RÉCIPROCS

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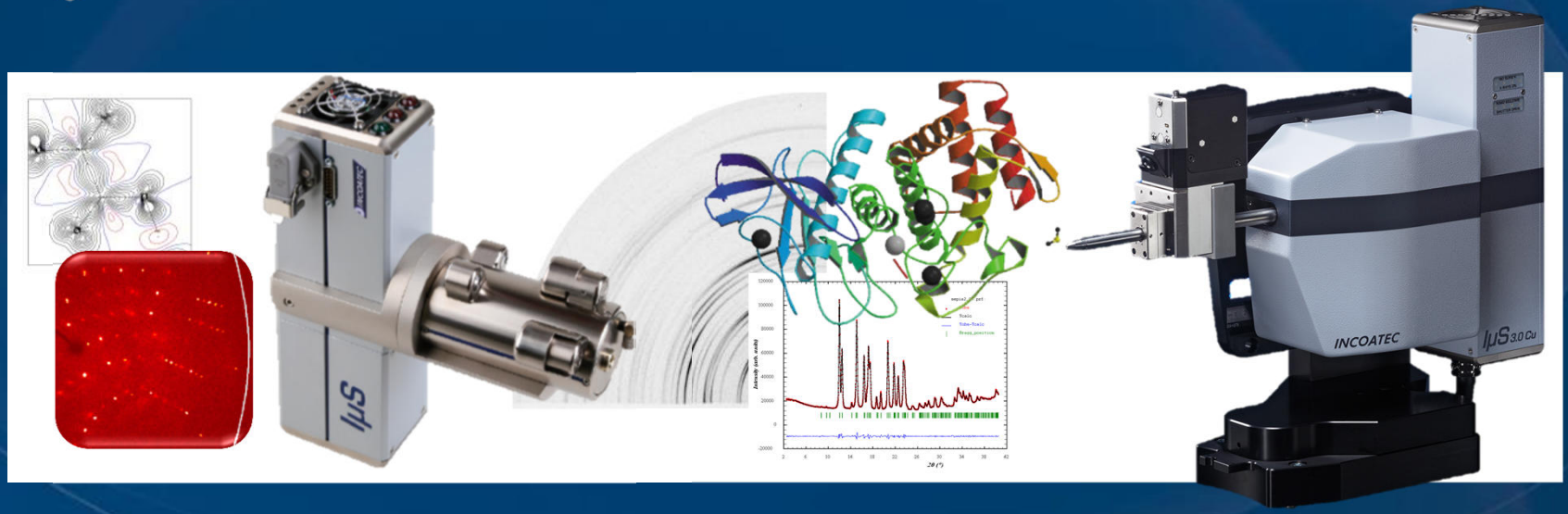
innovative coating technologies

The Incoatec Microfocus Source IμS

10 Years of IμS – A Retrospective



Jürgen Graf – Incoatec GmbH, Geesthacht, Germany

Agenda

- Introduction
- What‘s new with the I*μ*S 3.0
- Microfocus Sources and Multilayer Mirrors – Technical Background
- Applications of the I*μ*S
  - Single Crystal Diffraction
  - Small Angle Scattering and General XRD

Incoatec: Innovative Coating Technologies

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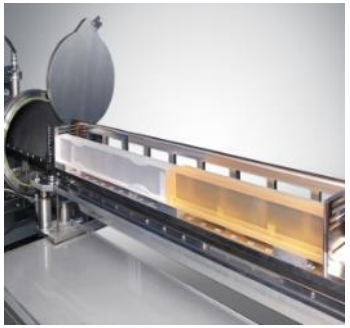
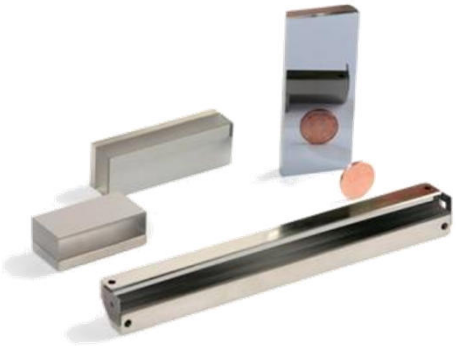


- Founded 2002 as **joint venture with Bruker AXS**, located in Geesthacht (near Hamburg)
- Production & development of **X-ray optics** and **microfocus X-ray sources**
- Equipment for **home-lab** instruments and **synchrotron** beamlines
- > 50 employees, new headquarter 4200 m^2, > 10% invest for R&D



Multilayers @ Incoatec
A Brief History of Time

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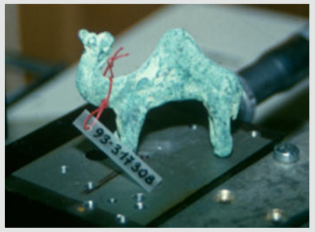
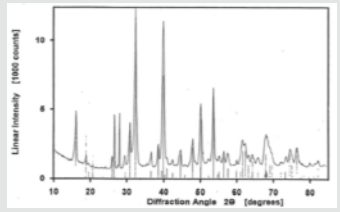
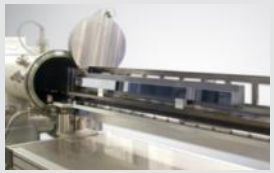
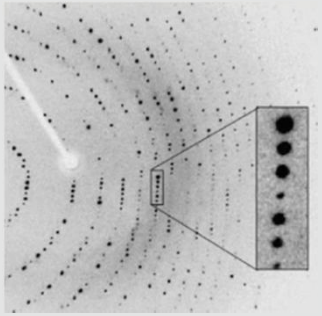
1989	**Research on deposition and properties of thin films at GKSS research center**
1993/94	**Introduction of „Göbel-Mirrors“ by Herbert Göbel**
1995	**Joint development of Göbel mirrors in cooperation with Siemens**
99 - 01	**1st synchrotron mirrors for FEL** **1st Montel Optics for SC-XRD**
2002	**Incoatec is incorporated as spin-off and joint venture with Bruker AXS**
2006	**Launch of the Cu-I*μ*S** (I*μ*S 1.0,
2015	**I*μ*S 3.0** (AsCA Kolkatta)
2016	**> 700 I*μ*S's sold world wide**




The Incoatec Microfocus Source IμS

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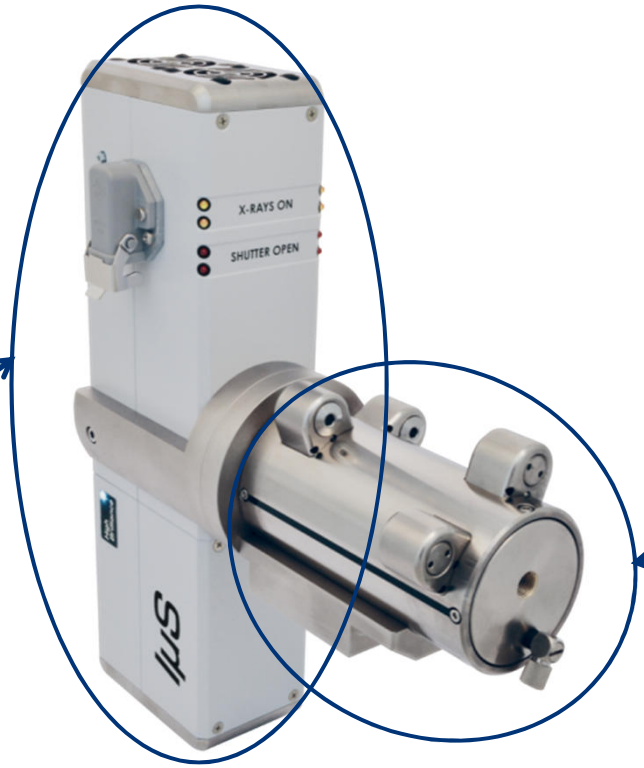

- Spot size < 40µm
- Air-cooled
- No moving parts
- 3 years warranty
- > 700 sold world-wide

X-ray tube:
defines
- wavelength
- **brilliance**

ML Optics:
defines
- divergence
- image spot size
- image distance

Intensity Comparable to Traditional Rotating Anode Generators

10 Years Incoatec Microfocus Source IμS

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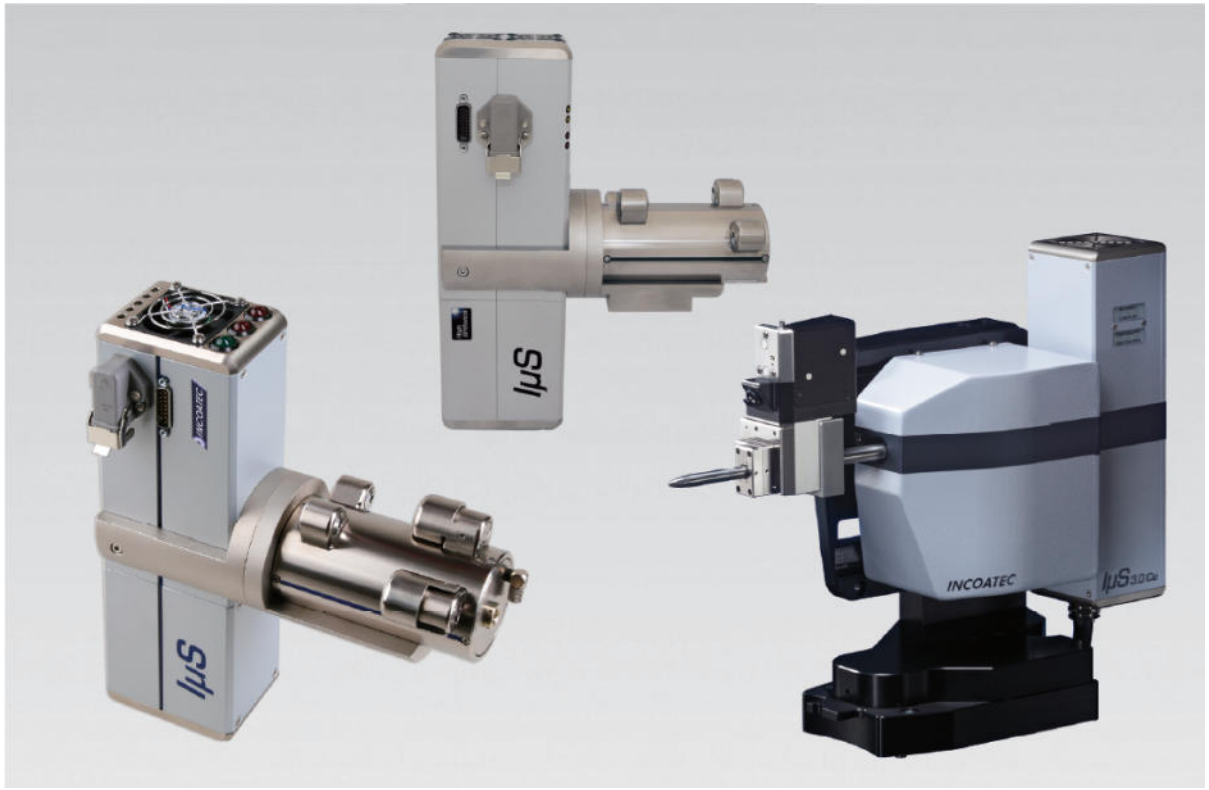

What's NEW?

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The IμS 3.0 – The Next Generation IμS

- **First** and **only** microfocus X-ray source designed for SC-XRD applications
- **New Tube**: Optimized tube parameters and improved heat management
- **New Beampath Concept**: Tube and optics are **pre-aligned** and **separately mounted** to an adapter plate for **true downstream alignment**
- **New** "**Quick-Lock**" mount for highly precise and reproducible mounting of source and optics
- **New Optics**: **MX-3 mirror**
- **More intensity without compromises in lifetime – always air-cooled !**

30 – 40 % Intensity gain over IμS HB

The IμS 3.0 – The Next Generation IμS
Optimizing the IμS 3.0 X-ray Tube

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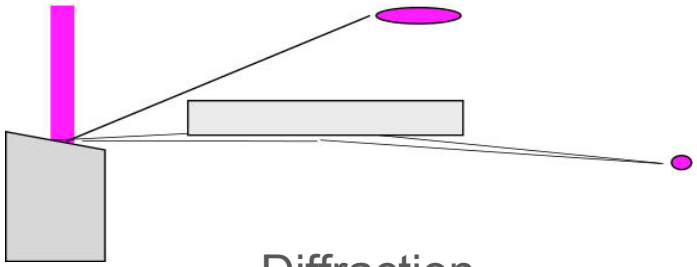
- **Conventional microfocus sealed tubes**
  - Designed primarily for radiography (medical or NDT)
  - **High take-off angles** and **smaller electron beam focus** in order to preserve resolution over a wide field of view

Radiography

- **The IμS 3.0 tube:**
  - Designed for X-ray diffraction
  - No need for wide FOV and coherence
  - **Reduced take-off angle** and **line-focused** e beam focus to match the view angle of the X-ray mirror
  - **New filament** and **e beam optics**: more homogeneous and dense illumination



Diffraction

The IμS 3.0 – The Next Generation IμS

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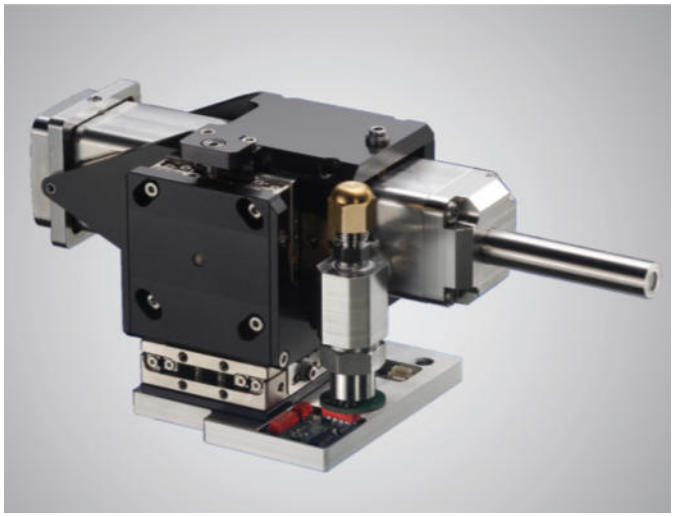
The Optics:

- New optics housing with **pre-set Bragg angle**
- **Improved X-ray optics** for Mo and Ag
- **New X-ray mirror** for Cu (**MX-3 optics**)
- **Encapsulated optics** housing filled with He
- He pressure (~ 1.4 bar) is monitored by a **pressure sensor**
- Less absorption due to **He flight tube**
- New „**Quick-Lock**“ mounting



The IμS 3.0 – The Next Generation IμS

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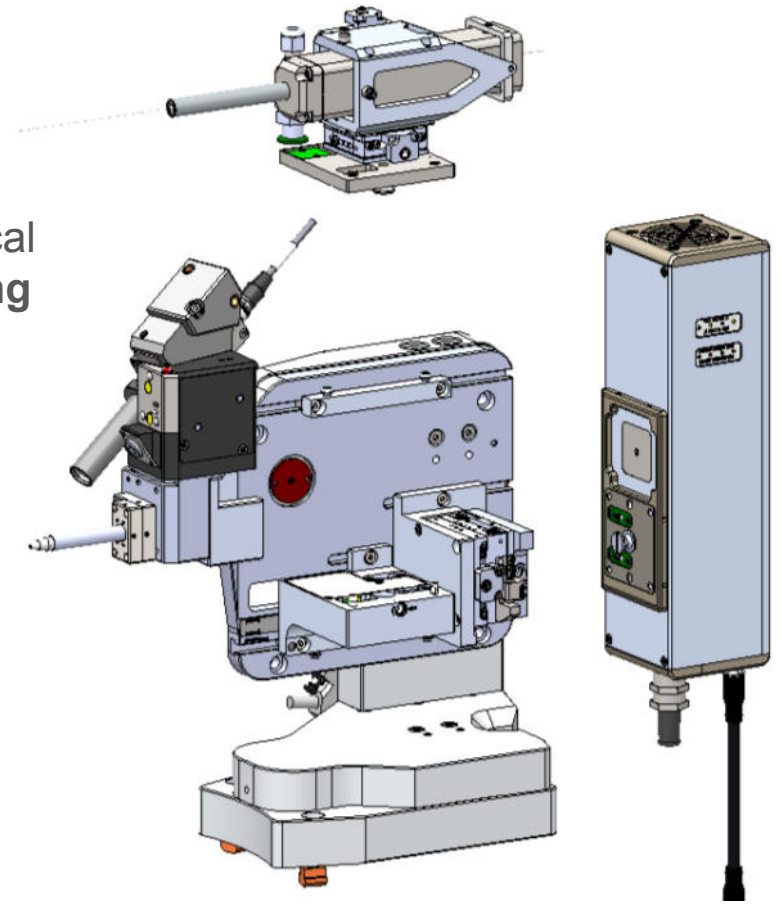
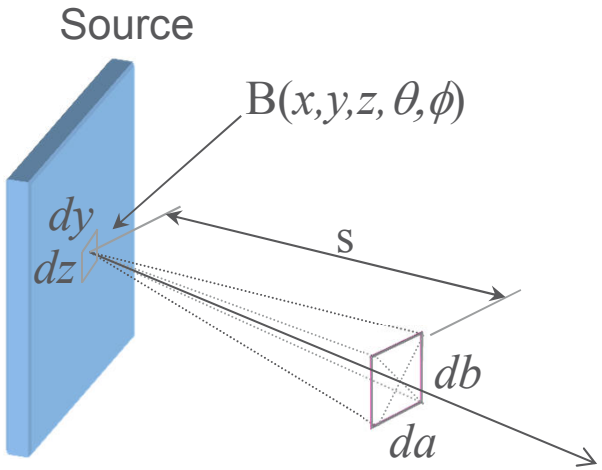
- **The Quick-Lock Concept:**
  - **Pre-aligned source** and **optics**:
    - both separately mounted on an optical bench **with just single screws using a well-defined torque** and high-precision mounts with pinning
    - **continuously adjustable** along the beam direction for precise setting of source-to-sample distance
  - **Maximum intensity** on the sample
  - Optics is **swappable** with a precision of below 10 μm at the sample position (!!!)
  - **No instrument realignment** necessary after optics swap or tube change



Brilliance and Spectral Brightness B

- **Brilliance = Spectral Brightness B:**
  - is the number of photons in a given bandwidth emitted per unit time, per unit area and per unit solid angle
  - B: [phts/s/mm^2/mrad^2/0.1% bandwidth]
  - is a property of the source
  - is proportional to the power load at the target and is, therefore, limited by the heat dissipation mechanism
  - is a good measure for comparing sources



High brightness means that the X-rays are highly “concentrated”.

D. M. Mills et al., *J. Synchrotron Rad.* (2005), *12*, 385

Why Microfocus X-ray Sources?

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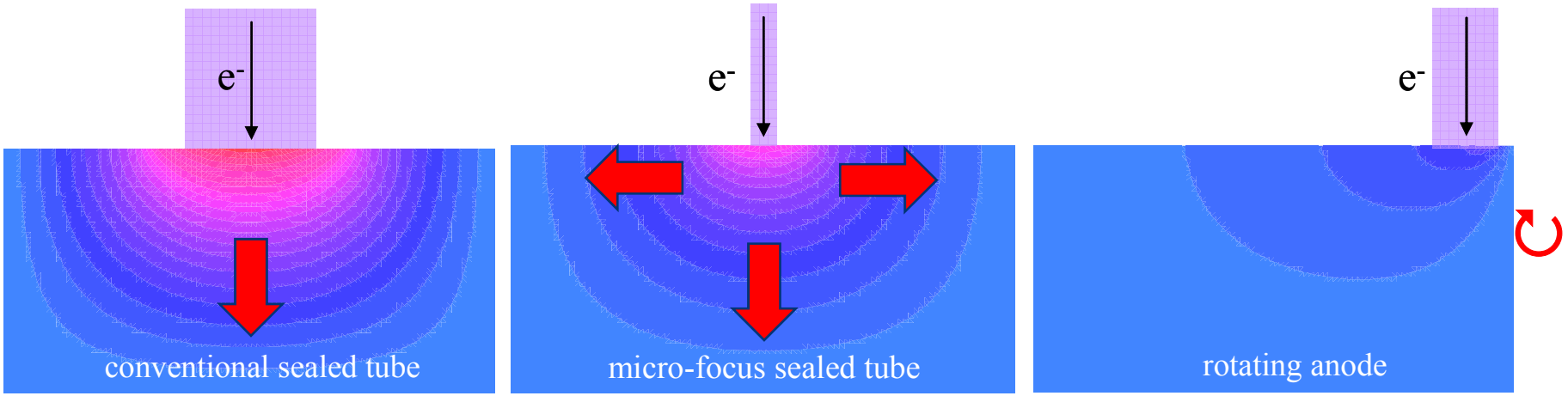
Power Load in All Solid-target X-ray Sources is Limited by Heat Dissipation



- Large Spot
- Quasi-1D heat flow limits power density
- ~ 0.5 kW/mm^2

Relative B: 1

- Small Spot
- 2D heat flow allows more efficient cooling
- ~ 5 kW/mm^2

Relative B: ~ 10

- Large or Small Spot
- Additional heat spread by rotation
- > 15 kW/mm^2

Relative B: > 10

Bruker AXS, Technical Note SCD 4, 2007
D. E. Gryder, A. Wright & P.C. Ausburn, *J. Phys. D: Appl. Phys*. 1986, *19* , 2281

Why Microfocus X-ray Sources?

