High-Brilliance Micro-Focus X-ray Sources for Diffractometry

T. Samtleben, J. Wiesmann, B. Hasse, J. Graf, C. Michaelsen - Incoatec GmbH, Geesthacht, Germany

Göbel mirrors
development
Multilayer X-ray Optics
10 years of innovations
made in Germany
IμS IμS High Brilliance
Monte Optics Microfocus Sources

Incoatec Microfocus Source IμS and 2D Montel Optics

The IμS is the perfect upgrade for equipment in X-ray diffractometry. It combines the advantages of a sealed-tube system with the superior data quality of conventional rotating anode systems. The IμS is also included in the new equipment of Bruker AXS: for SAXS in the NANOSTAR and for SC-XRD in the D8 QUEST and VENTURE solutions for crystallography. The IμS consists of a microfocus source and a specially designed multilayer optics named Quazar.

The source

- high brilliance & best spectral purity
- Cu, Mo, Cr and Ag radiation available
- air cooled
- < 40 μm spot size at the anode
- convenience of sealed-tube
- high voltage generator

3-Year warranty

Quazar Optics

- 2-dim beam shaping
- collimating, focussing or collimating + focusing (hybrid)
- patented housing for high stability and easy alignment
- motorized alignment (optional)

Optics	Divergence (mrad)	Beam size (mm)	Flux density (ph/s mm²)
Cu-MX	7.6	0.11	> 18 x 10⁹
Mo	4.9	0.11	> 1.9 x 10⁹
Ag	4.9	0.095	> 0.9 x 10⁹

Different types of Quazar Optics for the IμS$^{High\ Brilliance}$

Optics in high-stability housing

NEW IμS$^{High\ Brilliance}$

- up to 60 % more flux due to improved optical design
- for Cu, Mo & Ag
- component recognition
- improved safety features
- compliant with Machinery Directive 2006/42/EC

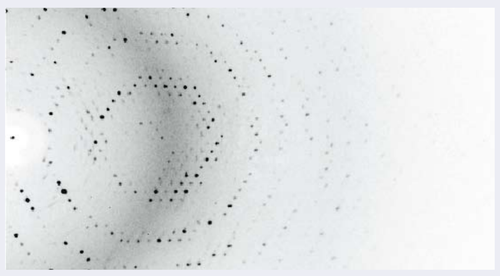
IμS integrated in a new Bruker D8 DISCOVER (left) and component recognition (right)

New IμS$^{High\ Brilliance}$ System with 19" wide stand-alone generator

IμS for Cu Radiation: SAD Phasing on Glucose Isomerase (Quazar Optics)

Sample: glucose isomerase
a = 93.88 Å, b = 99.68 Å, c = 102.90 Å; I222; T = 100 K;
388 amino acids / ASU

crystal size	0.24x0.24x0.15 mm³	<redundancy>	14.4 (4.5)
exposure time	40 s / 0.5°	<completeness>	98.0 % (89.8 %)
total time	~ 43 h	R_{int}	0.0652 (0.4191)
resolution	35-1.50Å (1.60-1.50Å)	$R_{p.i.m.}$	0.0149 (0.1959)
< I/σ >	21.8 (3.5)	R1(I > 2σ(I) ; all)	0.1587; 0.2047
anom. signal limit	2.7 Å (Ca^{2+}, 9 S)		

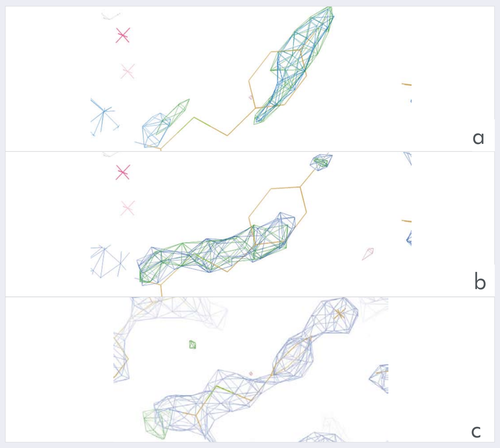


Data statistics and a typical diffraction pattern of glucose isomerase. Ca^{2+} site has been found in the initial phasing with SHELXD; measured with a Bruker Smart6000 diffractometer equipped with the IμS (Quazar optics).

