NEW POSSIBILITIES FOR
X-RAY DIFFRACTOMETRY

Bernd Hasse – Incoatec, Geesth
Incoatec Microfocus Source – IµS

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 – IµS

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 $I\mu S$
- Optics with Housing
- 2D detector (VÄNTEC 2000)
- Sample stage
- Bruker D8 GADDS with $I\mu S^TM$
IμSTM & VÅNTEC-2000

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

Sample: quartz powder
D8 Discover with GADDS

The five fingers of quartz
μSTM for XRD with focusing optics

- measurements in transmission geometry
- with Bruker D8 GADDS and VÄNTEC 2000
IµS (Cu-Kα) 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

IµS – 2D focussing optics
- Spotszie 230 µm
- 0.3 mm snout
- small slice for integration to obtain better resolution (poor detector calibration)

15 sec collection time
IµSTM focusing onto the detector: LaB$_6$

60 seconds,
292 mm sample-detector

FWHM of LaB$_6$ sample fitted with PV function

FWHM: 0.08°2θ
D8 DISCOVER with GADDS HTS ($I\mu S$): Combinatorial Screening Reflection & Transmission
IµS (Cu-Kα) with collimating optics for SAXS measurements

Silver behenate (H₃C-(CH₂)₂₀-COOAg) / Bruker AXS NANOSTAR / 3-Pinhole-geometry / 1000 s exposure time / 1035 mm detector distance

Total Intensity: 17,000 cps (IµS @ 30W)

For comparison:
Sealed tube with cc-Göbel mirrors @ 1.4 kW: 3,300 cps
$\mu$S with a marDTB
ASS with Cu-Kα

Whole tablet
Exposure time: 300 s
Focusing onto the detector
Temperature-dependent Phase Transition in Copper phthalocyananine

- 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:
  - $T_R = 295 \, ^\circ C$ (Lab data)
  - $T_R = 275 \, ^\circ C$ (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)
  - $E_A = 241 \, \text{kJ} \cdot \text{mol}^{-1}$ (isothermal investigation)

Calculated with the non-isothermal Avrami-theorie
μS (Mo-Kα) with focusing optics: LaB₆

Sample-Detector Distance: 260 mm
2θ: 30°
Slitsize: 0.2 x 0.2 mm²
Spot size: 110 μm
Exposure time: 1200 s

Frame integrated with Powder3DIP
Profile Matching with FullProf

<FWHM> = 0.13°2θ
Ibuprofen with Mo-K$_\alpha$
Comparison of IµS (Mo-K$\alpha$) and IµS (Ag-K$\alpha$)

Sample: LaB$_6$
Exposure Time: 300 s
Detector-Distance: 200 mm

End of measurement range for Mo-K$\alpha$
IμS (Ag-Kα) with focusing optics: Pigment Red 170 in diamond anvil cell

Exposure time: 1200 s

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B. Hasse – PPXRD 2009, Glasgow
Summary (1): XRD with İμS

Incoatec Microfocus Source İμS

- Microfocus Source
- 30 W, air-cooled
- New 2D - Quazar Multilayer Optics
- Low maintenance
- for Cu, Mo, Cr, and Ag
Summary (2): XRD with IµS

- 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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