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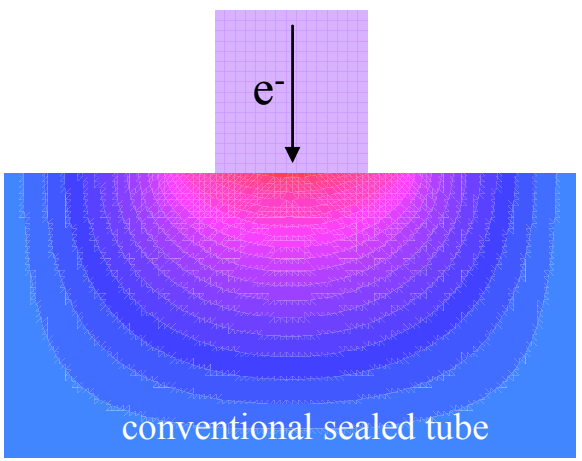
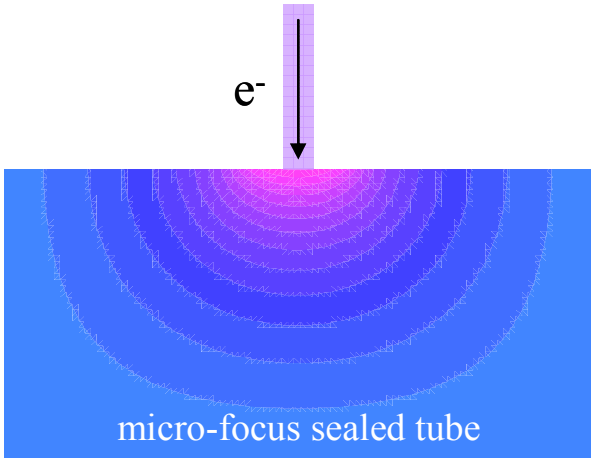
Power Load in All Solid-target X-ray Sources is Limited by Heat Dissipation



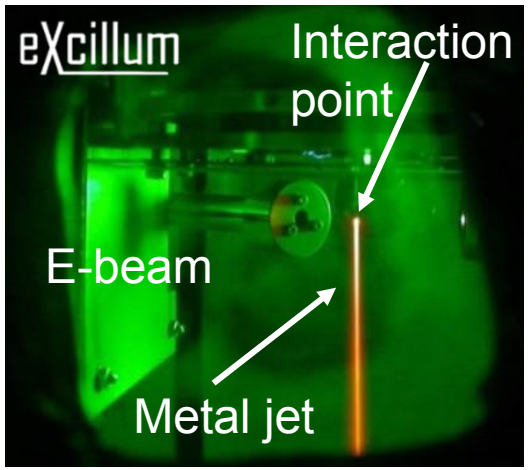
- Large Spot
- Quasi-1D heat flow limits power density
- ~ 0.5 kW/mm^2

Relative B: 1



- Small Spot
- 2D heat flow allows more efficient cooling
- ~ 5 kW/mm^2

Relative B: ~ 10



- Very small Spot
- High speed jet of liquid metal alloy
- > 100 kW/mm^2

Relative B: > 100

M. Otendal *et al.*, *Rev. Sci. Instrum.* (2008), *79*, 016102

Why Microfocus X-ray Sources plus ML Optics?

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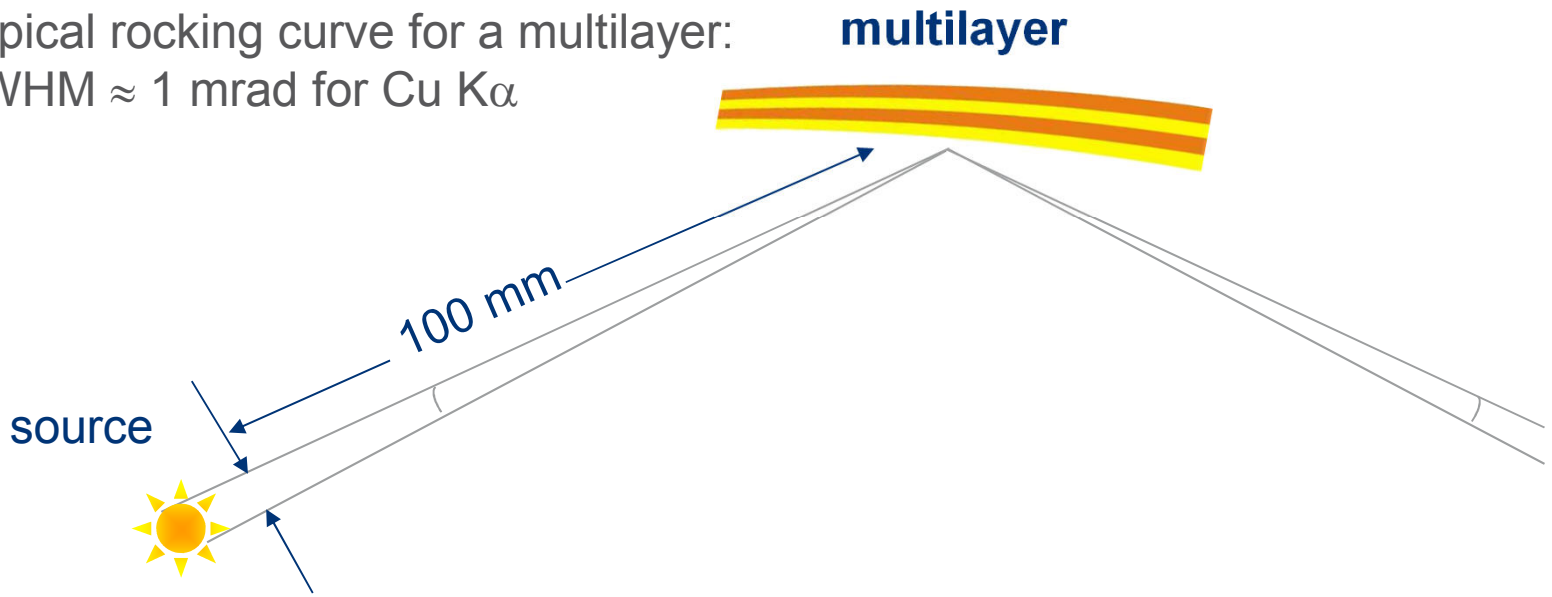
Perfect Match: Multilayer Mirrors and Microfocus Sources

View angle = Bragg peak width

Typical rocking curve for a multilayer:
FWHM ≈ 1 mrad for Cu Kα

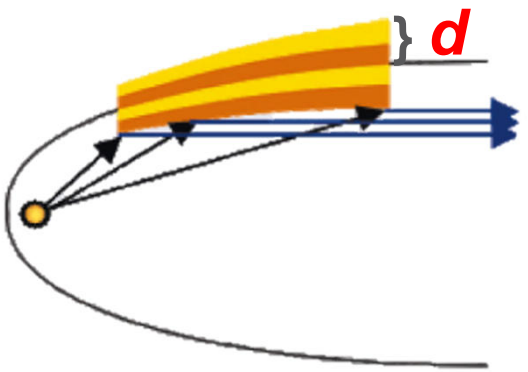


100 mm * 1 mrad = 100 *µ*m

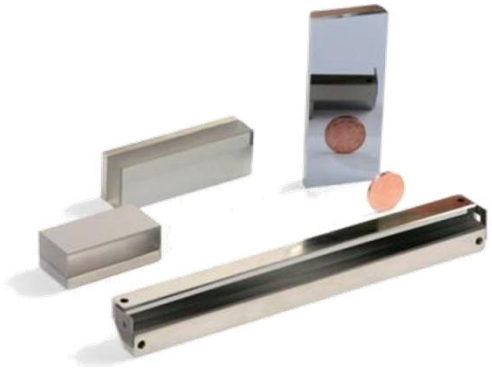
1 mrad = 0.057 deg

What Are Multilayer X-ray Optics?

- **Multilayer Mirrors act as Bragg Reflector:**
  - collect as many photons as possible, select photons of a certain energy and redirect those to a sample by either focusing or collimating the X-ray beam
  - alternating amorphous layers of a spacer (C, B_4C, …) and a reflector material (W, Ni, …)
  - following Bragg´s law, the X-rays get reflected at the interfaces of the multilayer optics



- **Advantages:**
  - High reflectivity and high spectral purity
  - Flexible in design (*d*-spacing, divergence, beam size, …)



M. Schuster, H. Göbel *et al.*, *Proc. SPIE* (1999) *3767*, 183 – 198

Multilayer X-ray Optics

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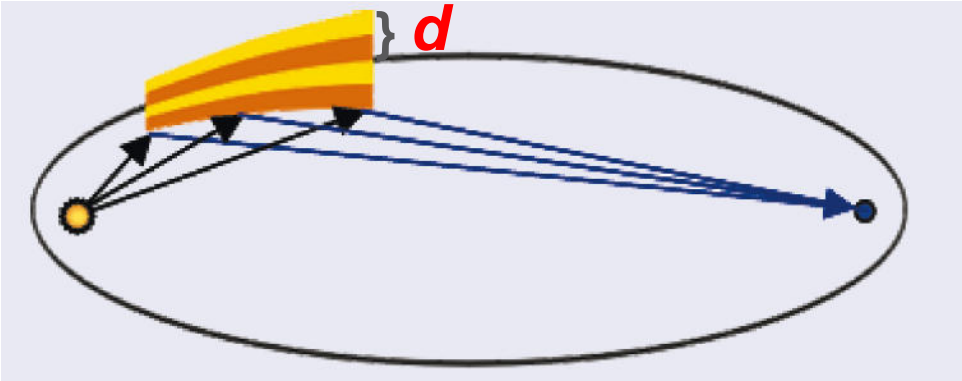
d Spacing Gradient

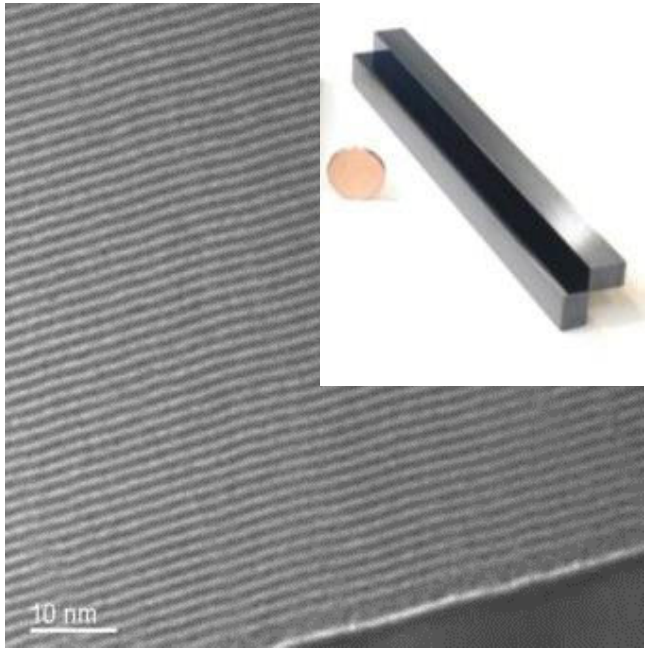


$$m \cdot \lambda = 2 \cdot d \cdot \sin\Theta$$

d = 2 – 6 nm

$\theta_m \approx 1.0°$ (Cu-K_α)

$\theta_m \approx 0.5°$ (Mo-K_α)



W/C Multilayer (TEM, Uni Kiel, Prof. Jäger)

~ 100 layer pairs

Tolerance in *d* spacing +/- 1%

M. Schuster, H. Göbel *et al.*, *Proc. SPIE* (1999) *3767*, 183 – 198

Manufacturing of a Multilayer X-ray Mirror

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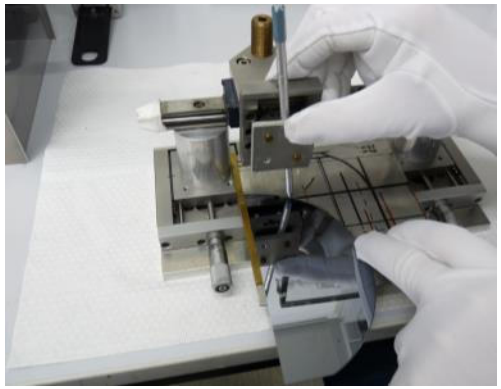
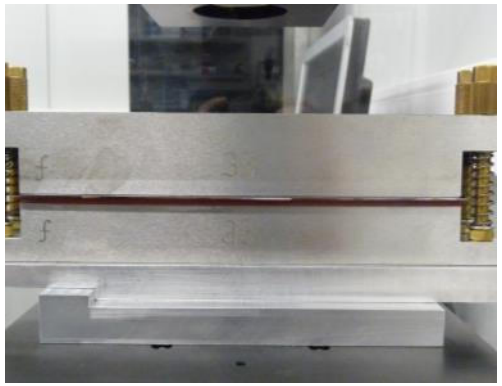


Cutting of Si wafer



Bending of a substrate

2 „Fries“ (almost) ready for coating

Coating

Quality Control and Assembly

Magnetron Sputtering for Layer Deposition

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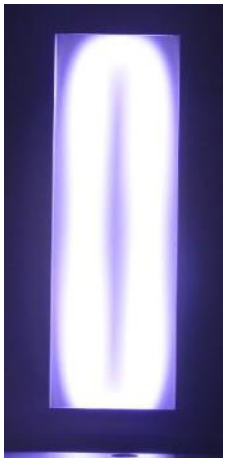
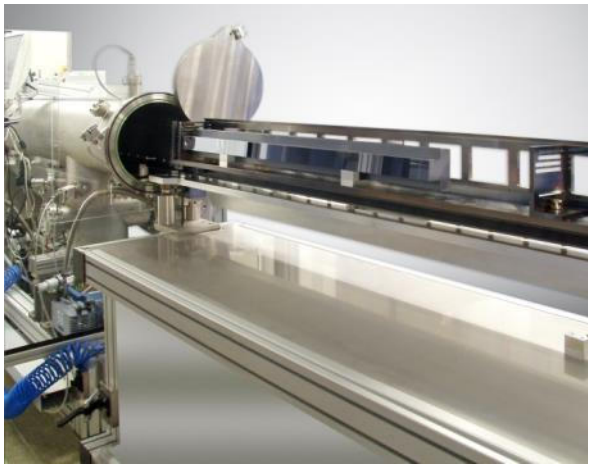


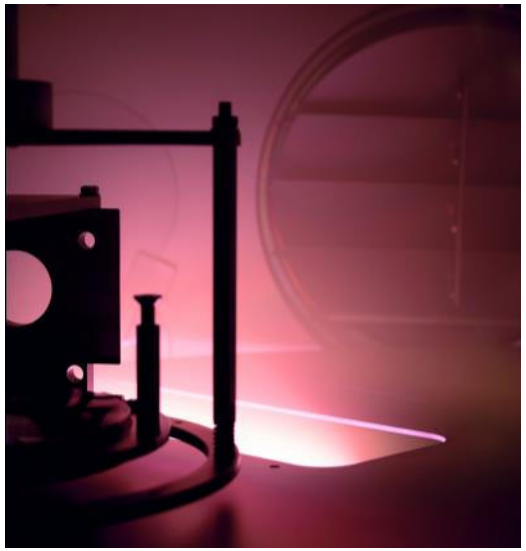


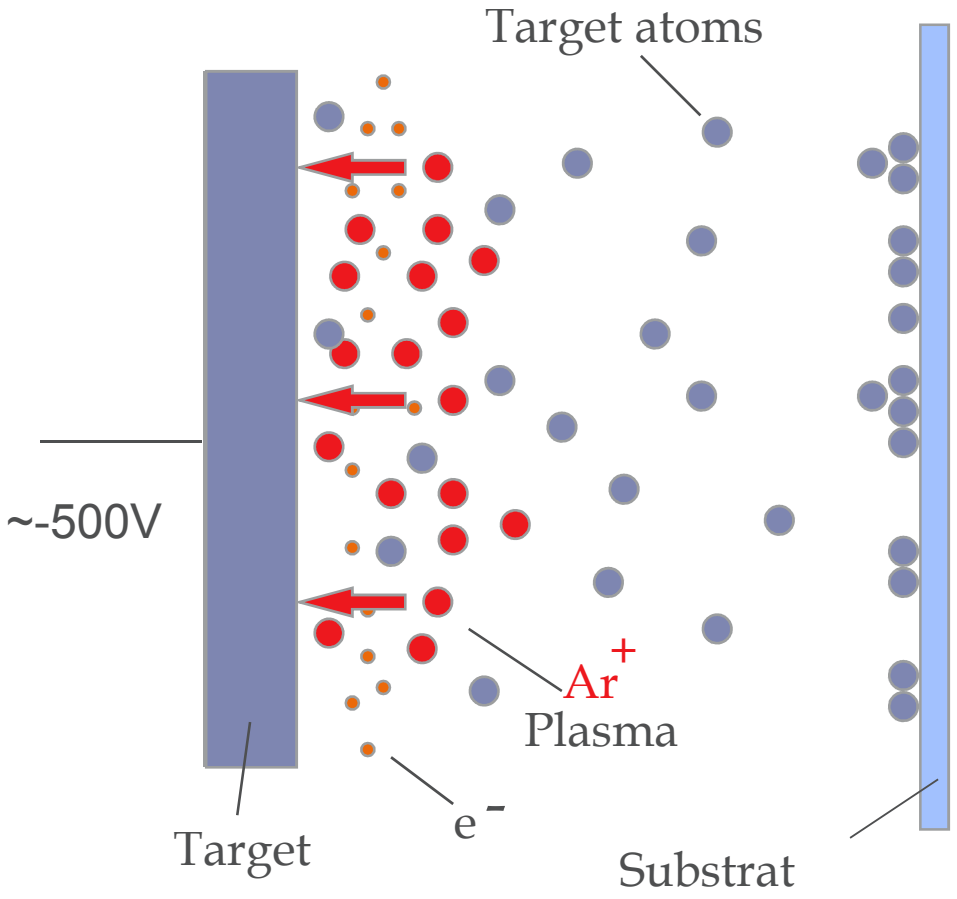
Argon Plasma

Optimized Deposition Facilities for Different Sizes, Gradients and Precisions

Multilayer Deposition by Magnetron Sputtering

Vacuum < 10^{-7} mbar
Inert gas (Ar) 10^{-3} mbar





Requirements for high-quality multilayers:

- well-defined interfaces
- reproducibility of the pairs

X-ray Reflectometry for Characterization of Graded Multilayers

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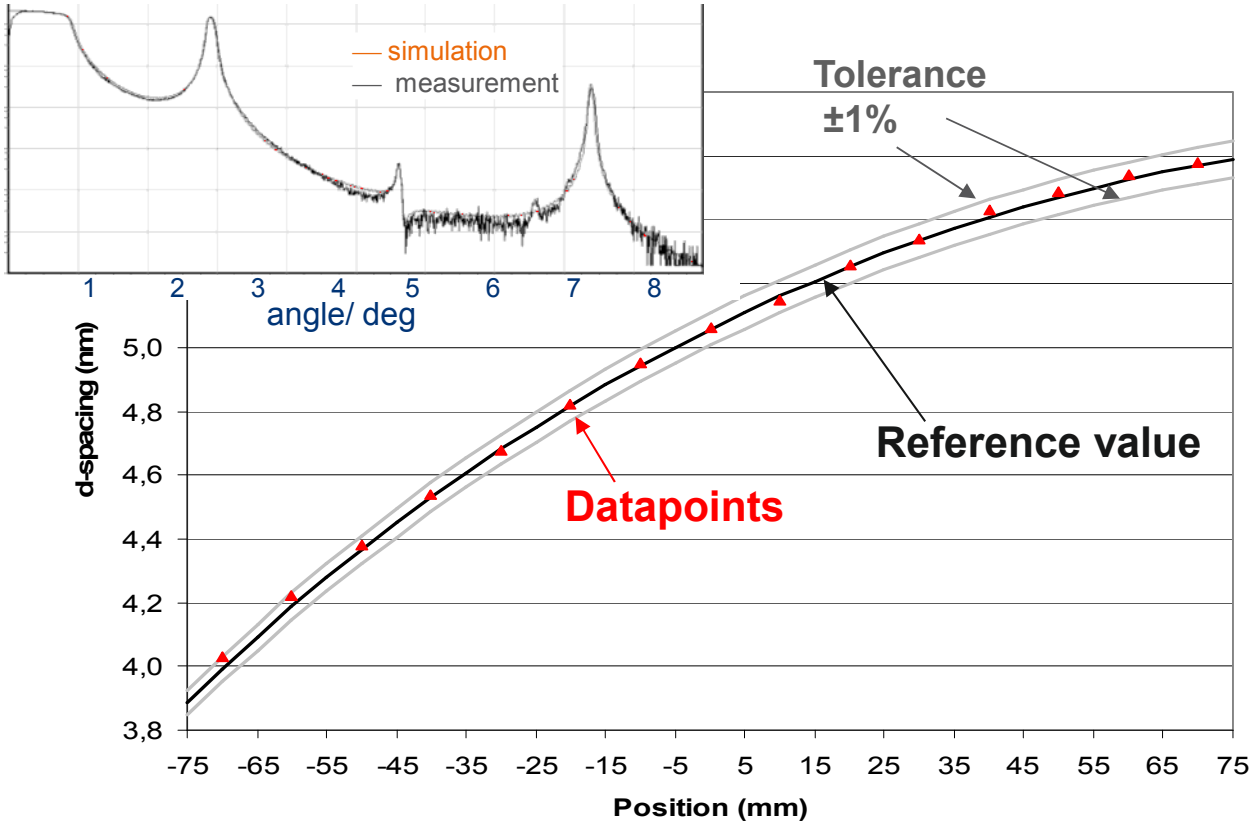
Multilayer Optics for Crystallography in the Home-lab



TEM-Picture of a Multilayer Coating

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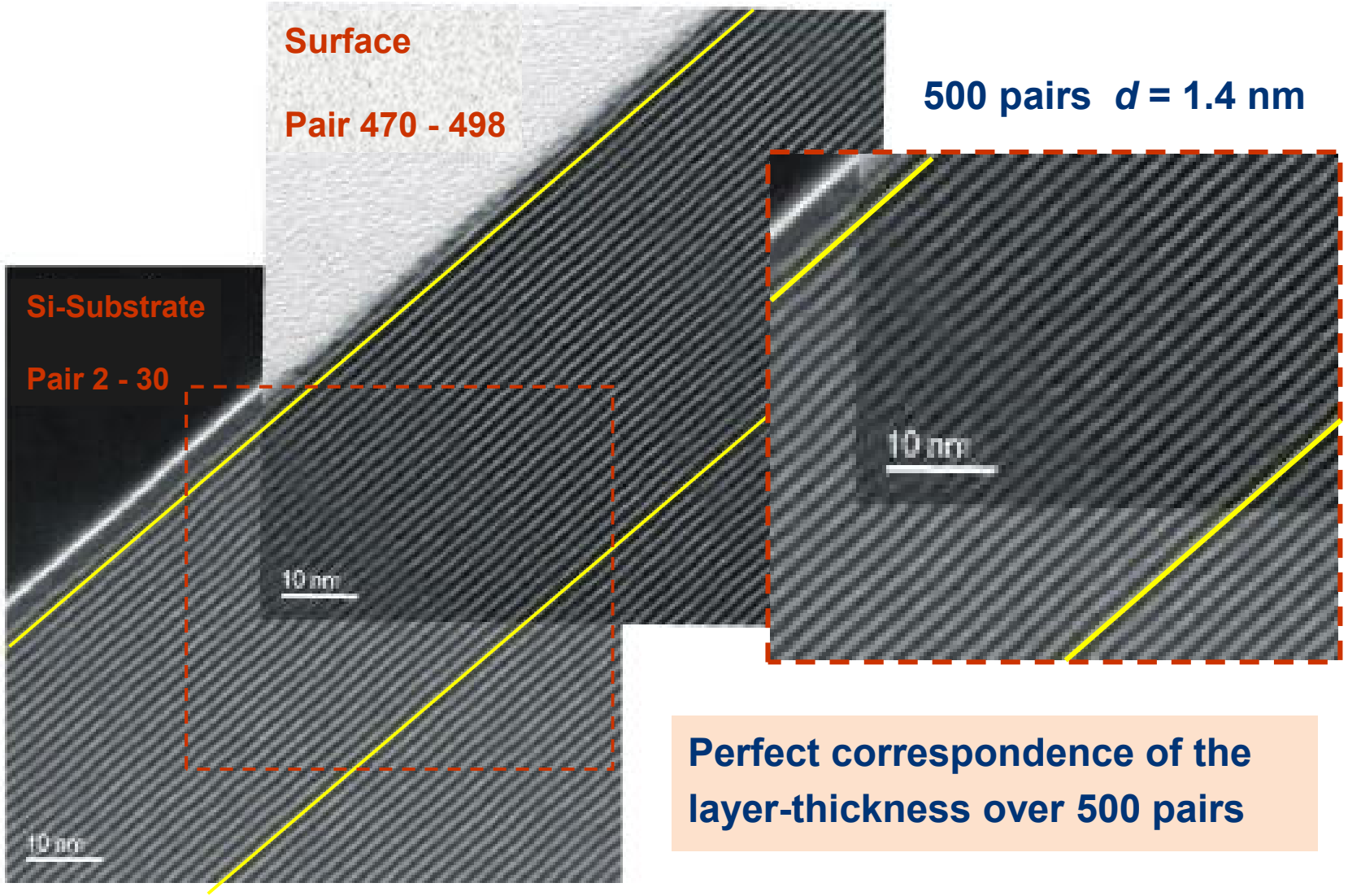


Perfect correspondence of the layer-thickness over 500 pairs

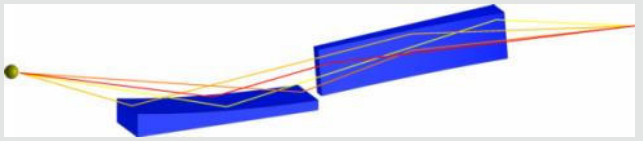
Montel Multilayer Mirrors

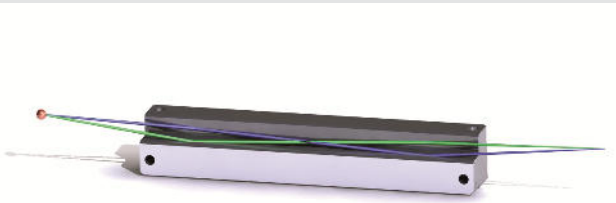
Beam Profile of Montel Multilayer Mirrors

- **Modified Kirkpatrick-Baez Arrangement**
  - Two (identical) 1D bent mirrors in a side-by-side configuration

- **Benefits of the Montel Scheme**
  - More compact
  - Easy alignment
  - Symmetrical divergence spectrum

www.x-ray-optics.de





Beam Path of Kirkpatrick-Baez (Cross-coupled) Mirrors and Montel Mirrors; Typical Beam Pattern from Montel Mirrors.