Fragment Screening (Quazar MX Optics)

Sample: Twinned crystal of an enzyme in complex with a small molecule fragment

crystal size	0.45 x 0.07 x 0.04 mm³	<completeness>	95.9 % (85.8 %)
exposure time	15 s / 0.5°	R_{int}	0.0679 (0.1052)
total time	3.5 h	R_{σ}	0.0642 (0.1936)
resolution	41–2.59Å (2.69–2.59Å)	R_1	0.1331
< I/σ >	18.0 (5.7)		

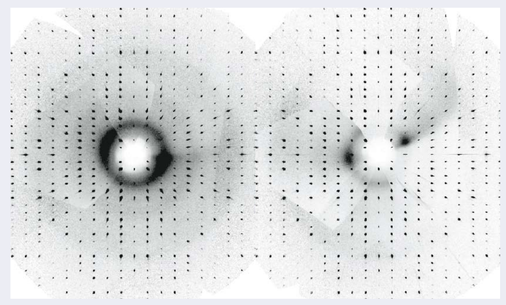


Electron density map (Fo-Fc, 1.5σ in blue, 2Fo-mFc 3σ in green) of the ligand after rigid body refinement with 60 % completeness (a, after 1 h), 80 % completeness (b, after 1.3 h) and 96 % data completeness (c, after 3.5 h and SHELXL refinement). (data from A. Heine, Philipps University Marburg, Germany)

IμS for Mo Radiation: Perfect for Small Crystals; Good for Big Ones

Source	IμS	Sealed Tube	IμS	Sealed Tube
Size [mm³]	0.10 x 0.05 x 0.05		0.30 x 0.25 x 0.15	
Power [kW]	0.03	2.0	0.03	2.0
Exposure time [s/°]	100	300	33	50
<I>#	139	19	4763	1286
<I/σ>	15.3; 3.5*	10.7; 2.1*	37.6; 21.1*	30.4; 13.6*
R_{int}	0.039	0.062	0.021	0.023
R1 (all); wR2 (all)	0.039; 0.092	0.056; 0.105	0.030; 0.082	0.031; 0.082

*Details of four sets of data collected on $C_{24}H_{21}N_3O_8$ (# normalized to 1°/min; * values for highest resolution shell 0.85 - 0.75 Å) (data from T. Schulz, D. Stalke, University Göttingen, Germany)*

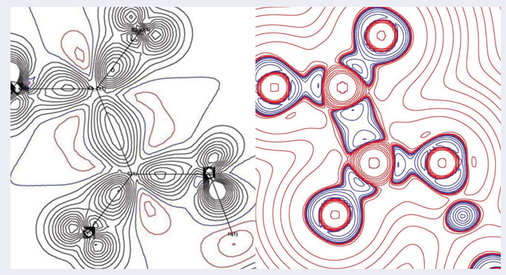


Calculated precession plots of the hk0 layer for an organic sample: Mo-Sealed Tube (67 s/°, left), Mo-IμS (16 s/°, right)

Challenging Rotating Anodes

Source	IμS	RAG	IμS	RAG
Size [mm³]	0.02 x 0.04 x 0.04		d=0.18 (sphere)	
Power [kW]	0.03	4.0	0.02	4.0
Exposure time [s/°]	150	150	20	20
<I>	17.1	5.9	344.1	537.9
<σ>	1.6	1.1	5.1	6.8
R1 (all); wR2 (all)	0.084; 0.240	0.090; 0.241	0.036; 0.082	0.041; 0.082

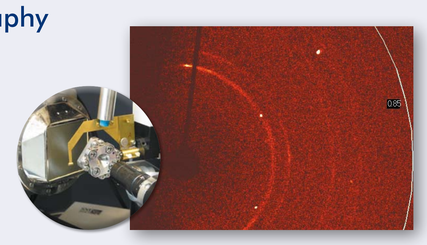
Details for sets of data recorded on a smaller quartz crystal SiO_2 and a larger crystal of ammonium-bitartrate $C_4H_9O_6N$. (data from C.W. Lehmann, MPI Mühlheim, Germany)



Charge density study on oxalic acid. Multipole refinement : No. of unique 3784; Refinement incl. 2450;R(F) 0,036; N(ref)/N(var) 23.1; H-Atoms constrained.

