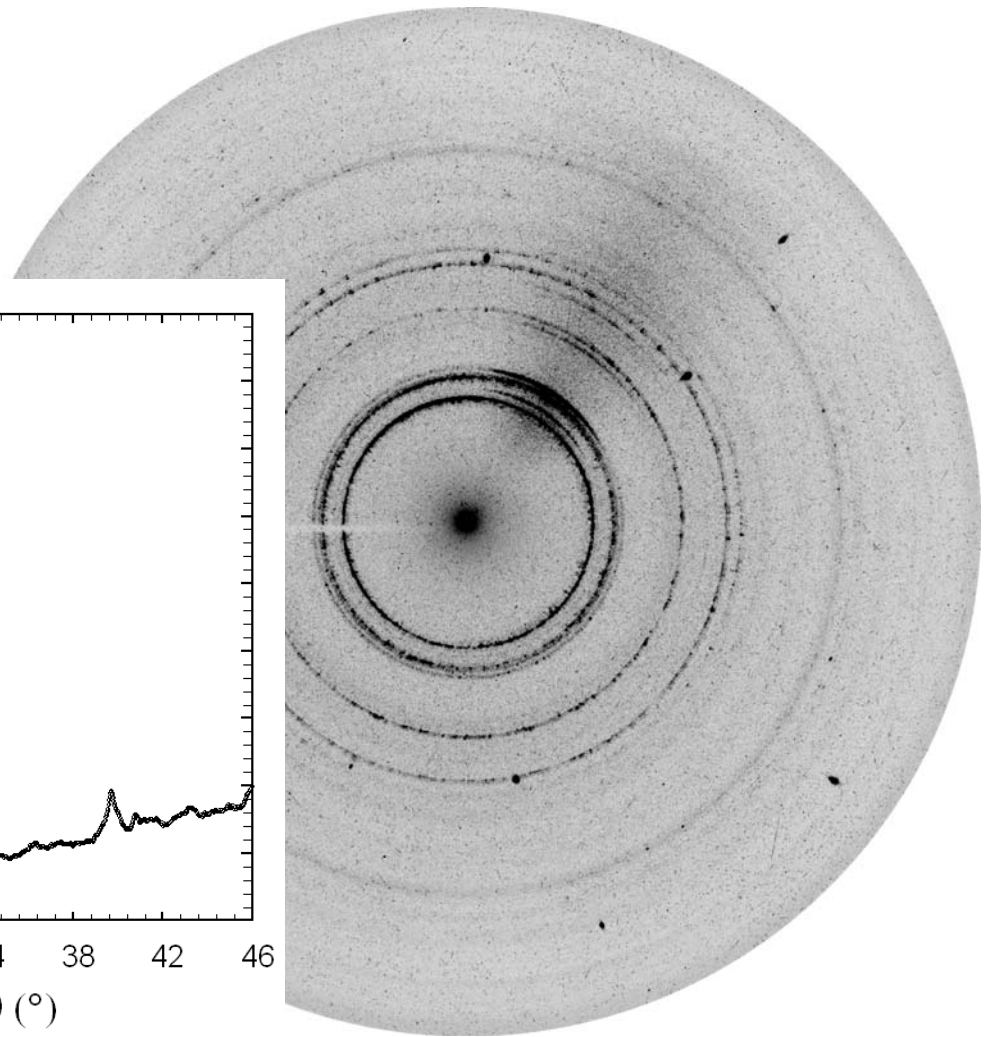
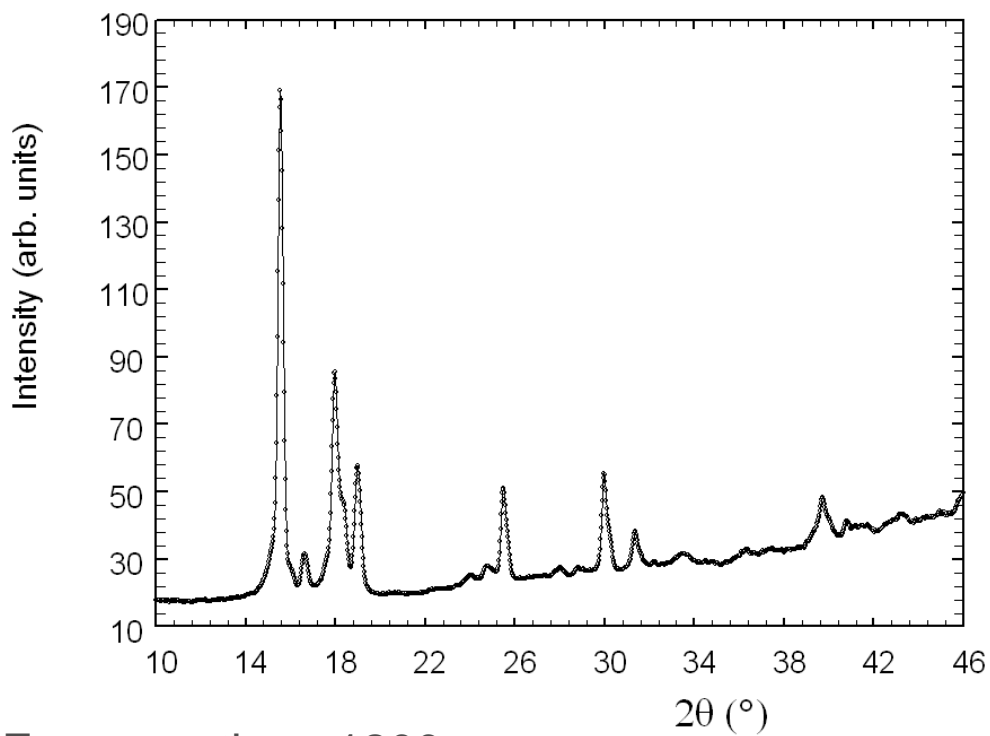