New Bruker AXS Instruments with one or two IμS's

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D8 QUEST / VENTURE

D8 ADVANCE / DISCOVER

D8 FABLINE

MICRO Series

BRUKER

NANOSTAR

IμS Upgrades on Bruker Instruments

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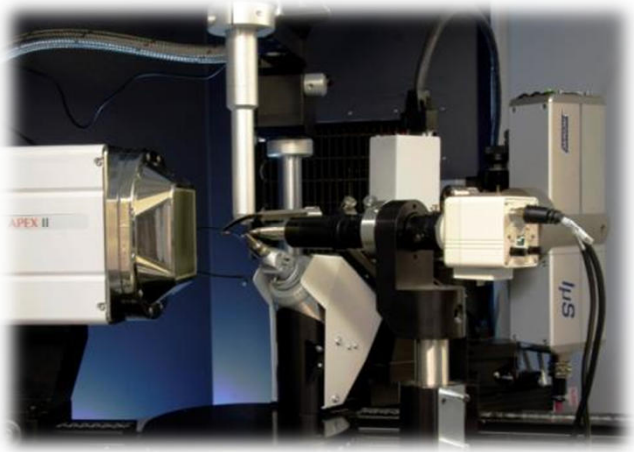
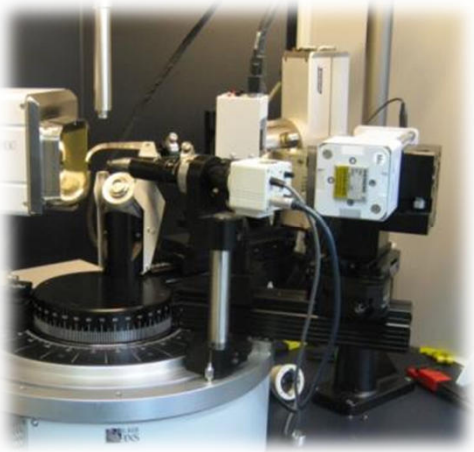
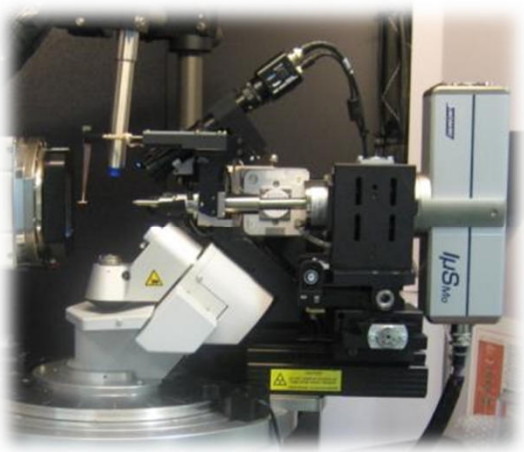


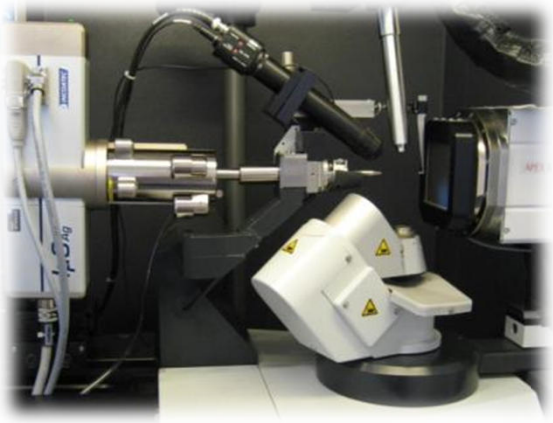
SMART APEX II



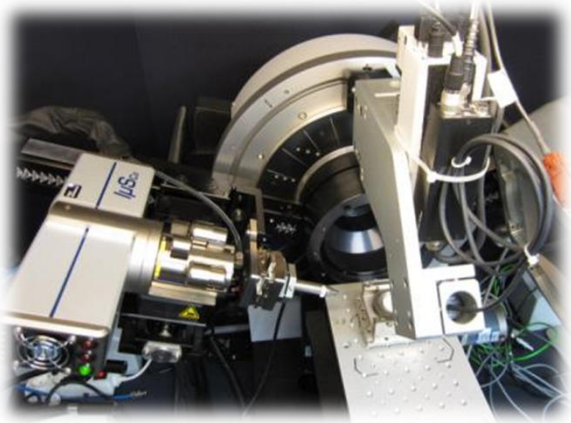
SMART APEX as DUO



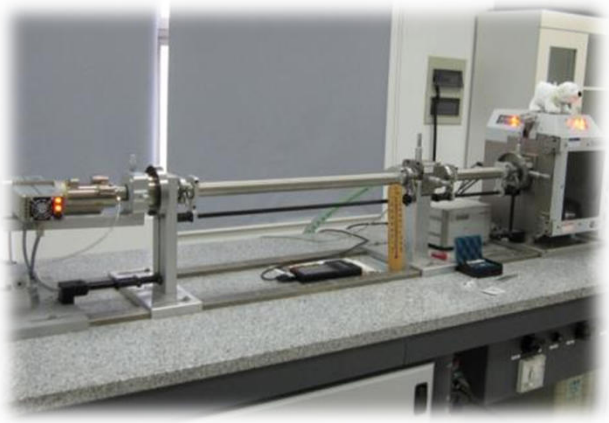
KAPPA APEX II as DUO



X8 APEX / NONIUS KAPPA CCD



GADDS w/ VANTEC (XRD^2)



NANOSTAR

IμS Upgrades on Other Vendor's Instruments

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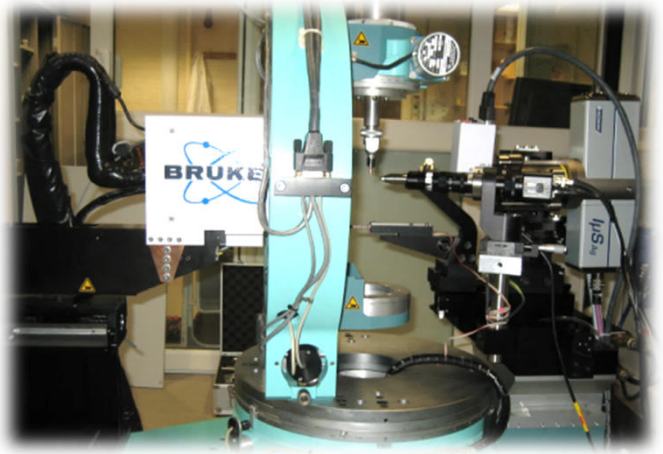
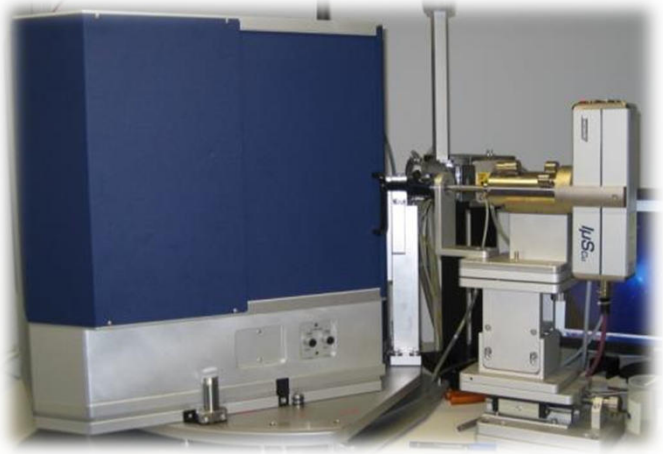
marresearch mar345

Rigaku R-AXIS IV^{++}



Huber Eulerian Cradle



STOE IPDS

IμS Upgrades on Customized Instruments

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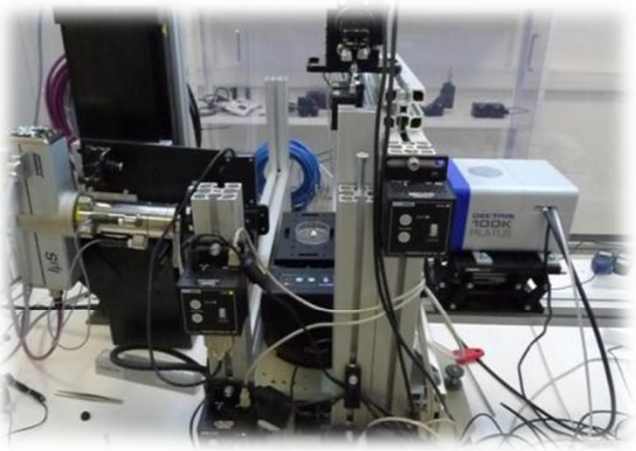
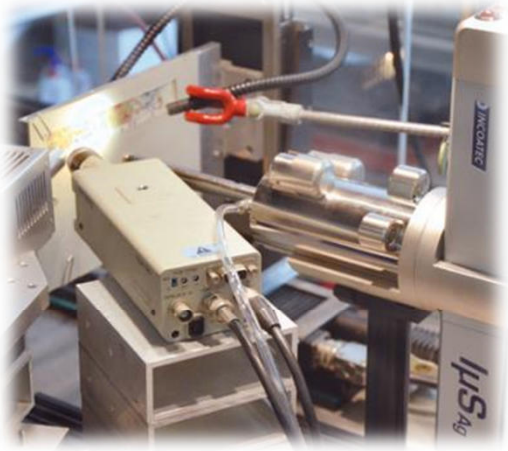
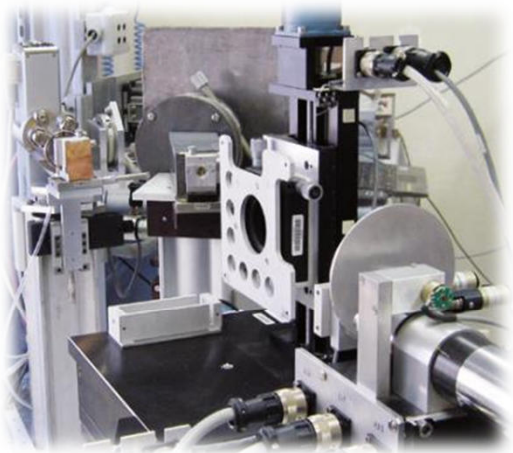


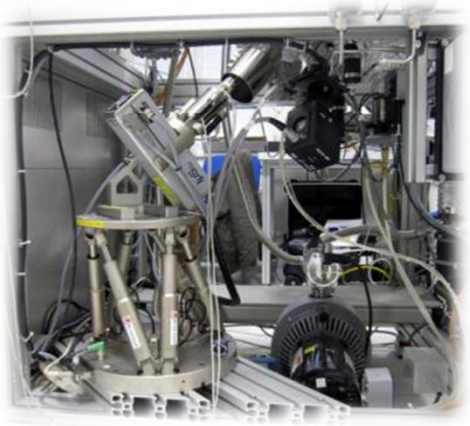
(GI)SAXS w/ PILATUS



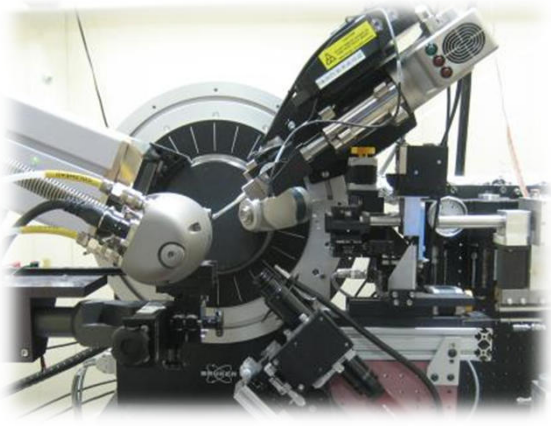
XRD / XRF w/ PILATUS



XRD w/ Scintillation Counter



In-situ (GI)SAXS w/ PILATUS



SC-XRD Duo IμS / Synchrotron

SAXS w/ marCCD

What else is NEW?

INCOATEC
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- **The Incoatec X-ray Enclosure IXE**

  - Spacious cabinet for all common goniometers, detectors and dual wavelengths set-ups
  - Guaranteed radiation safety for all IμS sources
  - Compliant with the latest functional safety standards
  - Tapped hole pattern plate for flexibility of experimental set-ups
  - Removable panels for easy access
  - Various radiation safe feedthroughs
  - Dedicated space for a Dewar underneath the cabinet

Solely Available with IμS and Stand-Alone HV Generator

News: IncoatecTV on YouTube and

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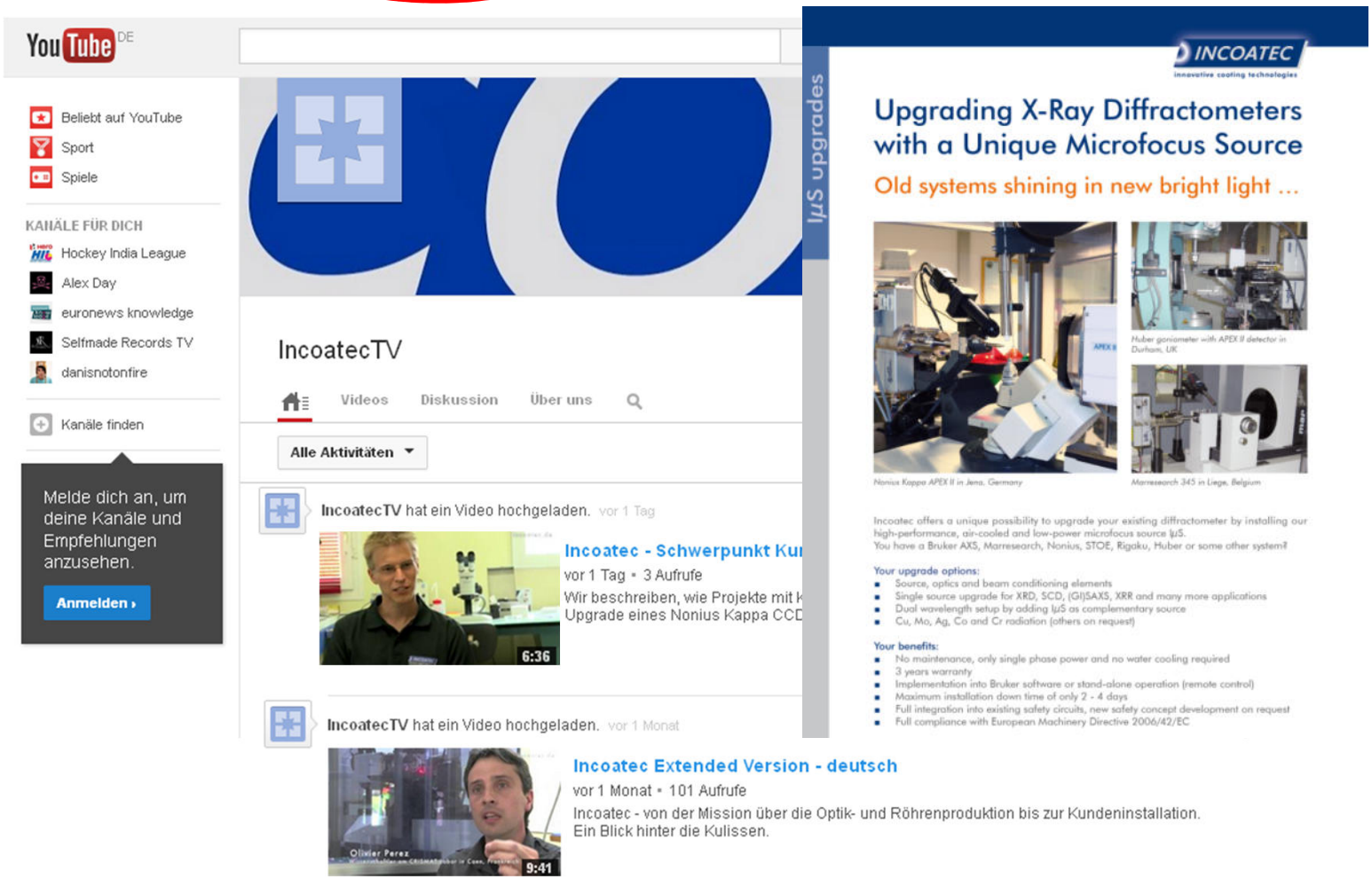
www.youtube.com/user/IncoatecTV

www.incoatec.de/download



Protein Screening

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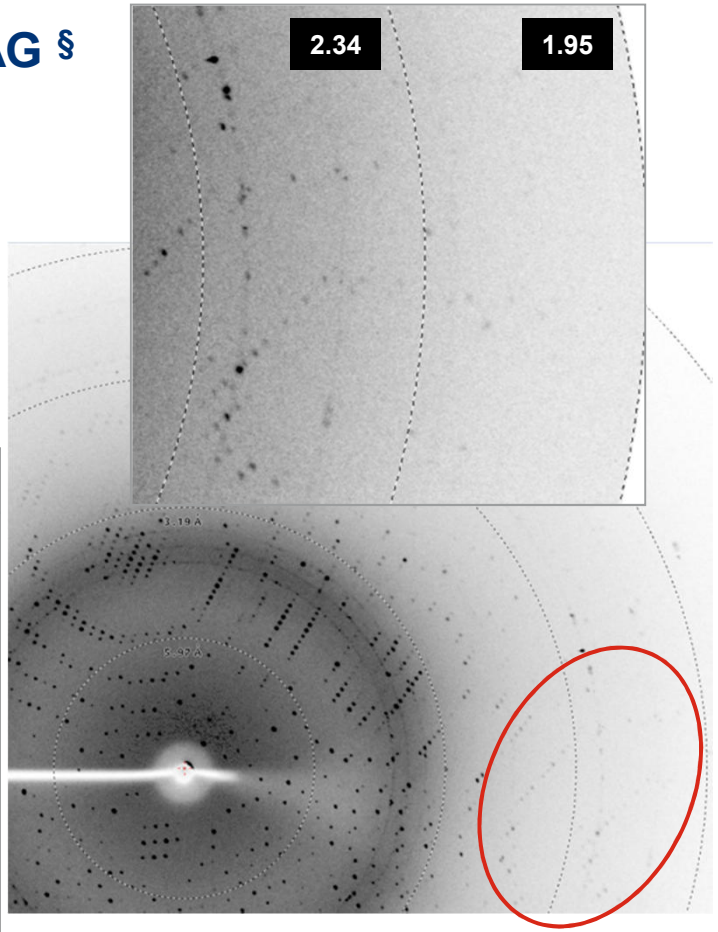
Very 1st Comparison of Cu-IμS *vs.* 4 kW Cu-RAG §

- Thermolysin Crystal (316 AA)
- Crystal Size: 0.20 x 0.06 x 0.04 mm^3
- Space group: $P6_122$
- Unit cell: $a = b = 92.8$ Å, $c = 130.0$ Å

Source	Cu-IμS	Rigaku RU 300
Detector	mar345	R-AXIS IV
Exposure time [min/°]	20	20
Resolution [Å]	**2.00 (2.03 - 2.00)**	**2.10 (2.14-2.10)**
R (sym) [%]	**8.1 (46.7)**	**9.7 (45.1)**
< I/σ >	**17.1 (3.2)**	**20.2 (4.4)**
Completeness [%]	97.9 (94.6)	99.9 (100)
Multiplicity	4.3 (4.5)	6.7 (6.6)

§ Rigaku RU 300 H3R, 1st generation multilayer mirror



Typical diffraction pattern of Thermolysin recorded with a Cu-IμS on a mardtb.