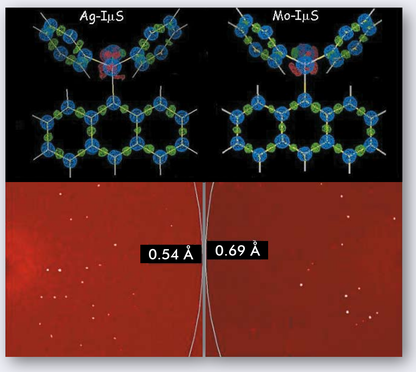
IμS for Ag Radiation: Best for High-Pressure Crystallography

The small beam cross-section of the IμS for Mo and Ag radiation reveals what an interesting alternative they are to sealed tube sources for diffraction studies on single crystals and powders using diamond anvil cells for high-pressure crystallography. The focused beam reduces part of the background caused by scattering at the gasket, thus improving the signal-to-noise ratio.



Highly Absorbing Crystals

In Ag radiation the IμS delivers a peak flux density of about 0.9 x 10^9 photons/(s mm²) in a 90 μm beam (FWHM), which is ideal for small crystals, especially of absorbing materials. The advantage of such short wavelengths is the reduced absorption and extinction, as well as the „compressed" reciprocal space thus gaining access to a larger range of *d*-spacings at a fixed 2θ setting.
The figure shows the comparison of two single crystal diffraction experiments with the Ag-IμS and the Mo-IμS on an organic sample.



Residual density and the resolution limit (at 2θ = 31°; DX = 51 mm) for $C_{26}H_{19}PS$: Ag-IμS (200 s/°, left), Mo-IμS (77 s/°, right) (data from F.P.A. Fabbiani, University Göttingen, Germany)

IμS for Small Angle X-ray Scattering

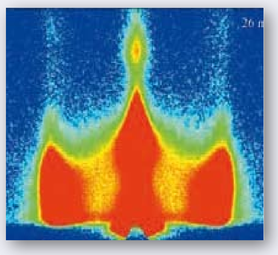
The IμS was integrated in the Bruker Nanostar, an equipment for SAXS and simultaneous WAXS measurements with a 3-pinhole geometry. The beam was collimated using a Quazar optics with a low divergence of 1mrad or smaller.

in-situ GISAXS with Liquid Samples

For rapid GISAXS measurements of liquid samples our IμS was combined with a Dectris Pilatus detector. The set-up can be changed from liquids to capillary samples within <30 min, alignment included (picture and data courtesy of P. Šiffalovič, Slovak Academy of Science). The measurement shows an ordering of the particles. It was possible to study the evolution of the ordering with increasing surface pressure in-situ.

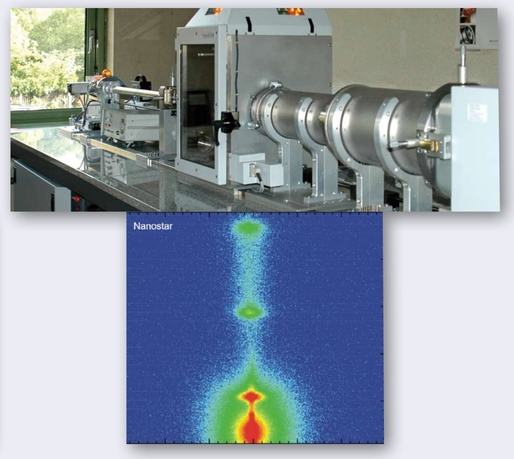
Sample: Langmuir film with silver nanoparticles

- the surface was pressed with 26mN/m
- 180 s measurement time, aperture 350 μm



GISAXS

With the IμS GISAXS measurements of high data quality can also be carried out in the lab. The figure shows the results of an ion-beam sputtered Mo/Si multilayer. The total reflection and three Bragg sheets are clearly visible.



www.incoatec.de

INCOATEC
innovative coating technologies