Sample: LaB_6
 Exposure Time:
 300 s
 Detector-Distance:
 200 mm



End of measurement range for Mo- $K\alpha$

μ S (Ag-K α) with focusing optics: Pigment Red 170 in diamond anvil cell



Exposure time: 1200 s

Summary (1): XRD with μ S

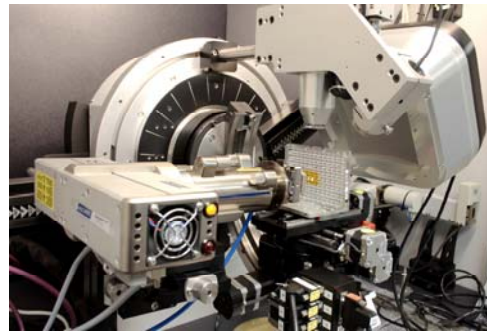
Incoatec Microfocus Source μ S

- Microfocus Source
- 30 W, air-cooled
- New 2D - Quazar Multilayer Optics
- Low maintenance
- for Cu, Mo, Cr, and Ag

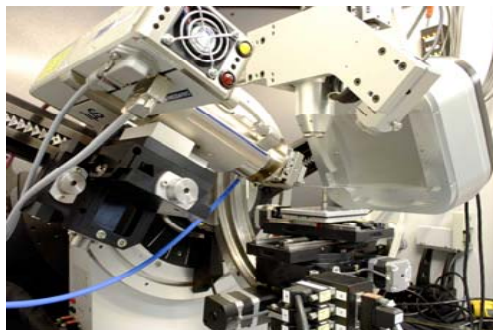


Summary (2): XRD with μS

- 1D collimating beam + 1D focusing beam
- XRD in reflection
- Texture
- Phase identification



- 2D collimating beam
- SAXS
- Texture



- 2D focusing beam
- XRD in transmission
- Well plate screening
- Position sensitive measurements
- Single crystal diffraction



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All colleagues from Bruker AXS

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