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Very 1st Comparison of Mo-IμS *vs.* 4 kW Mo-RAG §

	SiO_2		$CuSO_4$	
Size [mm³]	**0.04 x 0.04 x 0.02**		**0.07 x 0.05 x 0.05**	
Source	**Mo-IμS**	**FR 591**	**Mo-IμS**	**FR 591**
Power [W]	30	4000	20	4000
Exposure time [s]	**150 §**	**150 §**	**20 §**	**20 §**
Normalized < I >	17.1	5.9	175.6	80.6
< σ >	1.6	1.1	5.2	3.3
*R*1, *wR*2 [%]	**8.4, 24.0**	**9.0, 24.1**	**3.5, 11.5**	**3.9, 12.2**

§ Nonius FR591, flat graphite monochromator, 0.3 mm collimator, Nonius KappaCCD

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Very 1st Comparison of Mo-IμS *vs.* 4 kW Mo-RAG §

$CuSO_4$

Size [mm³]		.07 x 0.05 x 0.05	
Source		S	FR 591
Power [W]			4000
Exposure time [s]			**20 §**
Normalized < I >			80.6
< σ >			3.3
*R*1, *wR*2 [%]		5	**3.9, 12.2**

§ Nonius FR591, flat graphite monochromator, 0.3 mm collimator, Nonius KappaCCD

Mo-Kα for Chemical Crystallography

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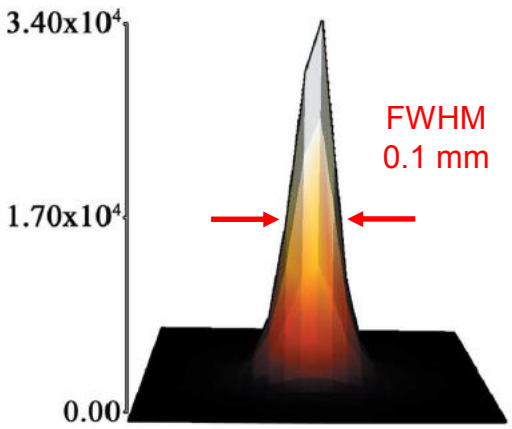
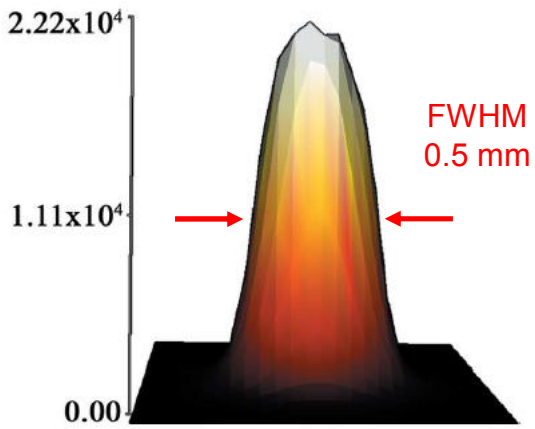
Comparison of beam profiles from a focusing multilayer X-ray mirror and from a flat graphite monochromator

Multilayer Mirror



Flat Graphite Monochromator



- Symmetric Gaussian shaped beam
- Average FWHM:
  - Mo-IμS: 0.110 mm
  - Ag-IμS: 0.095 mm

T. Schulz et al. , *J. Appl. Cryst.*, 2009, *42*, 885 – 891

Mo-Kα for Chemical Crystallography

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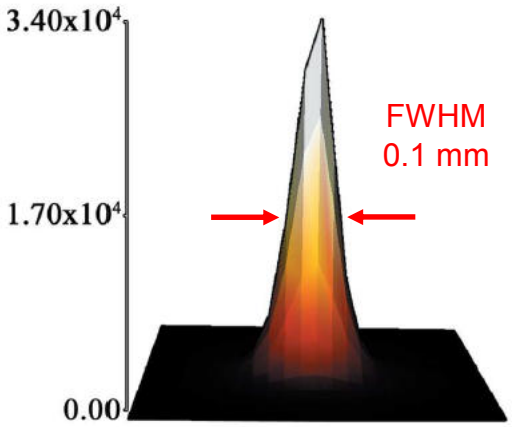
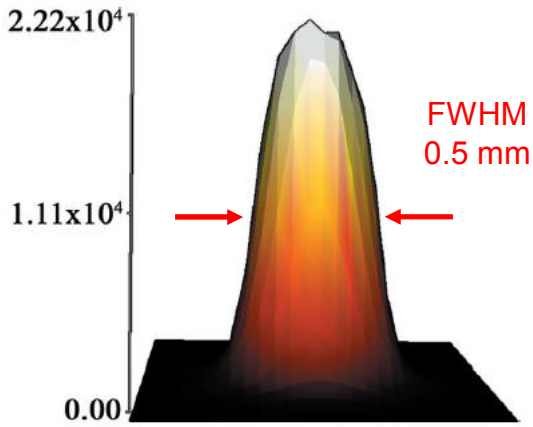
Comparison of beam profiles from a focusing multilayer X-ray mirror and from a flat graphite monochromator

Multilayer Mirror



Flat Graphite Monochromator



Maximum Intensity on Small Diameter

Ideal small crystals (in air or gasket)

- Mo-IμS: 0.110 mm
- Ag-IμS: 0.095 mm

T. Schulz et al. , *J. Appl. Cryst.*, 2009, *42*, 885 – 891

Mo-Kα for Chemical Crystallography

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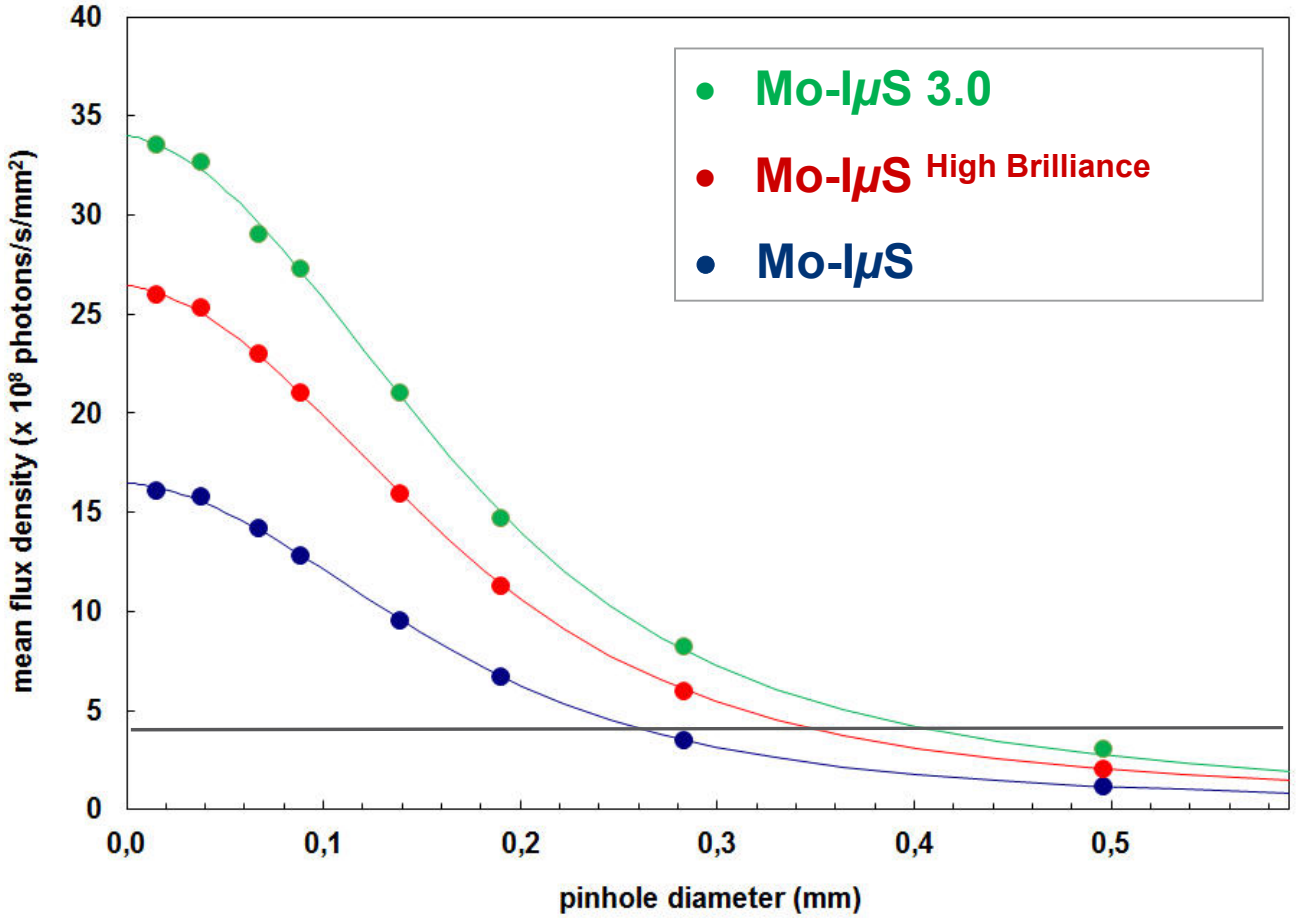
Flux through pinholes w/ different diameter (calibrated PN diode)



2 kW Mo ST

Comparison 2 kW Mo ST *vs.* Mo-IμS

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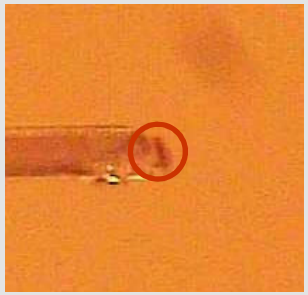
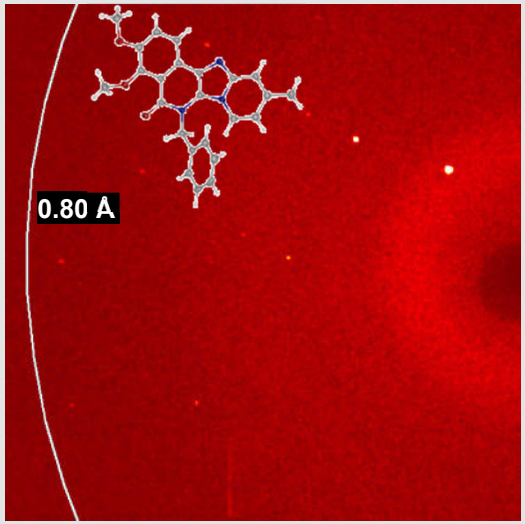
Data from Small Crystal of Organic Compound

Size [mm^3]	0.10 x 0.05 x 0.05	
Source	Mo ST	Mo-IμS
Power [W]	2000	30
Exposure time	90 s/0.3°	30 s/0.3°
Resolution [Å]	0.75 (0.85 – 0.75)	0.75 (0.85 – 0.75)
Normalized < I > *	19	139
<I/σ>	10.7 (2.1)	15.3 (3.5)
*R*1, *wR*2 [%]	5.55, 10.44	3.88; 9.16
Distance N – C [Å]	1.325(4)	1.325(3)

$C_{24}H_{21}N_3O_3$

*** Normalized to 1 °/min**





Typical diffraction pattern ($P2_1$, a = 8.3628(6) Å, b = 7.0469(5) Å, c = 15.9737(11) Å, β = 92.210(1)°, Z = 2)

T. Schulz et al. , *J. Appl. Cryst.*, 2009, *42*, 885 – 891

Comparison 2 kW Mo ST *vs.* Mo-IμS

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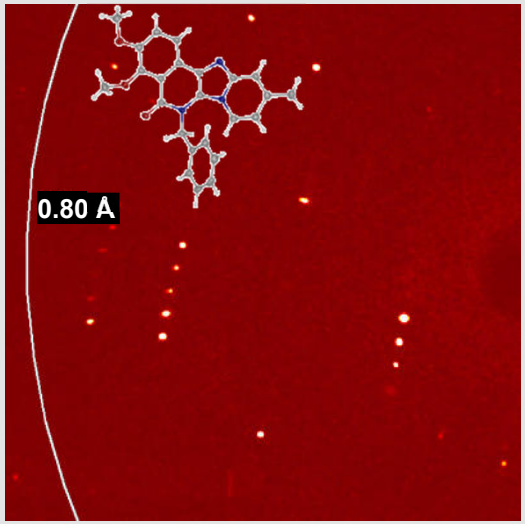
Data from Larger Crystal of Organic Compound

Size [mm³]	0.30 x 0.25 x 0.15	
Source	Mo ST	Mo-IμS
Power [W]	2000	30
Exposure time	15 s/0.3°	10 s/0.3°
Resolution [Å]	0.75 (0.85 – 0.75)	0.75 (0.85 – 0.75)
Normalized < I > *	1286	4763
<I/σ>	30.4 (13.6)	37.6 (21.1)
*R*1, *wR*2 [%]	3.17, 8.16	3.08; 8.18
Distance N – C [Å]	1.324(2)	1.323(2)

$C_{24}H_{21}N_3O_3$

*** Normalized to 1 °/min**



Typical diffraction pattern ($P2_1$, a = 8.3628(6) Å, b = 7.0469(5) Å, c = 15.9737(11) Å, β = 92.210(1)°, Z = 2)

T. Schulz et al. , *J. Appl. Cryst.*, 2009, *42*, 885 – 891

Comparison 2 kW Mo ST *vs*. Mo-I*μ*S HB

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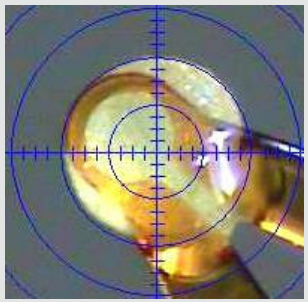
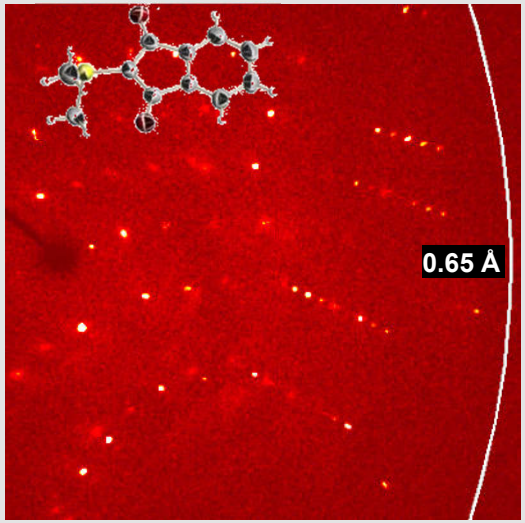
Ylid Data from 2 kW Mo ST vs. Mo-I*μ*S HB

Size [mm³]	0.40 x 0.40 x 0.40	
Source	Mo ST	Mo-I*μ*S HB
Power [W]	2000	50
Exposure time	20 s/°, 4 h	20 s/°, 4 h
Resolution [Å]	0.65 (0.75 – 0.65)	0.65 (0.75 – 0.65)
Normalized < *I* >	2252.7	12285.4
<I/σ>	27.9 (7.1)	46.6 (17.3)
*R*1, *wR*2 [%]	3.36, 9.02	2.97; 8.37
Flack *x*(*u*)	0.02(3)	0.04(1)
Distance C – C [Å]	1.398(3)	1.399(2)

Better Data despite the fact that Crystal is Larger than the Beam





Typical diffraction pattern ($P2_12_12_1$, a = 5.9598(2) Å, b = 9.0361(2) Å, c = 18.3870(5) Å, Z = 4)

Mo-IμS HB *vs.* Mo-IμS 3.0

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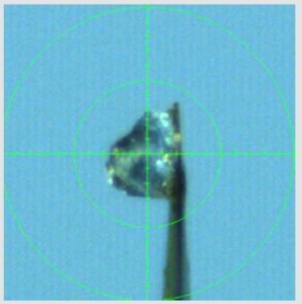
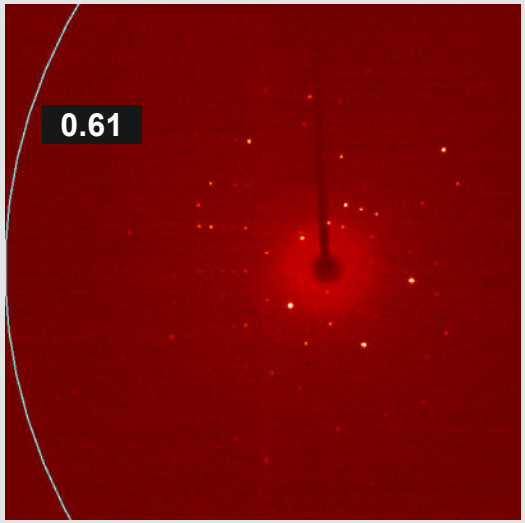
Real Life Example: Diphenyloxazolidin-2-one Derivate

Source	Mo-IμS HB	Mo-IμS 3.0
Detector	**PHOTON100**	**PHOTON100**
Exposure time	**35 s/0.3°, 15 h**	**35 s/0.3°, 15 h**
Resolution [Å]	**0.64** (0.74 – **0.64**)	**0.61** (0.71 – **0.61**)
Multiplicity	9.3 (4.5)	8.4 (2.8)
<I/σ>	**23.0 (3.1)**	**27.5 (3.4)**
*R*1, *wR*2 [%]	**7.17, 14.77**	**6.53, 14.36**
d(C-C) [Å]	1.387(**4**)	1.390(**3**)
Parsons *z*(*v*)	**0.0(3)**	**0.1(2)**

$C_{25}H_{31}NO_5$, 0.12 x 0.10 x 0.09 mm^3,
comparable strategies





Typical diffraction pattern ($P2_12_12_1$,
a = 8.4394(2) Å, b = 13.7322(4) Å,
c = 19.3182(6) Å, Z = 4)

Evolution of Data Quality

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Comparison of Different Instrument Generations with a Real Life Crystal

- Comparison of data from same purely organic crystal, collected at 100 K with Mo-Kα radiation and comparable data collection strategies

APEX II QUAZAR:

Mo-IμS w/ APEX II

D8 VENTURE:

Mo-IμS HB w/ PHOTON100

D8 VENTURE 2nd Gen.:

Mo-IμS 3.0 w/ PHOTON II

Evolution of Data Quality

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Diphenyloxazolidin-2-one Derivate: Increased Depth of Information

Source	Mo-IμS	Mo-IμS HB	Mo-IμS 3.0
Detector	**APEX II**	**PHOTON100**	**PHOTON II**
Exposure time	**20 s/0.3°, 17 h**	**35 s/0.3°, 15 h**	**35 s/0.3°, 15 h**
Max. Res. [Å]	**0.73**	**0.64**	**< 0.56**
Resolution [Å]	0.80 (0.90 – 0.80)	0.80 (0.90 – 0.80)	0.80 (0.90 – 0.80)
Multiplicity	10.1 (**8.4**)	12.7 (**9.1**)	11.7 (**9.3**)
<I/σ>	32.0 (**4.8**)	41.6 (**9.7**)	**55.2 (18.8)**
*R*1(all), *wR*2(all) [%]	**4.24, 11.48**	**7.17, 14.77**	**8.11, 18.43**
d(C-C) [Å]	1.384(**4**)	1.387(**4**)	1.390(**3**)
Parsons *z*(*v*)	**0.0(4)**	**0.0(3)**	**0.1(2)**
Highest peak [eÅ^{-3}]	**0.14**	**0.36**	**0.63**

Evolution of Data Quality

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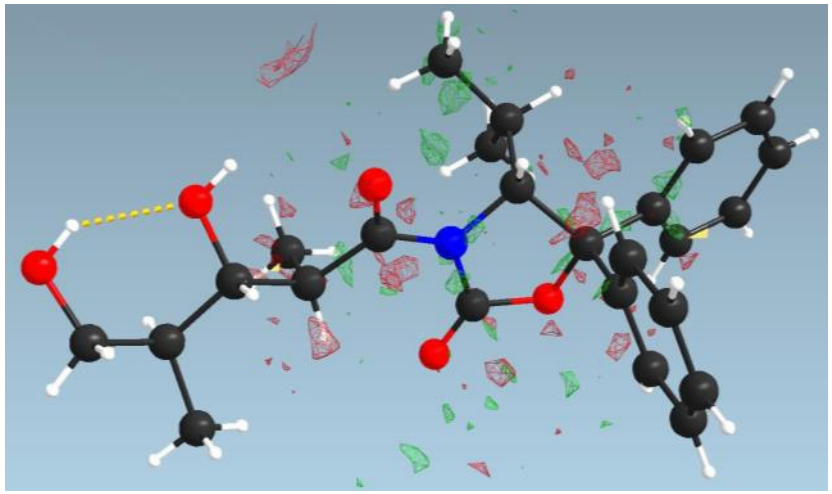
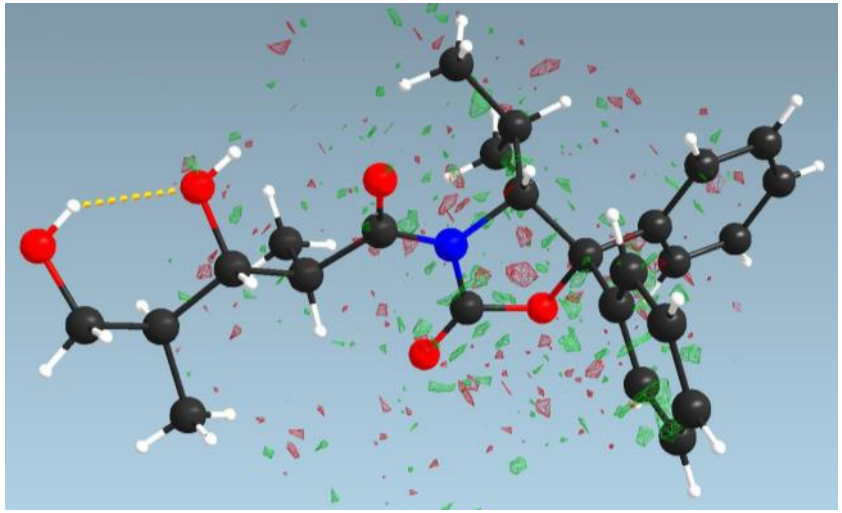
Real Life Example: Diphenyloxazolidin-2-one Derivate: Residual Densities



Mo-IμS plus APEX II, max. resolution 0.73 Å, ±**0.08 e/Å^3**



Mo-IμS HB plus PHOTON100, max. resolution 0.64 Å, ±**0.25 e/Å^3**

Evolution of Data Quality

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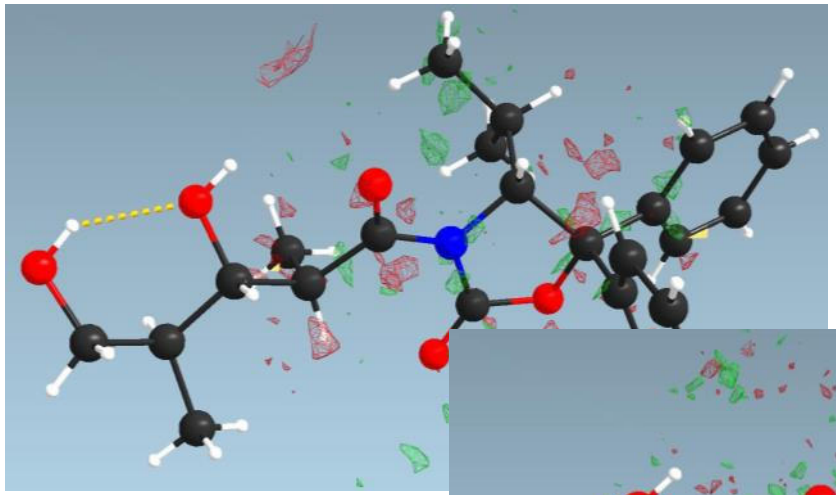
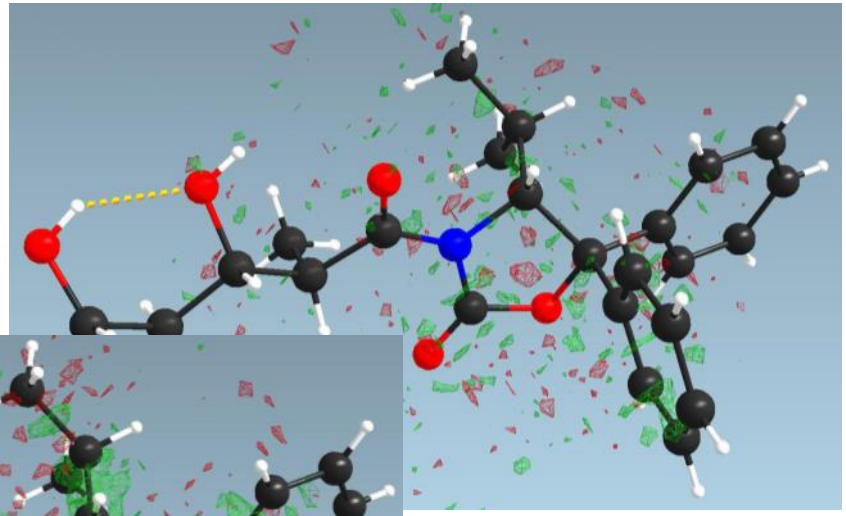
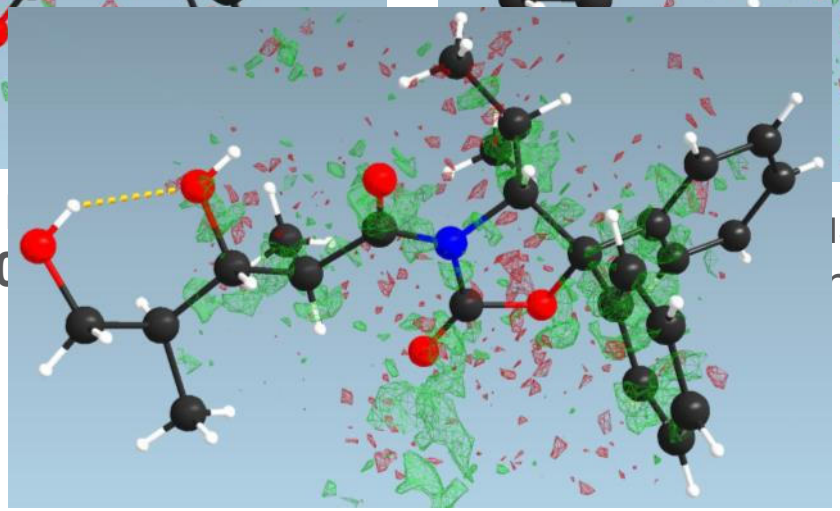
Real Life Example: Diphenyloxazolidin-2-one Derivate: Residual Densities



Mo-IμS plus APEX I
resolution 0.73 Å, ±0



s PHOTON100,
n 0.64 Å, ±**0.25 e/Å^3**



Mo-IμS 3.0 plus PHOTON II,
max. resolution < 0.56 Å, ±**0.25 e/Å^3**

Ag-Kα for Solid State Chemistry and High-Pressure Crystallography

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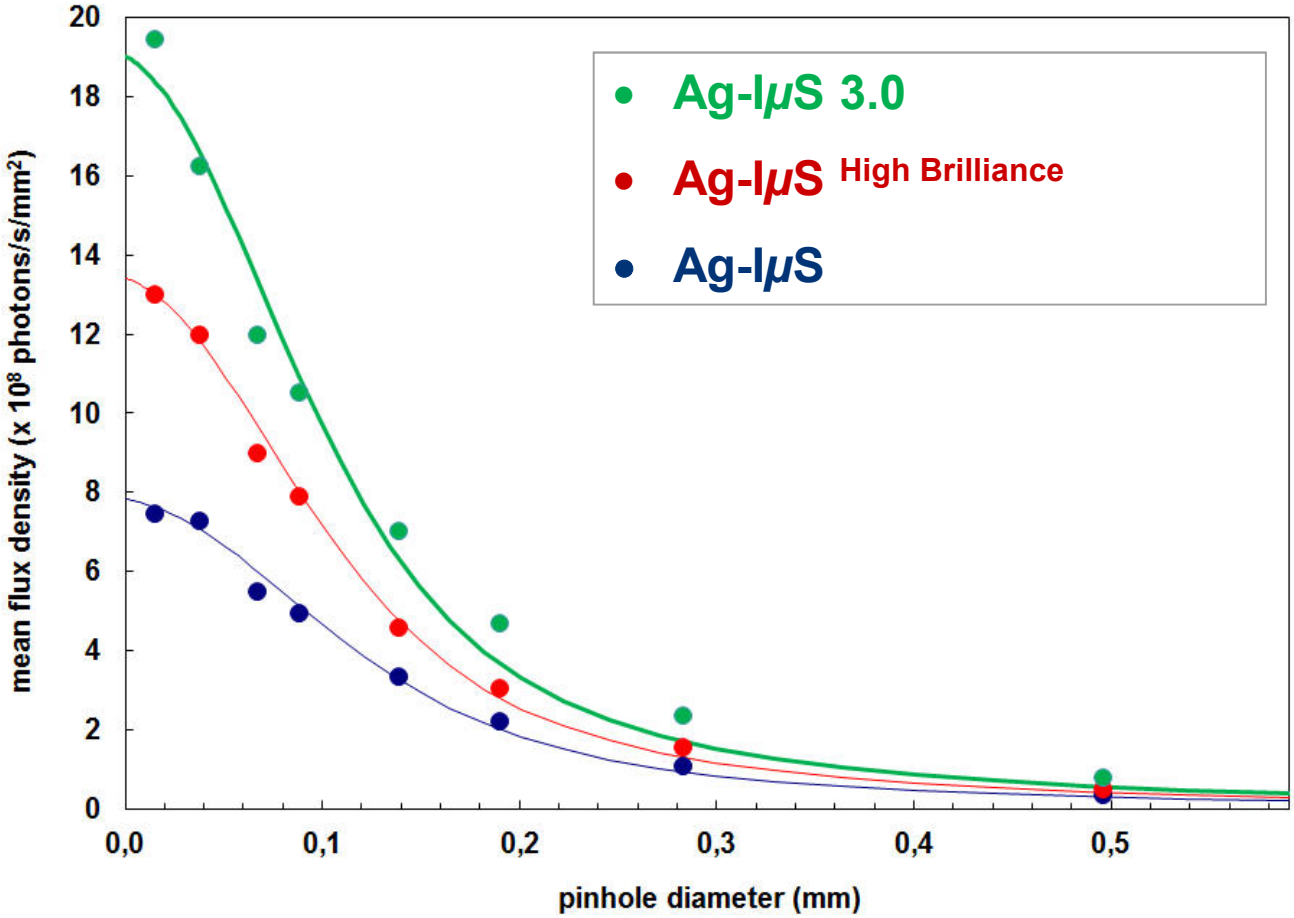
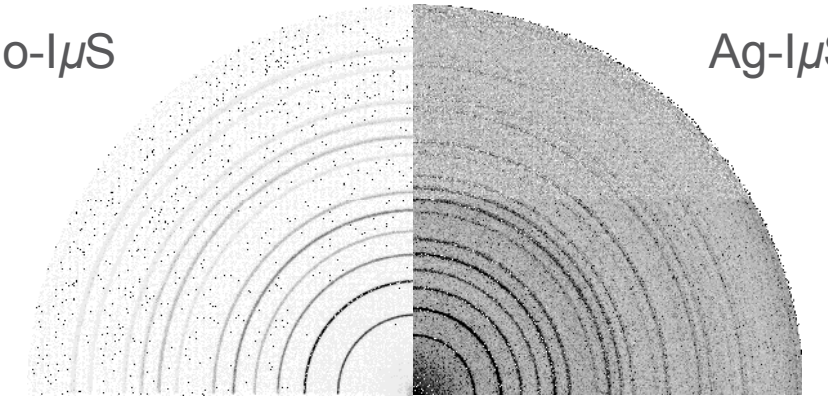
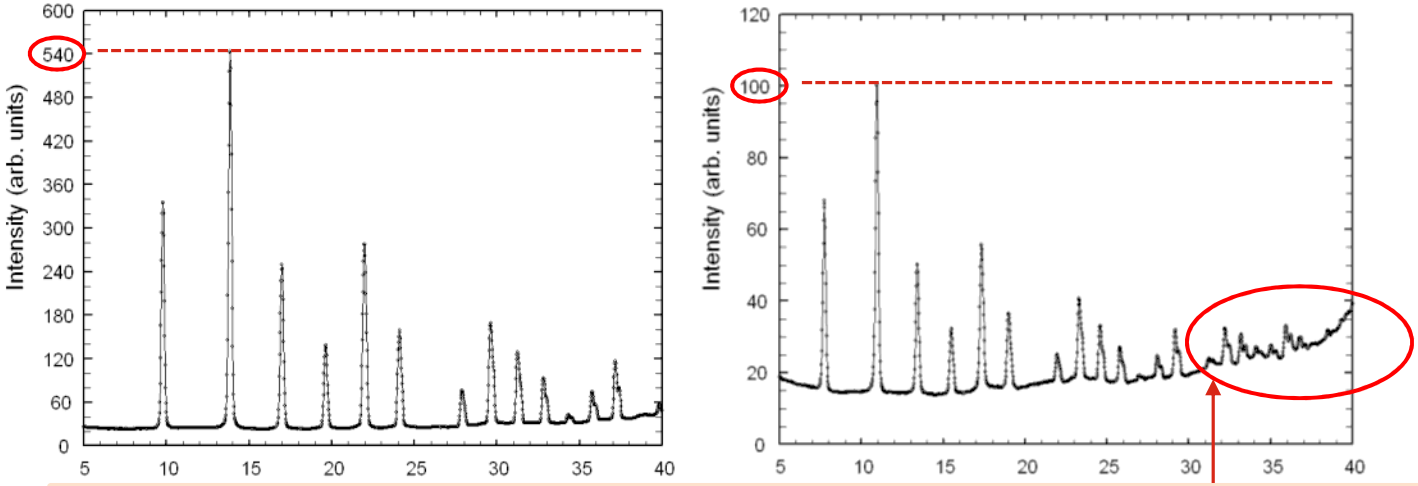
Flux through pinholes w/ different diameter (calibrated PN diode)



Which Wavelength to Use?



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



Ag-Kα for low absorbing materials:
2x More Data, but Less Diffracted Intensity

In-House X-ray Sources for High-Pressure Crystallography

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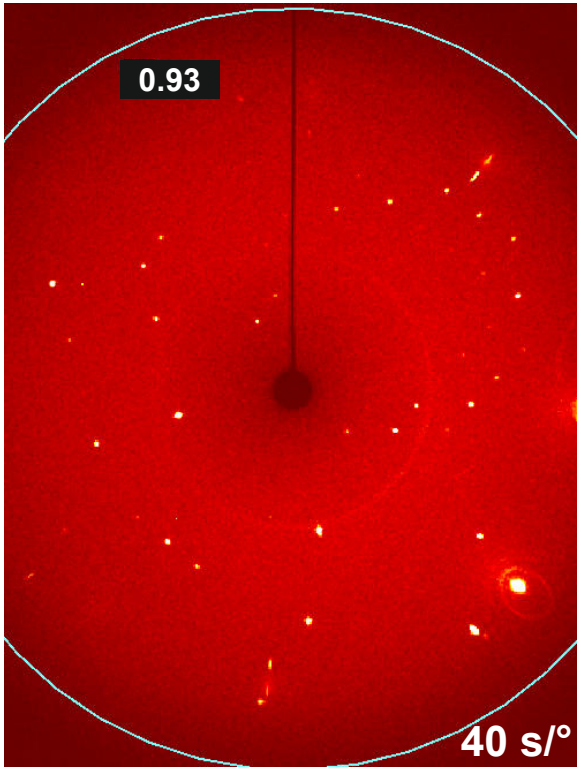
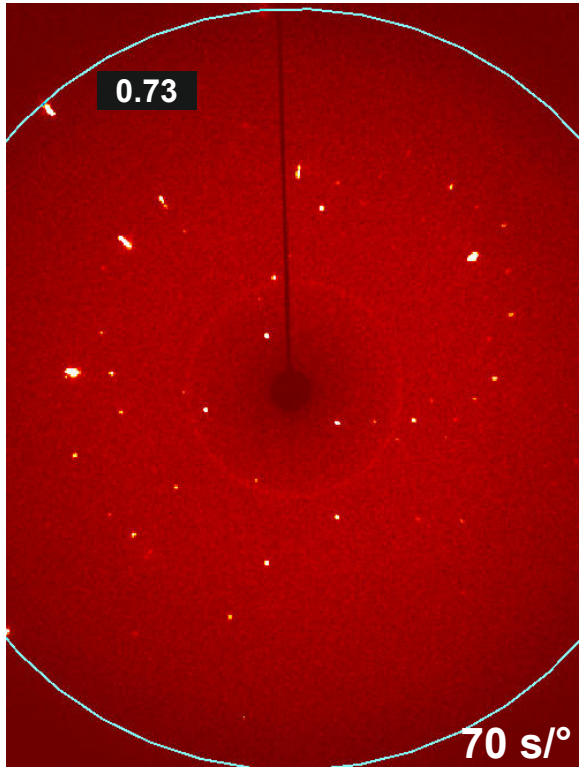
Purely Organic Small Molecule Compound in a DAC

Mo-IμS 3.0



Ag-IμS 3.0



Accessible Data	1	2

In-House X-ray Sources for High-Pressure Crystallography

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Comparison of High-Pressure Data from a Purely Organic Compound

Source	Mo-IμS 3.0	Ag-IμS 3.0
Exposure time	**40 s/°, 12 h**	**70 s/°, 22 h**
Max. Access. Res.	**0.65 Å**	**0.50 Å**
Max. Res. [Å]	**0.65**	**0.58**
# Unique	**368 (47)**	**686 (174)**
Resolution [Å]	0.65 (0.75 – 0.65)	0.65 (0.75 – 0.65)
Complet [%]	**21.9 (8.3)**	**32.8 (24.8)**
Multiplicity	3.8 (**0.8**)	5.4 (**2.9**)
<I/σ>	**91.8 (16.8)**	**48.0 (8.2)**
*R*1(all), *wR*2(all) [%]	3.89, 11.77	4.05, 10.94
d(C-C) [Å]	**1.524(11)**	**1.516(6)**

~0.15 mm diameter, $P2_1$,

Cu-Kα for Chemical and Biological Crystallography

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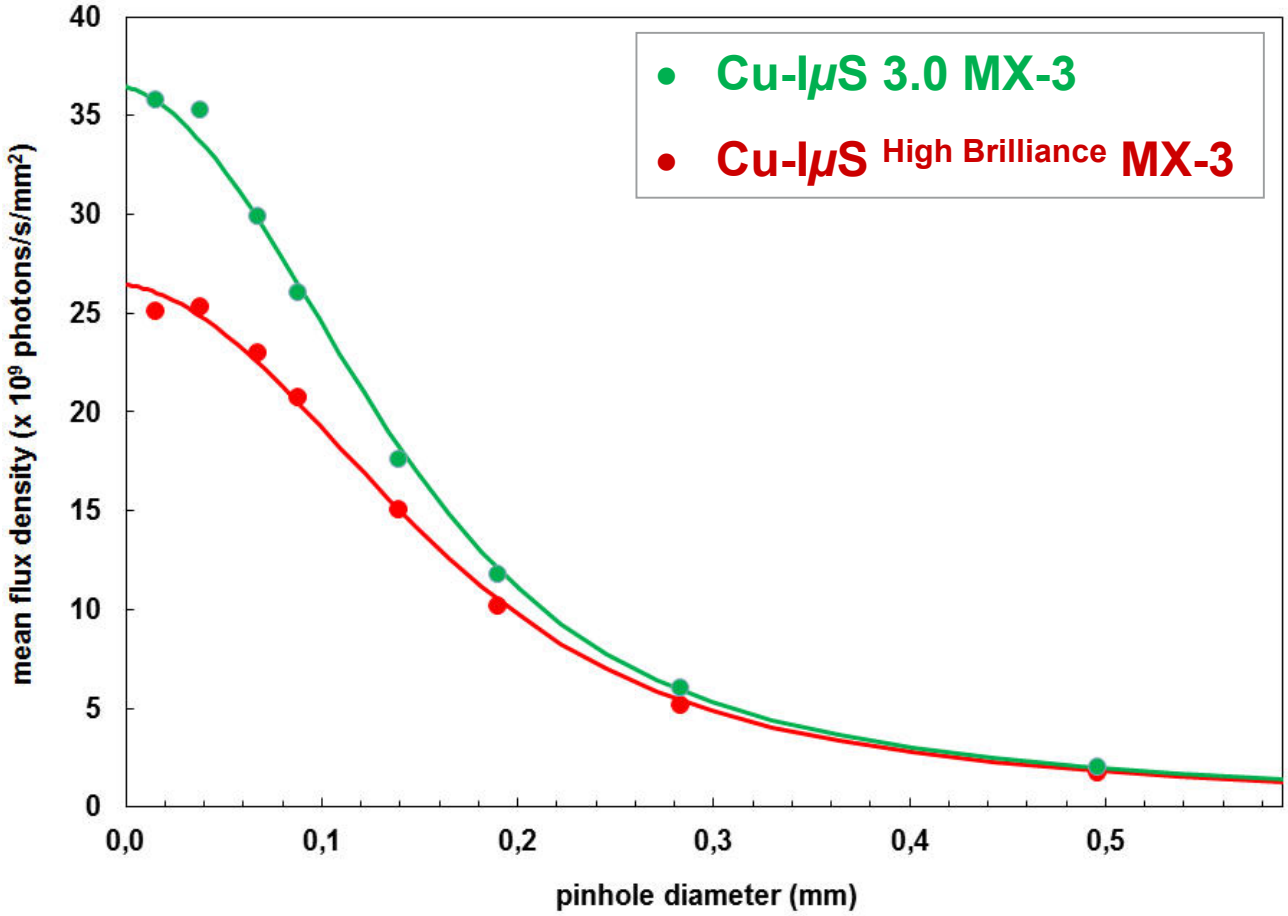
Flux through pinholes w/ different diameter (calibrated PN diode)



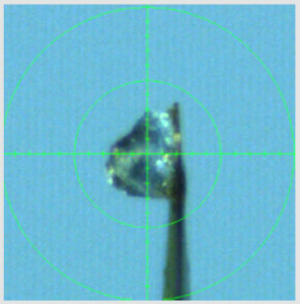
Cu-IμS HB *vs.* Cu-IμS 3.0

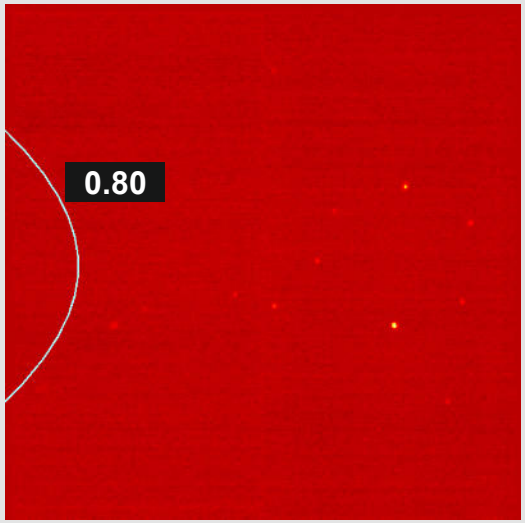
INCOATEC
innovative coating technologies

Real Life Example: Diphenyloxazolidin-2-one Derivate

Source	Cu-IμS HB	Cu-IμS 3.0
Detector	**PHOTON100**	**PHOTON100**
Exposure time	**4 – 12 s/°, 5 h**	**4 – 12 s/°, 5 h**
Resolution [Å]	**0.78** (0.88 – 0.78)	**0.78** (0.88 – 0.78)
Multiplicity	**10.8 (7.5)**	**10.7 (7.5)**
<I/σ>	**27.7 (11.7)**	**32.2 (14.8)**
*R*1, *wR*2 [%]	3.88; 9.88	**3.61; 9.20**
d(C-C) [Å]	1.386(4)	1.389(**3**)
Parsons *z*(*v*)	**0.07(9)**	**0.05(7)**

$C_{25}H_{31}NO_5$, 0.12 x 0.10 x 0.09 mm^3,
comparable strategies





Typical diffraction pattern ($P2_12_12_1$, *a* = 8.4167(17) Å, *b* = 13.761(3) Å, *c* = 19.304(4) Å, *Z* = 4)

Cu-Kα for Chemical and Biological Crystallography

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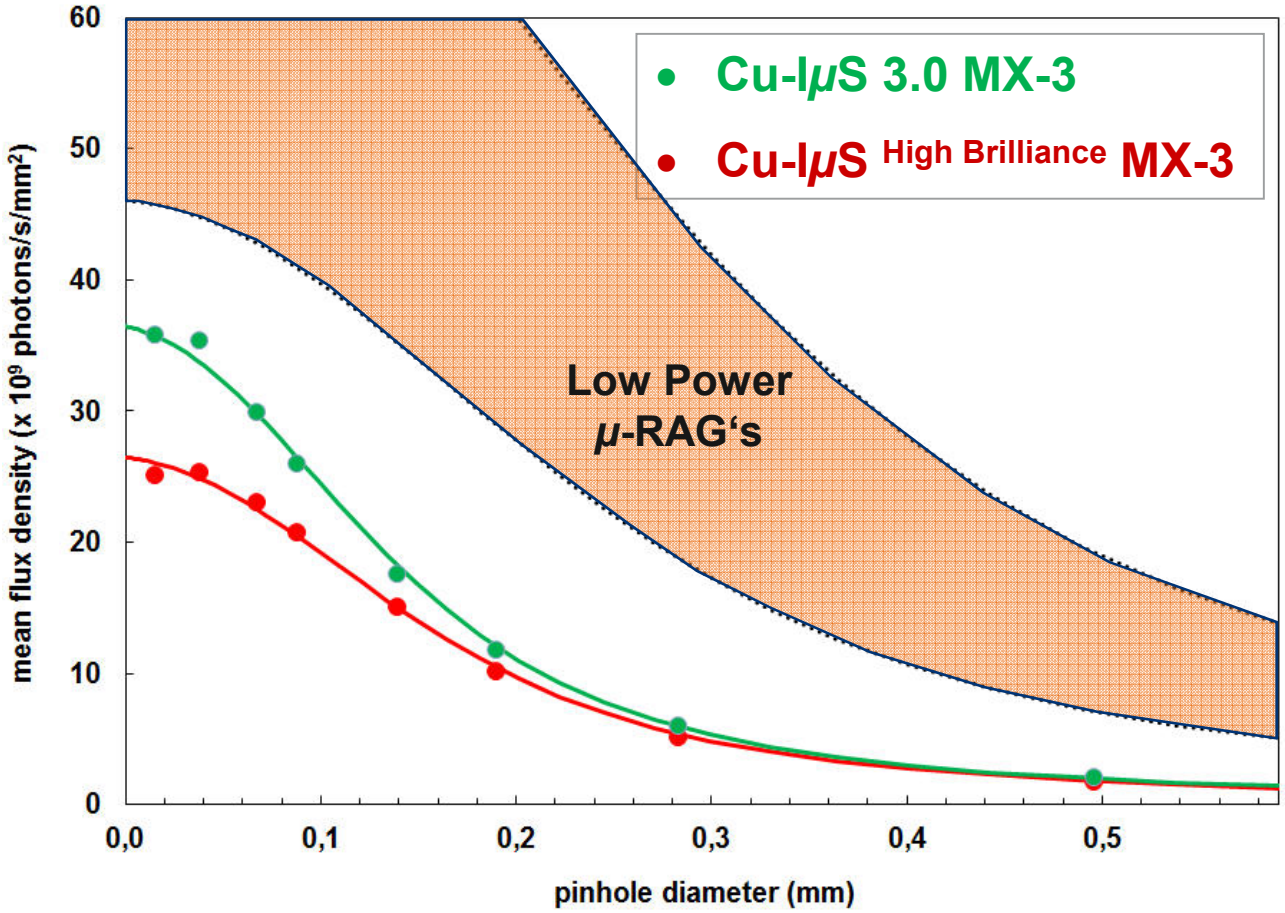
Flux through pinholes w/ different diameter (calibrated PN diode)



Cu-Kα for Chemical and Biological Crystallography

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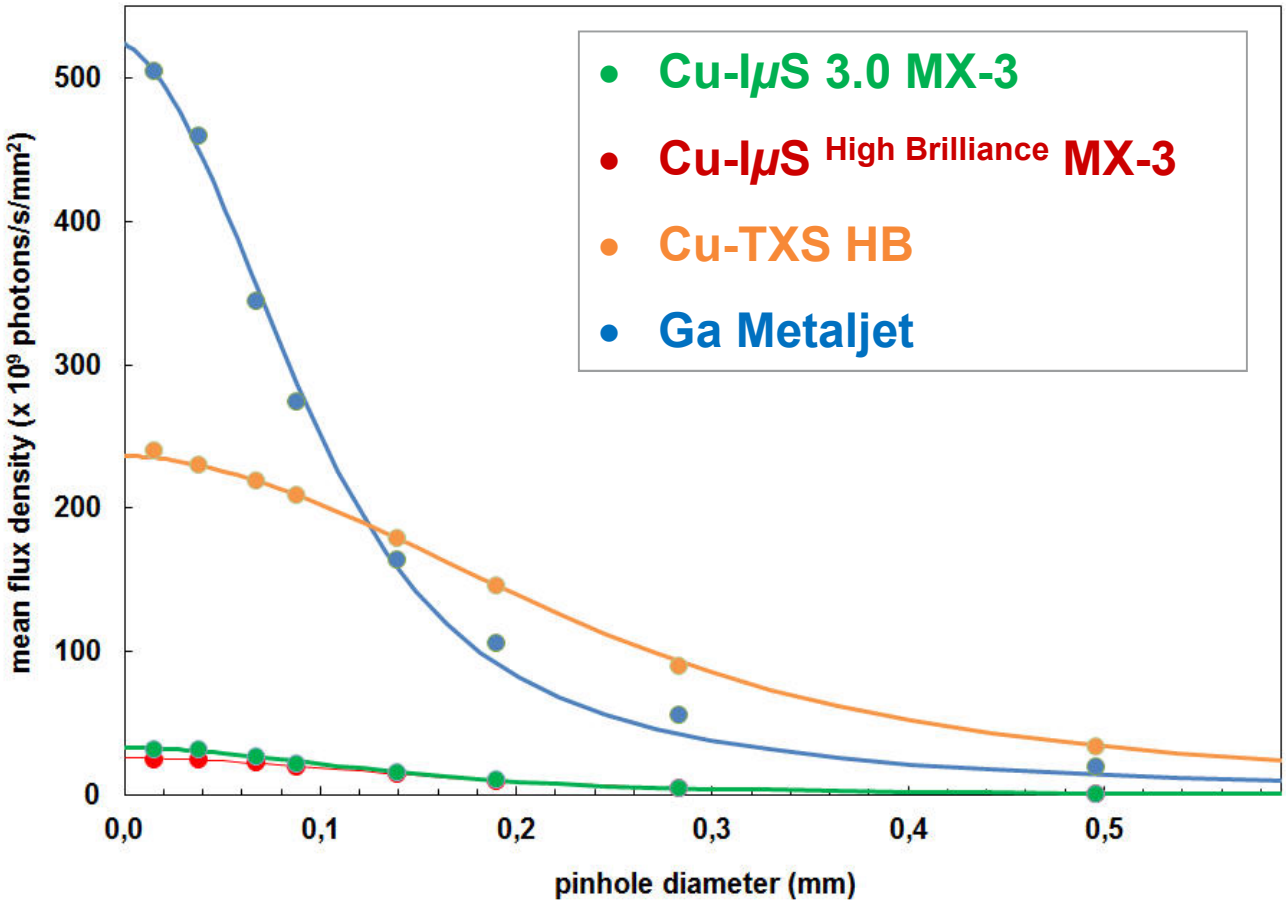
Flux through pinholes w/ different diameter (calibrated PN diode)



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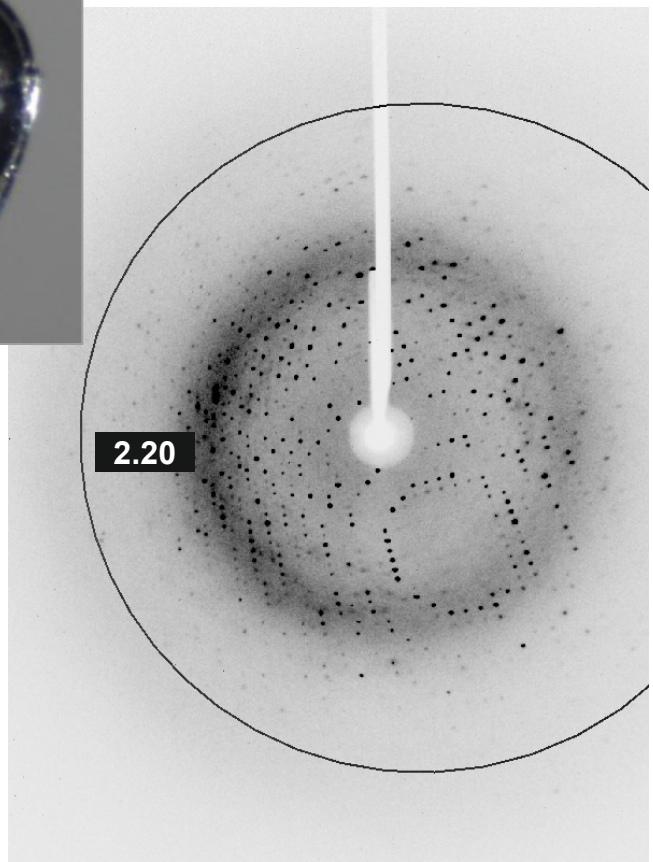
215° Data Collection on Thin Crystal of Human NEIL1 (Endonuclease VIII-like protein)

- Space group: *H*3
- Crystal size 0.15 x 0.12 x 0.02 mm^3
- Resolution on µ-RAG ~ 2.6 Å



Source	Cu-IμS 3.0
Exposure time	**100 s/0.5°**
Total time [h]	12
Resolution [Å]	**2.25 (2.32 – 2.25)**
Completeness [%]	99.6 (99.7)
Multiplicity	5.1 (3.5)
$\langle I/\sigma(I)\rangle$	**13.2 (2.6)**
R_{merge}	7.07 (32.34)
$CC_{½}$ at High res	89%

How Small? – Testing the Limits

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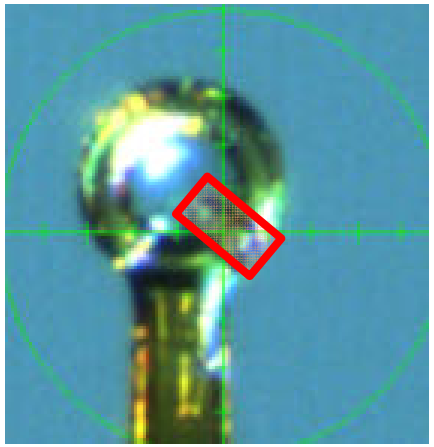
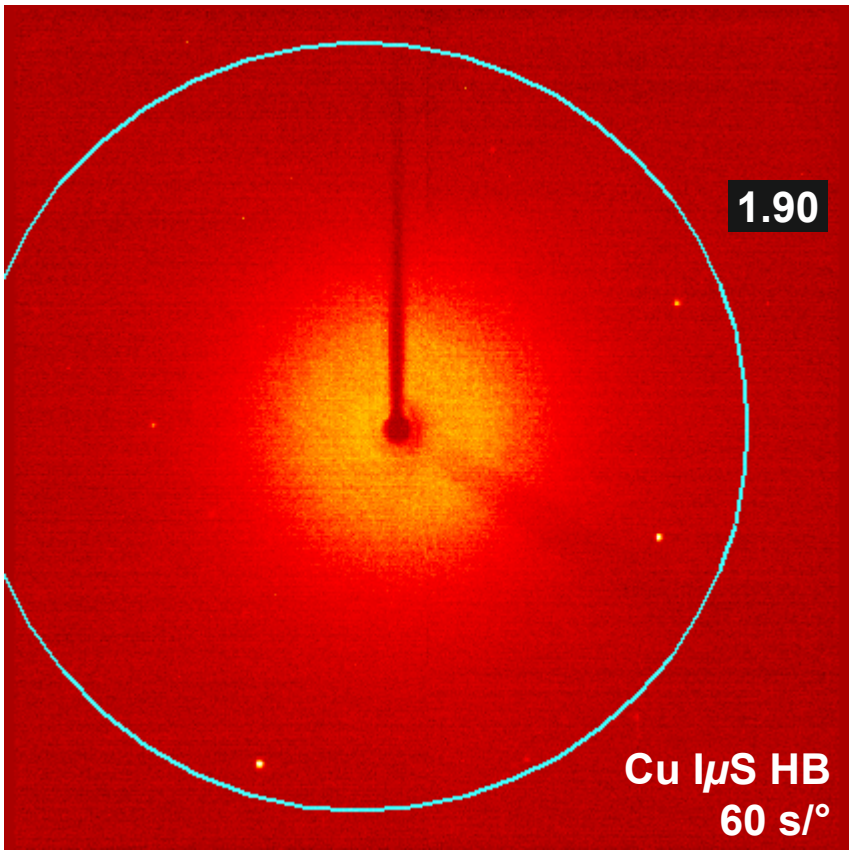
innovative coating technologies

Typical Diffraction Patterns from a 0.03 x 0.02 x 0.01 mm^3 Crystal (Purely Organic)





How Small? – Testing the Limits

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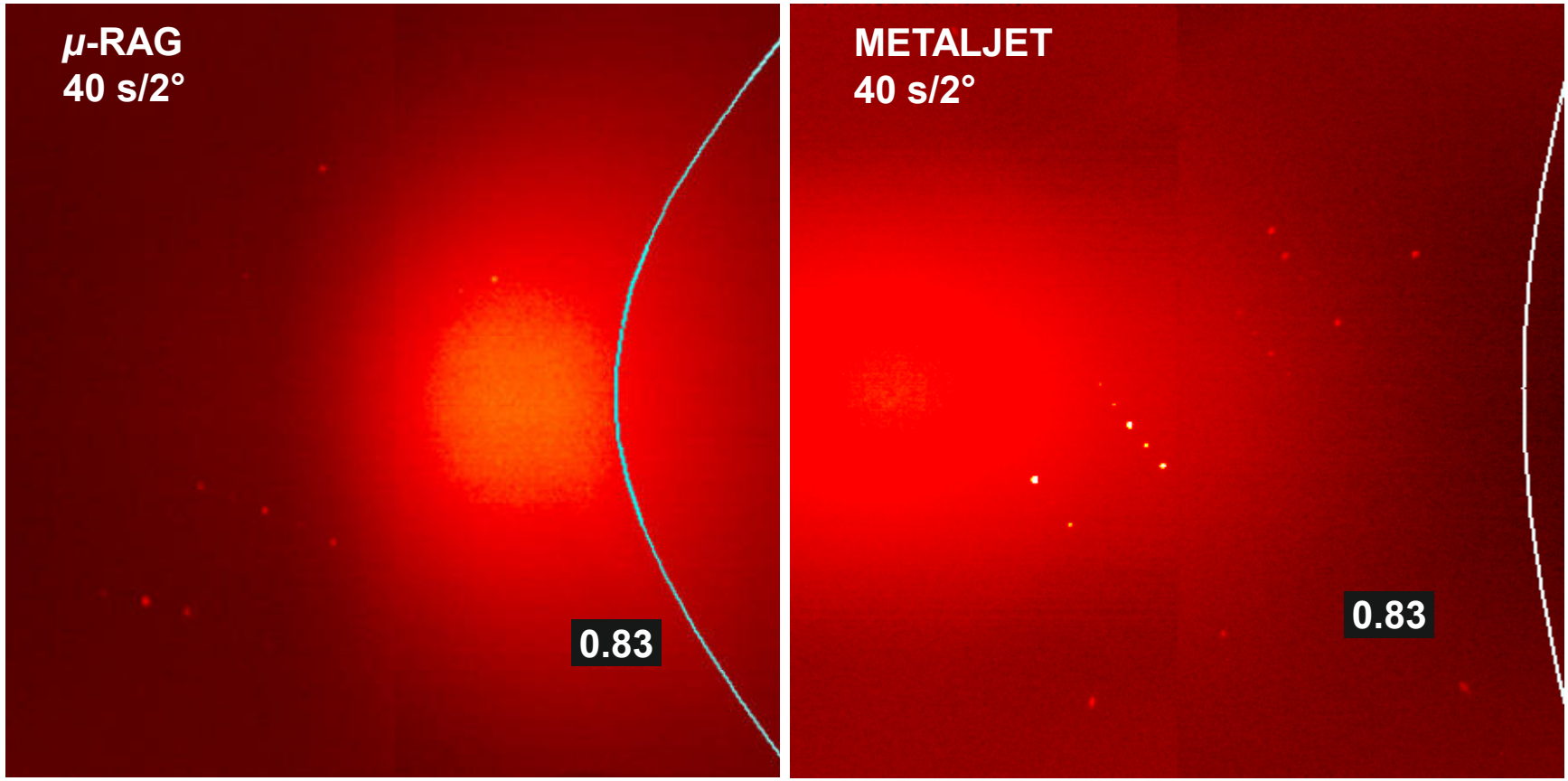
Perfect Sample for Diffraction on High-Brightness X-ray Source



Many More Applications: Charge Density

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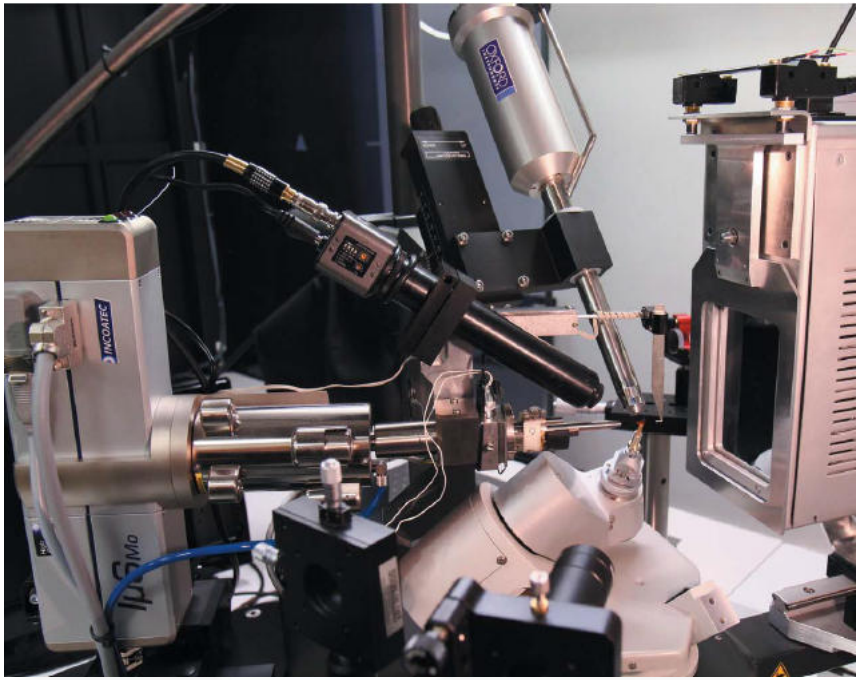
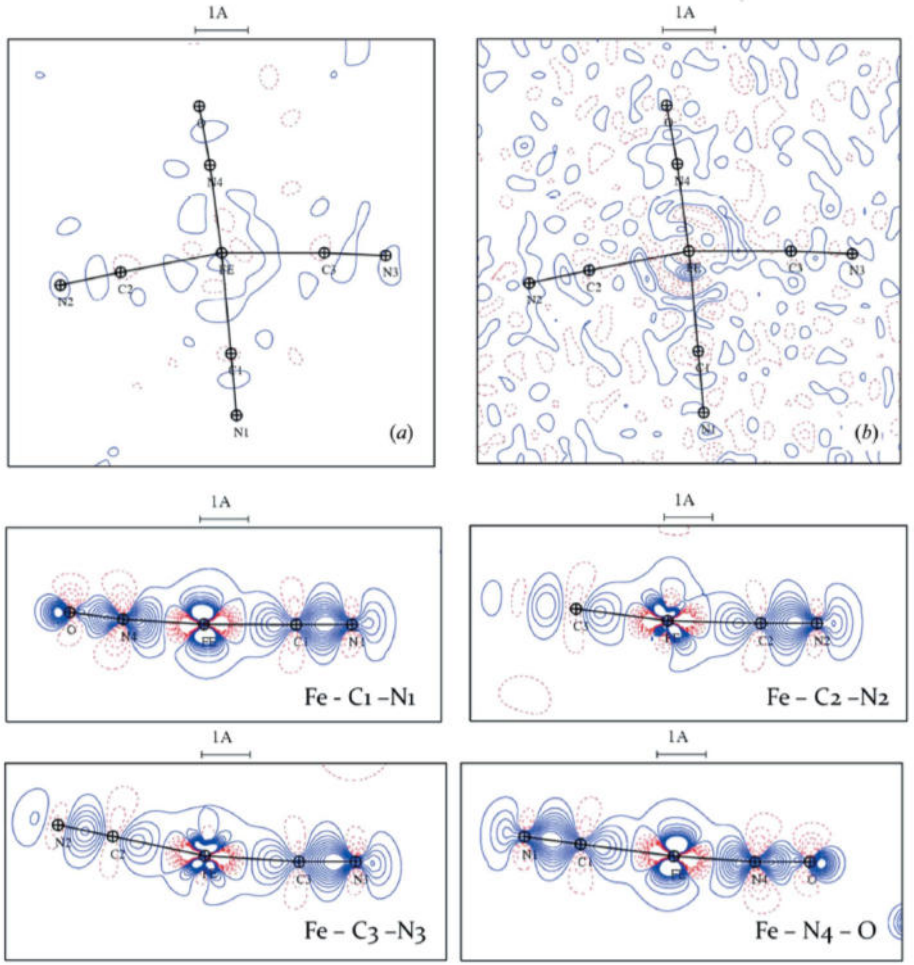
Charge density study on a sodium nitroprusside crystal



Dr. E. Wenger, Prof. D. Schaniel,
Prof. C. Lecomte,
Université de Lorraine, Nancy



E. Wenger *et al.*, *Acta Cryst.* 2014, *B70*, 783 – 791

Many More Applications: Charge Density

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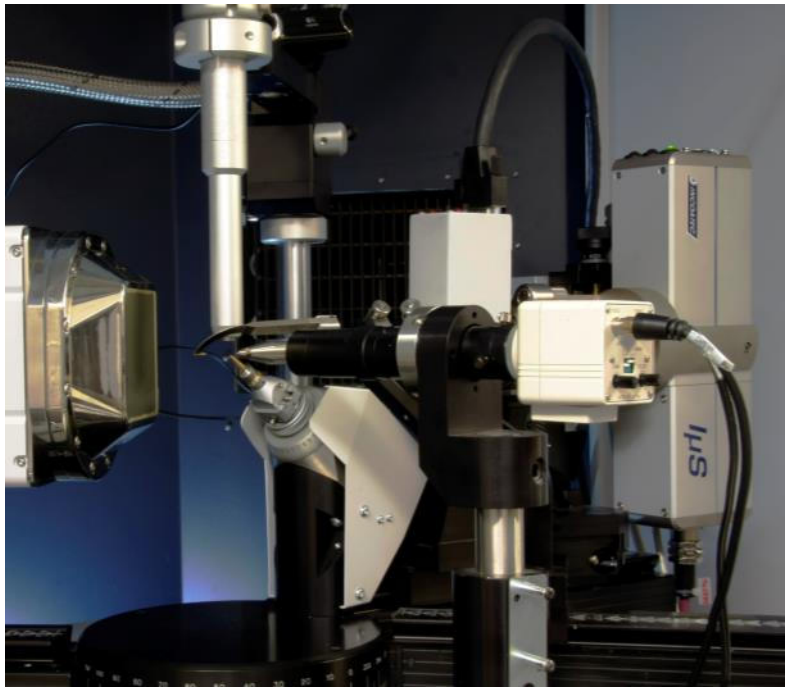
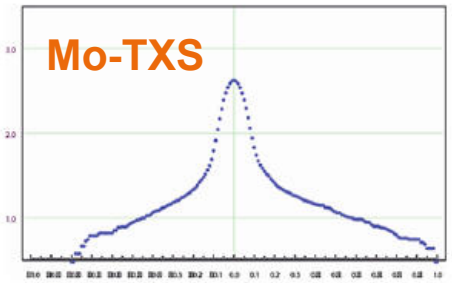
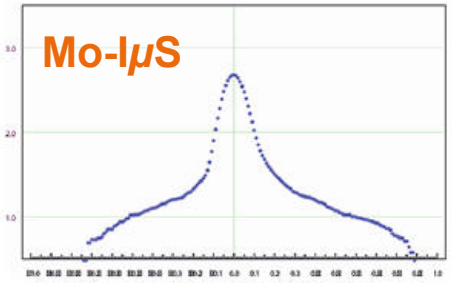
innovative coating technologies

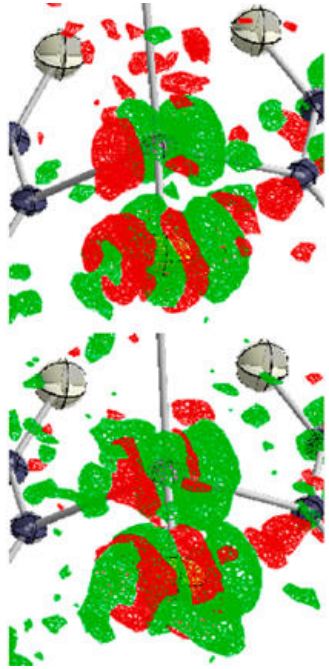
Charge density study on a 9-diphenyl-thiophosphinoyl-anthracene crystal: Comparison Mo-IμS vs. Mo-TXS data



Dr. R. Herbst-Irmer, Prof. D. Stalke,
Georg-August-Universität Göttingen







R. Herbst-Irmer *et al.*, *J. Phys. Chem.* 2013, *A 117*, 633 − 641

Many More Applications: High-Pressure XRD

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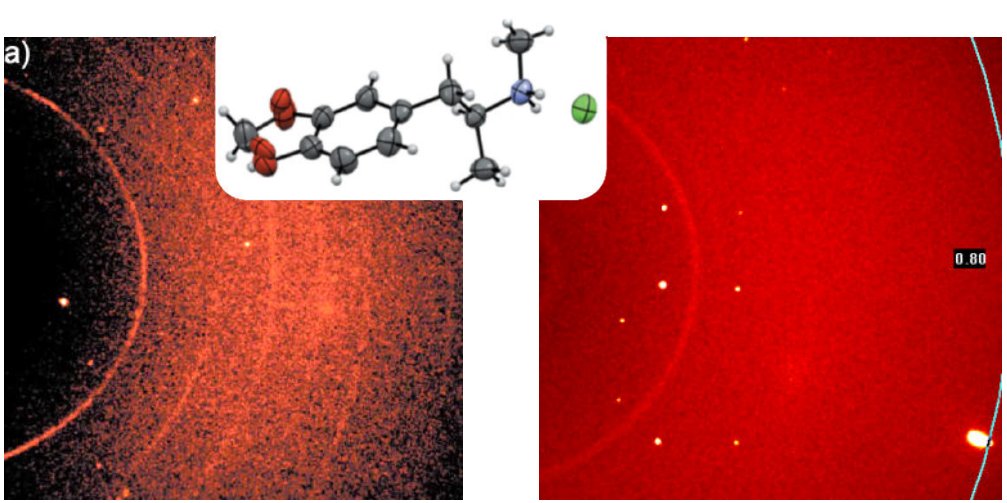
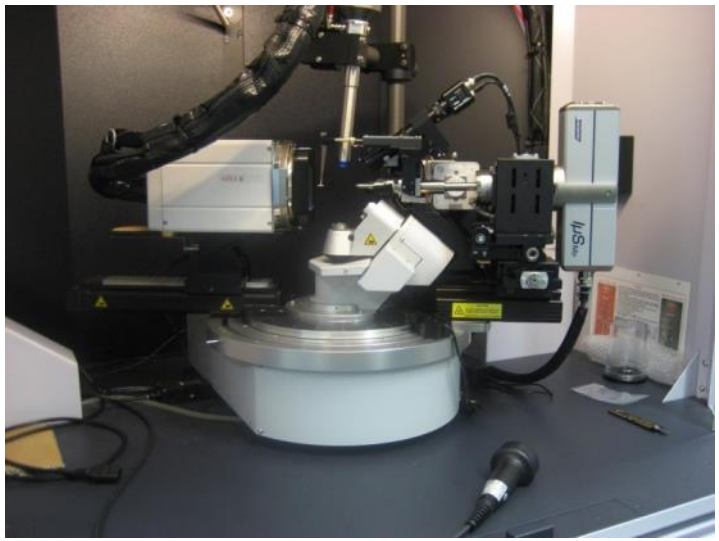
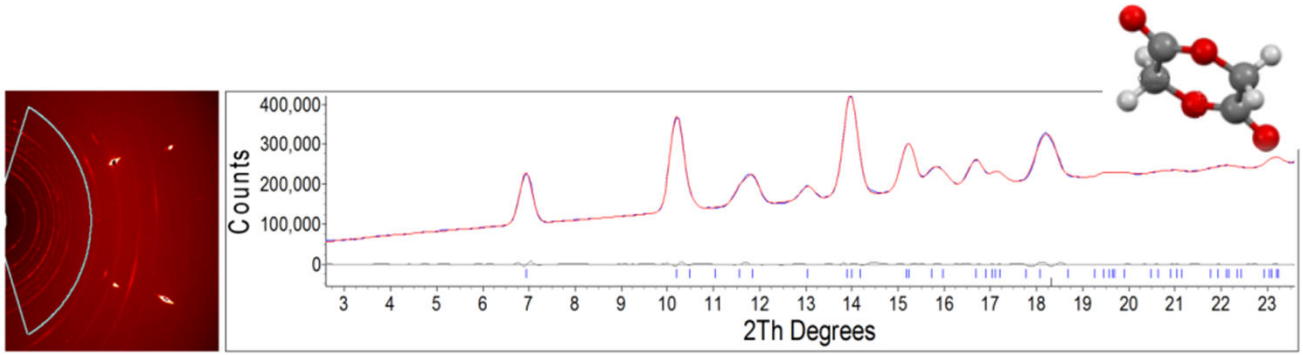
Single Crystal and Powder Diffraction Studies of Pharmaceutical Compounds under High Pressure







L. E. Connor et al. *Acta Cryst.* (2015). *B 71*, 3 – 9
I. B. Hutchison et al. *CrystEngComm*, (2015),*17*, 1778 – 1782

Many More Applications: Small Crystals

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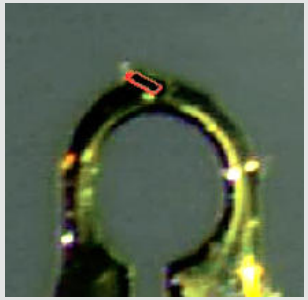
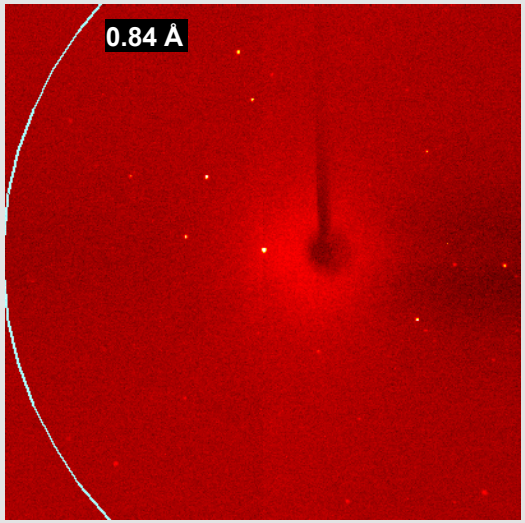
Tiny Crystal of MOF Compound

Size [mm³]	0.018 x 0.010 x 0.002
Source	Mo-IμS HB
Power [W]	50
Exposure time	240 s/°, 22 h
Resolution [Å]	0.83 (0.93 – 0.83)
Multiplicity	3.9 (2.2)
<I/σ>	15.1 (4.2)
*R*1, *wR*2 [%]	4.27; 8.83

$C_{18}H_{28}LaO_{23}P_6$





Typical diffraction pattern ($P2_1/m$, a = 8.9215(10) Å, b = 21.209(2) Å, c = 9.1052(10) Å, β = 107.923(3)°, Z = 2)

F. A. Almeida Paz , H. Ott *et. al.*, *Chem. Commun.* 2013, *49*, 6400 – 6402

Many More Applications: Small Crystals

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BRUKER

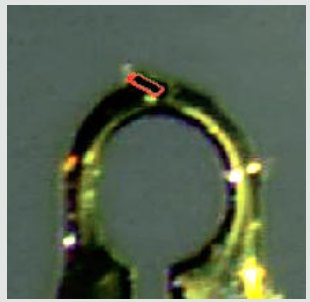
Application Note SC-XRD 502

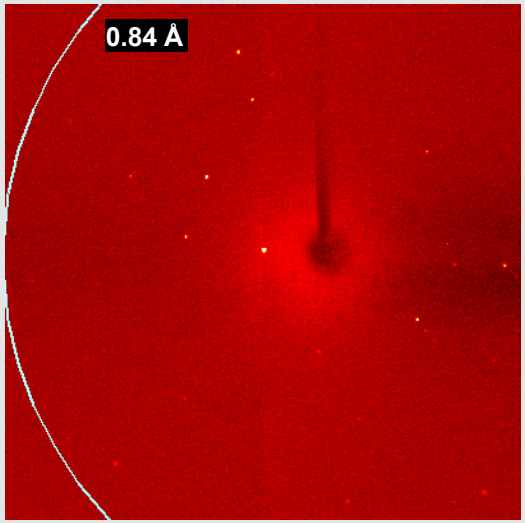
Structural Determination of a Two Micron-sized MOF Crystal

Metal-Organic Frameworks (MOFs) are structures of great industrial and academic interest as they can be at the genesis of a series of new materials with highly attractive properties. Nowadays MOFs can be prepared very fast, under mild conditions in high yields while still combining several functions, taking advantage of the metallic centers, the organic ligands and the framework itself. Some MOFs can even outperform known materials in common industrial applications. $[La(H_4bmt)(H_5bmt)(H_2O)_2]\cdot 3H_2O$ (**1**) has outstanding catalytic activity for the methanolysis of styrene oxide in comparison to that of related MOF-type heterogeneous catalysts. In addition, the inclusion of small amounts of other lanthanides can produce highly photoluminescent materials. [1] **1** can be quickly prepared in 5 minutes at 330 K using microwave-assisted synthesis from the tridentate (benzene-1,3,5-triyltris(methylene))triphosphonic acid (H_6bmt) and $LaCl_3\cdot 7H_2O$. Nevertheless, to fully understand microscope. The D8 VENTURE consists of a KAPPA goniometer and PHOTON 100 CMOS APS detector, equipped with a IμS microfocus X-ray source providing Mo-Kα radiation (λ = 0.71073 Å). During the entire experiment the sample was kept at 100 K using a KRYOFLEX II low temperature device, controlled with the APEX2 software package.

The experiment

Based on a number of initial scans the unit cell determination routine of the software suite indicated a monoclinic unit cell. From the same scans 0.5 degree frames were suggested by the data collection strategy optimizer and the refined data collection strategy consisted of only three scans. Due to the weak diffraction of this very tiny crystal, an appropriate frame exposure time of 120 s was selected. A total of 645 frames were collected within just 21.5 hours.





Typical diffraction pattern ($P2_1/m$, a = 8.9215(10) Å, b = 21.209(2) Å, c = 9.1052(10) Å, β = 107.923(3)°, Z = 2)

F. A. Almeida Paz , H. Ott *et. al.*, *Chem. Commun*. 2013, *49*, 6400 – 6402

How Small? – If You Don't Get Data w/ Mo-IμS…

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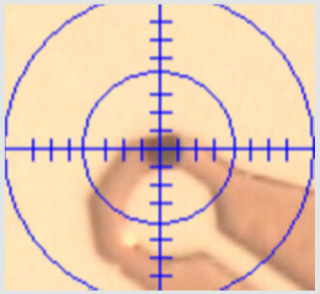
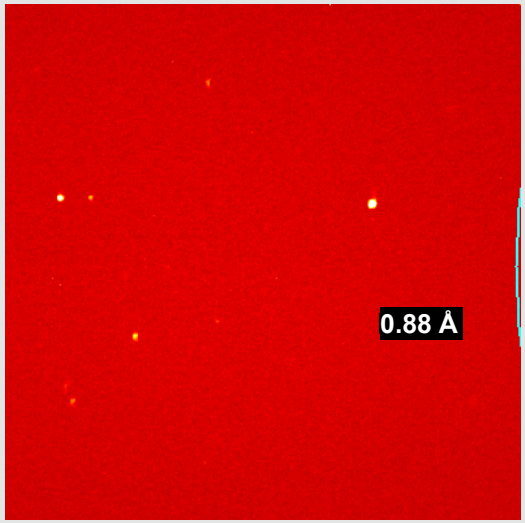
Tiny Crystal of a MOF Compound:

No Diffraction w/ Mo-IμS, but w/ Cu-IμS MX

Size [mm³]	0.03 x 0.02 x 0.01
Source	Cu-IμS MX
Power [W]	30
Exposure time	60 – 120 s/0.5°
Resolution [Å]	**0.88** (0.98 – 0.88)
<I/σ>	16.2 (3.4)
*R*1, *wR*2 [%]	5.3, 13.6

$$[Zn_4(C_{40}H_{24}N_8)_4] \cdot x\, C_3H_7NO \cdot y\, H_2O$$





Typical diffraction pattern with Cu-IμS MX ($P4_2/n$, 120 s/0.5°, APEX II DUO)

R. W. Seidel *et al.*, *Acta Crystallogr.* 2011, *E* 67, m236 – m237

Many More Applications: High-Throughput Fragment Screening

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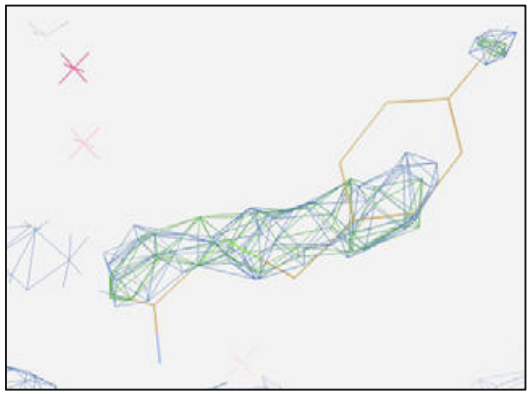
How much data is needed for finding the ligand?

Influence of completeness on the quality of the electron density map around the small molecule fragment ($C_8H_9N_2SCl$)

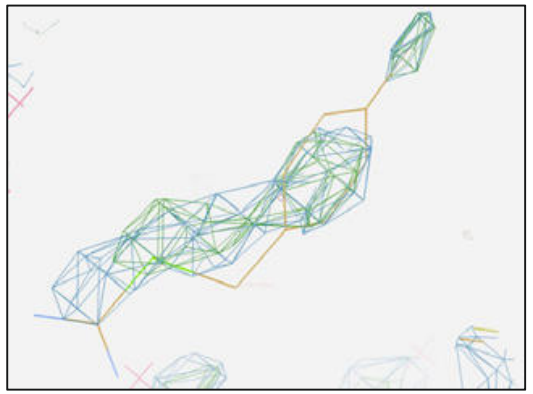
1 h Data set

60% completeness,
Multiplicity 0.9, < I/σ > 10.1



1.3 h Data set

80% completeness,
Multiplicity 1.4, < I/σ > 13.0



3.5 h Data set

96% completeness,
Multiplicity 1.8, < I/σ > 18.0

Electron density maps of high quality within a short period of time

Many More Applications: High-Throughput Fragment Screening

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How much data is needed for finding the ligand?

Influence of completen … density map around the small molecule fragme…

1 h Data set

60% completeness,
Multiplicity 0.9, < I/σ > 10.1

Application Note 6

Fragment Screening Using a Twinned Protein Crystal

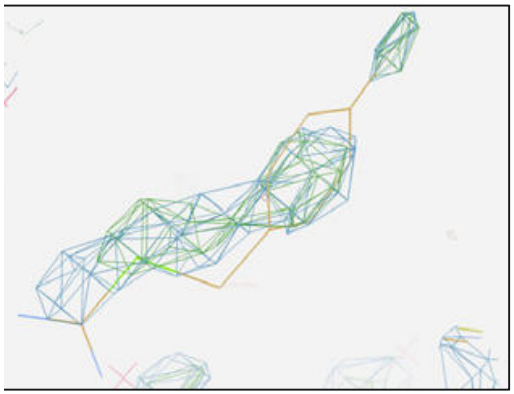
Introduction

Fragment-based screening using X-ray crystallography has become one of the major techniques in contemporary drug design. The aim of fragment screening is to run a rapid data collection of a protein crystal in complex with a small fragment molecule, in order to find out whether the fragment has bound to an active site of the protein or not. The increasing popularity benefits from recent developments of highly brilliant X-ray sources as well as fast and sensitive area detectors. This allows high quality data to be collected within a reasonable period of time using a large number of different fragment chemotypes. This is especially beneficial if one wants to screen a fragment library.

Here, we present results that were obtained by collecting data from a twinned endothiapepsin crystal in complex with a small fragment molecule. The influence of the data completeness on the quality of the obtained electron density maps was analyzed, in order to find the minimum data collection time that is needed to detect the inhibitor in the active site.

resolution of 2.6 Å. The data was processed to 60%, 80% and 96% completeness, resulting in data sets with total collection times of 1 h, 1 h 20 min and 3 h 30 min. The electron density map around the active site was inspected to see whether the fragment was bound. The data collection was performed using a Bruker AXS X8 PROSPECTOR equipped with an Incoatec Microfocus Source IμS with QUAZAR MX optics and an APEX II detector (Fig. 1). Data collection and processing were performed with the PROTEUM2 suite [1].

Figure 1: Diffraction pattern of the endothiapepsin crystal (left; 1.5 x/0.5°). X8 PROSPECTOR with a Cu-IμS MX microfocus X-ray source equipped with a QUAZAR MX optics and APEXII CCD detector (right).



3.5 h Data set

96% completeness,
Multiplicity 1.8, < I/σ > 18.0

Electron density maps of high quality within a short period of time

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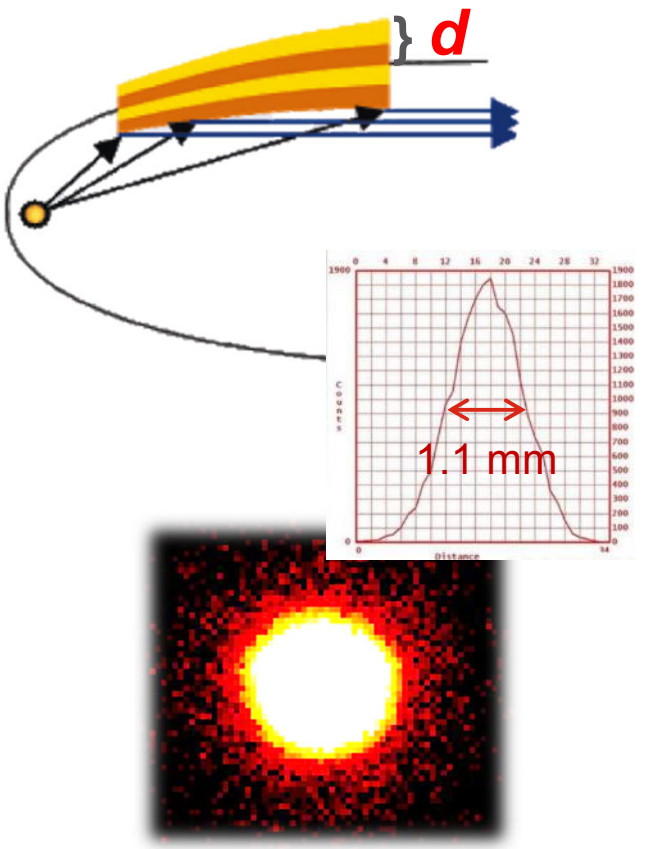
Characteristics of the IμS for XRD and SAXS

- **Parallel Beam Multilayer Optics**
  - High-resolution XRD and SAXS
  - Typical energies: Cu, Co, Mo, Ag
  - Typical FWHM at the sample ~ 1 – 2 mm
  - Typical divergence: ≤ 1 mrad

- **Focusing Multilayer Optics**
  - Spatially resolved XRD
  - Typical energies: Cu, Cr, Mo, Ag



SAXS: Total Flux Comparison

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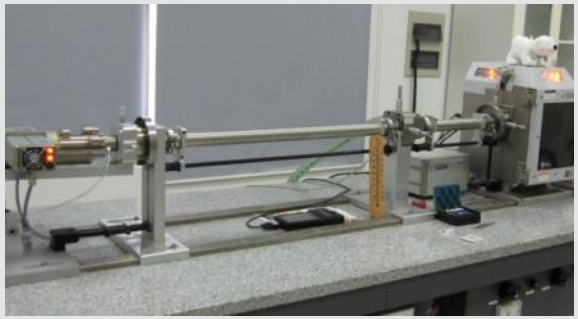
Raw Flux Comparison on a Bruker NANOSTAR

- IμS (30 W, Standard Set-up): 1.5×10^7 cps

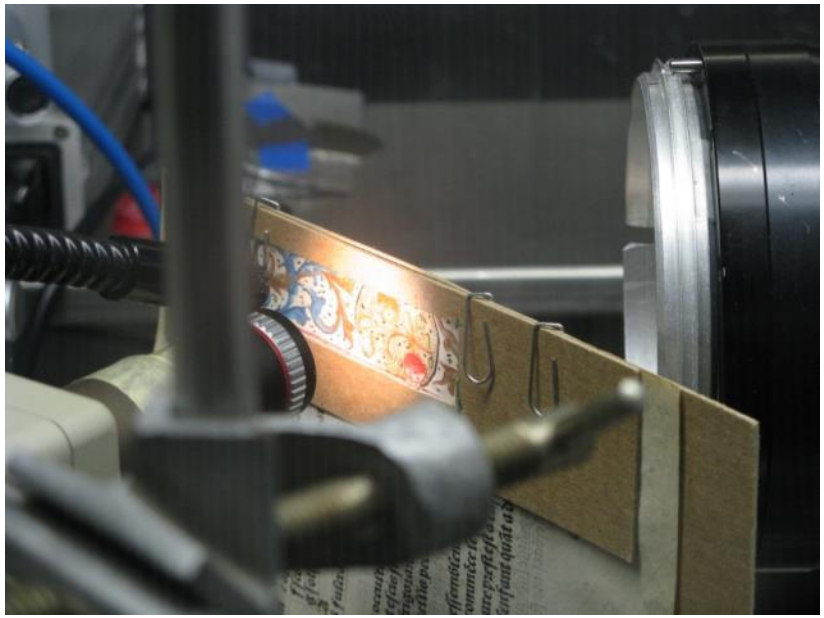
Relative Intensities (Sources w/ ML Mirrors)

- ST + cc-Göbel mirrors (1.4 kW, FF): 1
- IμS (30 W, 0.04 x 0.20 mm^2): 7
- IμS HB (50 W, 0.04 x 0.40 mm^2) 8
- RAG (4.0 kW, 0.3 x 3 mm^2): 8
- μ-RAG (1.2 kW, 0.1 x 1 mm^2): 15
- METALJET (200 W, 0.02 x 0.08 mm^2): > 100
- METALJET with scatter-free pin-holes: > 250

Note that same parallel beam mirror type and resolution settings were used for all data from microfocus X-ray sources.



Measurement of a Manuscript



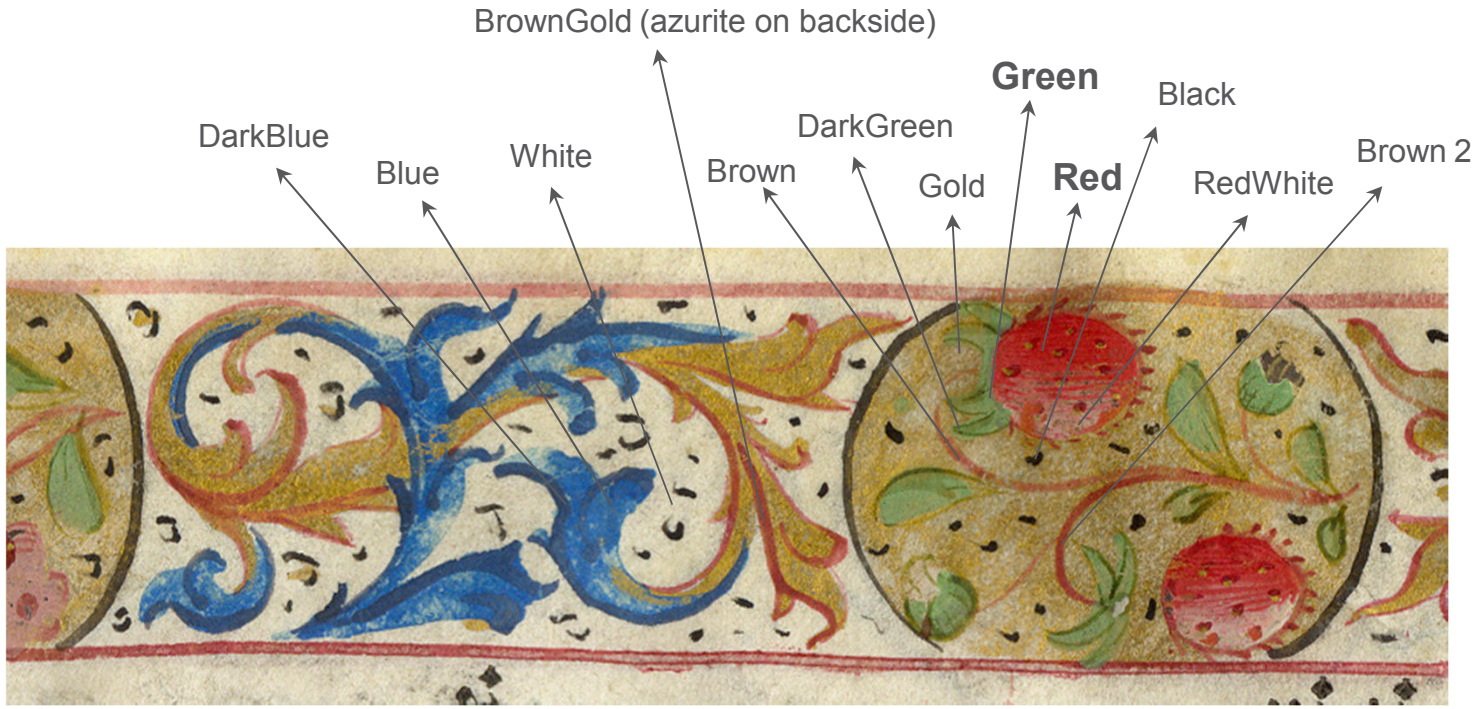
Simultaneous XRD and XRF measurements

Position sensitive measurements using focusing Mo-IµS

- Resolution 150 µm

K. Janssens, F. Vanmeert, Antwerpen

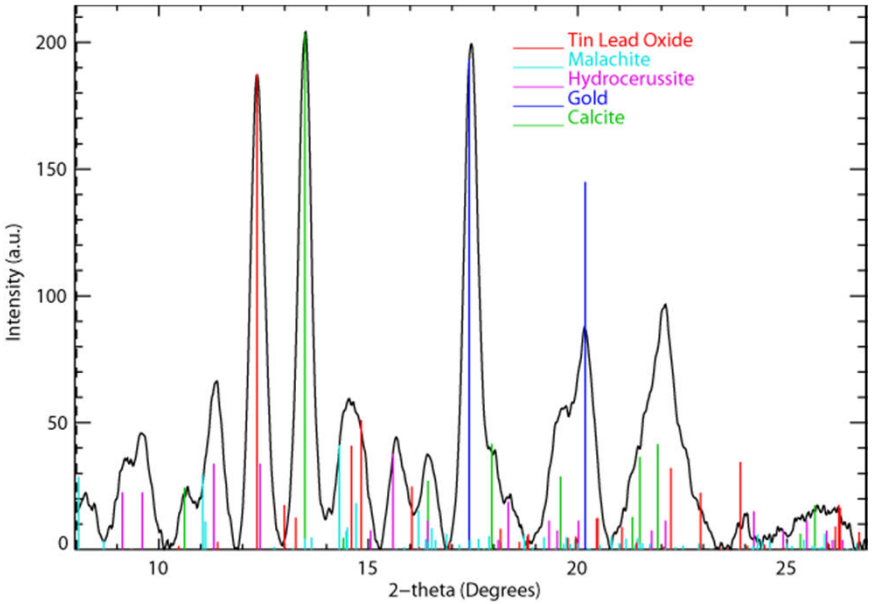
Illuminated Manuscript
Point Measurements



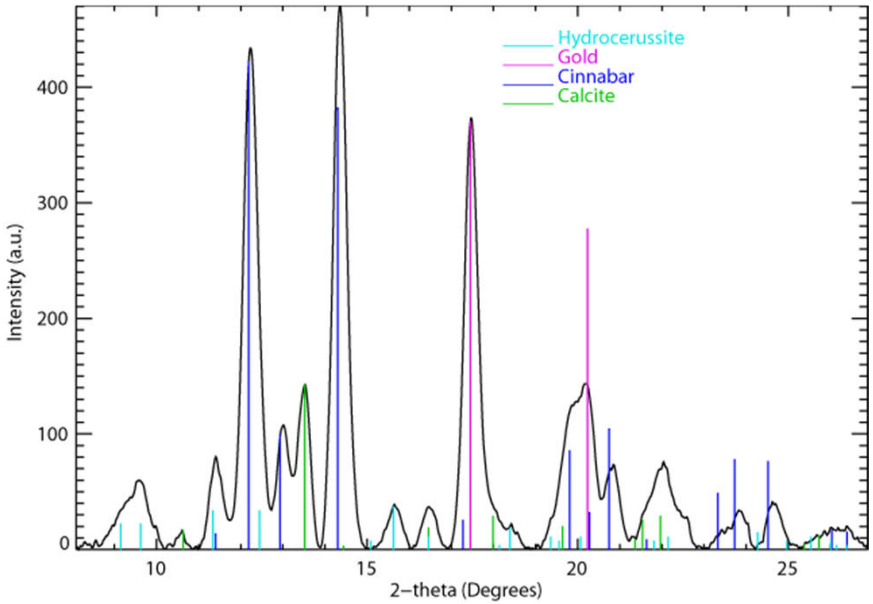
K. Janssens, F. Vanmeert, Antwerpen

Results

Green



Red



- Mo-IμS: 50 kV, 600 μA, 30 sec exposure time
- Scanning Micro diffraction (combined with XRF):

  4 x 4.5 mm², resolution 150 μm, Total measurement time: 18 h
- Measurements and data evaluation by Frederick Vanmeert

K. Janssens, F. Vanmeert, Antwerpen

Results

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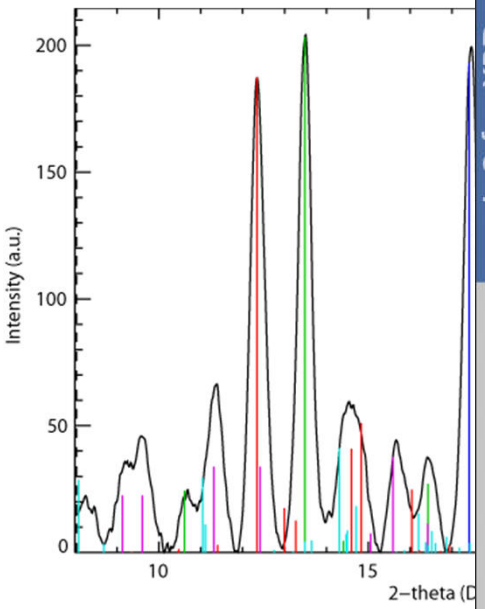
Gree



- Mo-IμS: 50 kV, 60
- Scanning Micro di
  4 x 4.5 mm²
- Measurements an

K. Janssens, F. Vanmeert

IμS for XRD

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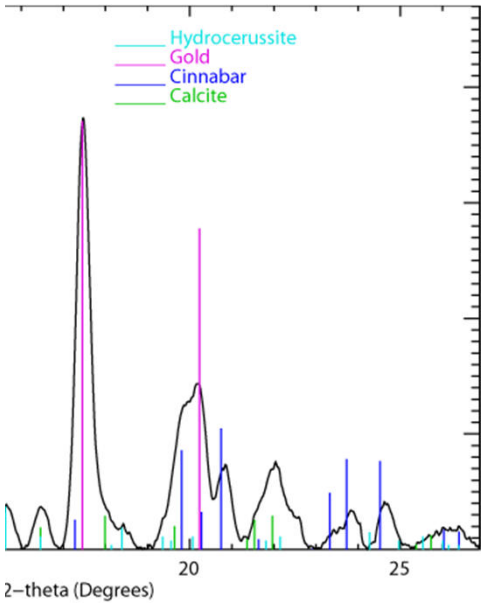
The Incoatec Microfocus Source IμS for XRD-Applications

Microdiffraction using an IμS

Frederik Vanmeert, University of Antwerp has examined De Heem´s painting 'Flowers and Insects', Royal Museum of Fine Arts Antwerp with the Incoatec Microfocus Source IμS high brilliance.

In recent years the interest in the non-destructive investigation of cultural heritage objects has risen strongly. Besides infrared and optical imaging and spectroscopic methods, X-ray methods like X-ray fluorescense, X-ray diffraction, X-ray radiography and X-ray imaging methods (e. g. tomography) are often used for the analysis of these artifacts [1].
Using for example X-ray fluorescence (XRF) the scrolls of Qumran were investigated [2], fragments could be identified as belonging together or not [3]. Using XRF analysis of paintings generally provides information about the possible presence of elements on the surface of art-objects due to pollution (e.g. sulphur or chlorine), about the elements and pigments used by the artist, about previous restored areas detected by the presence of "modern" elements like titanium or zinc, and identification of fraudulent submission. In combination with X-ray diffraction (XRD) also the crystallographic composition of the used pigments could be characterised. A study about the alteration involved the oxidation of cadmium yellow (CdS) to $CdSO_4 \cdot 2H_2O$ under the influence of light, oxygen and moisture is an example of the use of this technique in the investigation of paintings [1]. Other X-ray methods are used as well. A painting by Rembrandt van Rijn was analysed using an X-ray absorption imaging technique at ESRF and with XRF revealing an overpainting [4].

Red



ne: 18 h

IμS (Cr-Kα) with focusing optics for Residual Stress measurements

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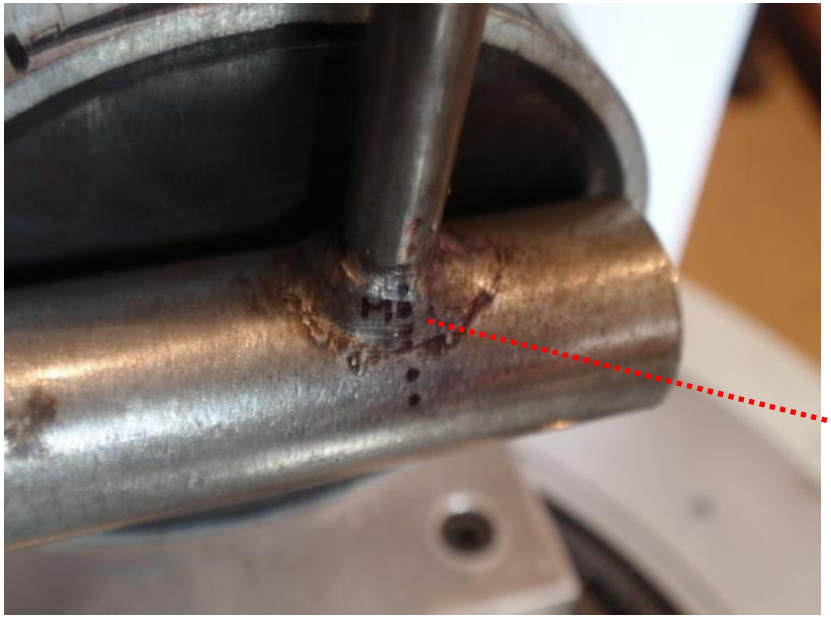
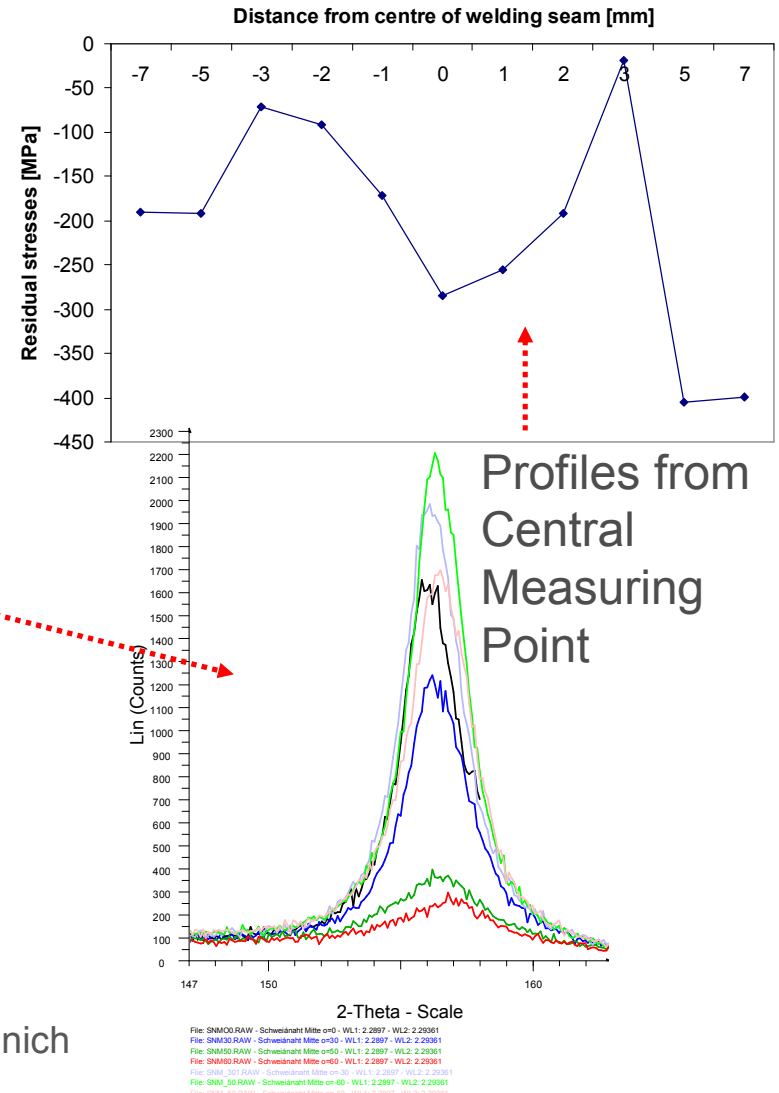
Profile across a 6 mm weld seam



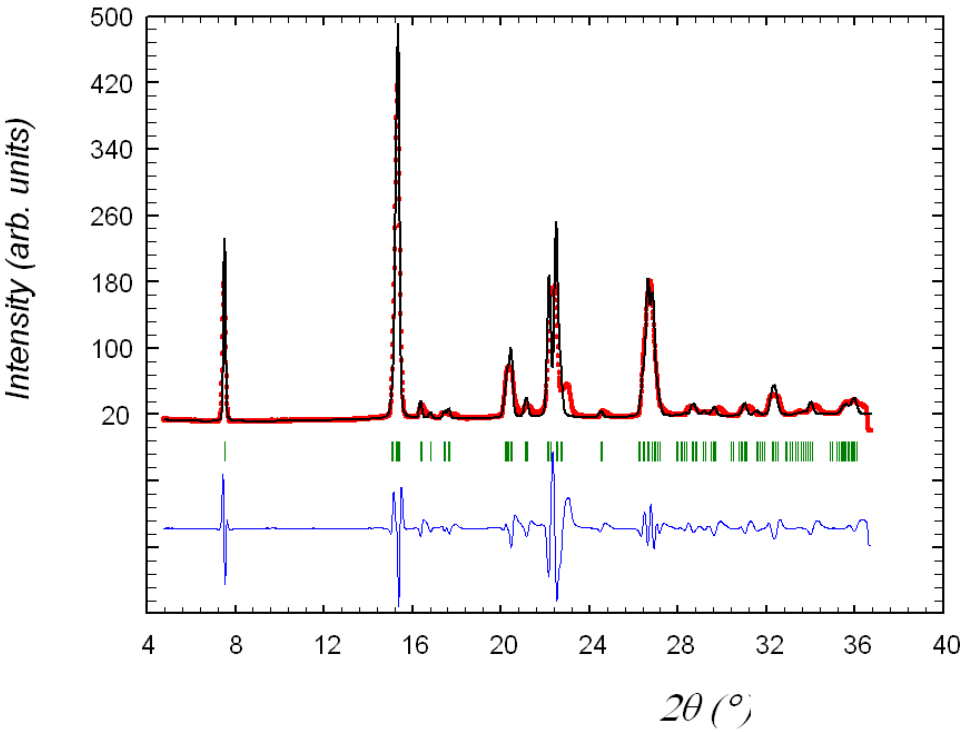
Measuring Steps 1mm



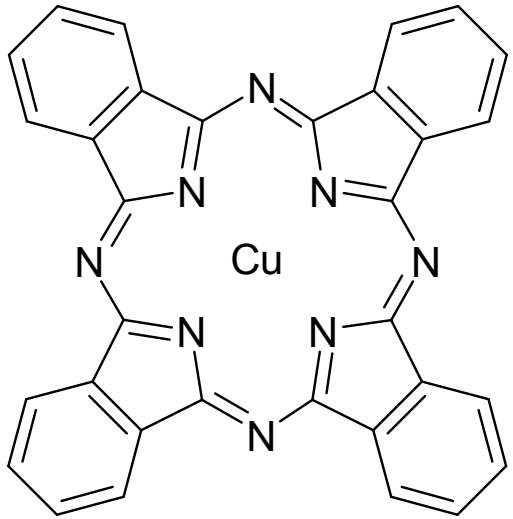
H. and U. Göbel, LabXA and M. Schuster, Siemens, Munich

ASS with Cu-Kα

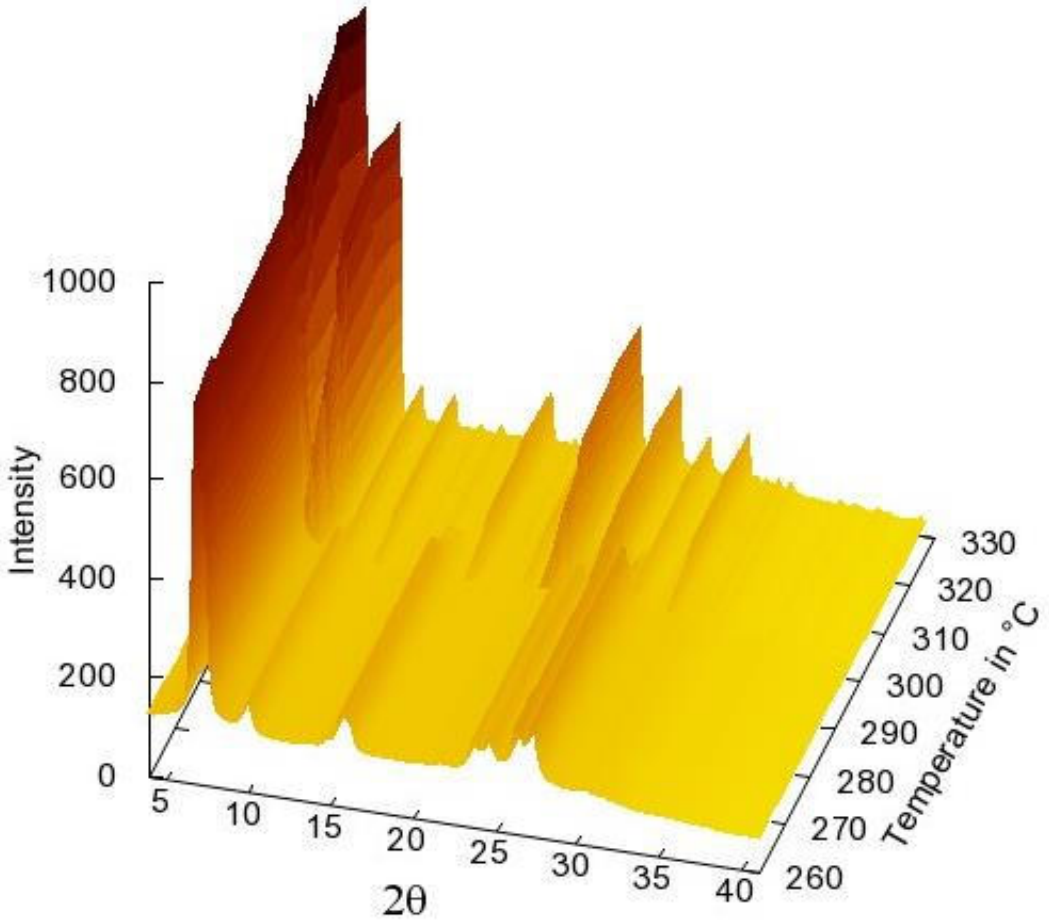
Whole tablet
Exposure time: 300 s
Focusing onto the detector



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



R.E. Dinnebier, M. Müller, MPI for Solid State Research, Stuttgart

Wrap Up

INCOATEC
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- **IμS**
  - **Air-cooled low-power high-brightness microfocus X-ray source**
  - **Intensity higher than conventional RAG, comparable to low-power μ-RAG**
  - **Different mirror designs allow for a wide range of applications**
  - Available for Cu-Kα, Mo-Kα, Ag-Kα and also for Cr-Kα, Co-Kα, …
  - **Upgrade for existing diffractometers, optinal with its own enlosure IXE**
  - **> 700 installations world wide**

- **NEW IμS 3.0**
  - **First and only microfocus X-ray sealed tube optimized for X-ray diffraction**
  - **New tube, new beampath concept, new optics**
  - **30 - 40% more intensity compared to the IµS HB**
  - **No compromises on life-time** and **ease-of-use**
  - Available for Cu-Kα, Mo-Kα and Ag-Kα
  - Available as upgrade of existing equipment
  - **Replacement for low power μ-RAG's**

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Thank You!

Incoatec - Your Partner for X-Ray Optics and Microfocus Sources

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