Bright.Brighter.Unique.
High-Brightness Microfocus Sources for Chemical Crystallography

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Since its introduction in 2006, the Incoatec Microfocus Source IμS has become the gold standard for home-lab X-ray sources. The IμS combines a low power microfocus X-ray sealed tube with dedicated Montel multilayer mirrors and delivers intensities beyond those of traditional rotating anode sources. With more than 650 sources sold world-wide, the IμS is the market-leading microfocus source for X-ray diffraction applications, such as single crystal diffraction on small molecule and protein crystals as well as small angle scattering. The latest generation of the IμS, the IμS 3.0, is the first microfocus X-ray source that is designed for X-ray diffraction resulting in a gain in intensity of about 30%. Here, we present selected examples for applications of the IμS in single crystal diffraction.

IμS for Cu-K_α Radiation

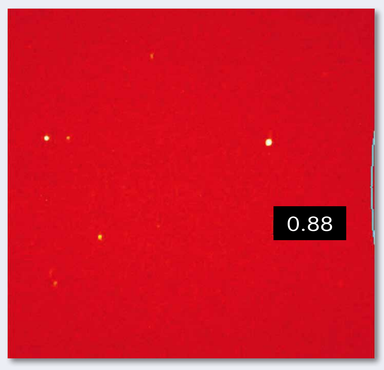
Due to the strong interaction of the Cu radiation with soft matter samples, the IμS for Cu radiation is the ideal home-lab source for most challenging and poorly diffracting organic samples and macromolecular crystallography delivering more than 2 x 10^{10} ph/s/mm^2. It also provides a sufficiently large anomalous signal for both absolute structure determination from chiral organic samples and SAD phasing from proteins.

The figure below shows an example of a tiny MOF crystal. MOF's are known to crystallize as small and poorly diffracting crystals. A tiny specimen of 10 x 20 x 30 μm^3 was studied on a Bruker D8 equipped with a Mo-IμS and a Cu-IμS. Very weak diffraction was observed even on a Mo-IμS. A mouse click later, however, good quality data could be collected by switching to the Cu-IμS. The softer radiation in combination with long exposure times provided the key to solve the structure of this tiny MOF crystal, which otherwise would likely have required synchrotron intensity. The example below shows results from data collection on a tiny MOF crystal.

Structure determination from a tiny crystal of a MOF compound

$a = b = 23.6710(7)$ Å, $c = 14.9878(5)$ Å, $Z = 8$, $P4_2/n$, $[Zn(C_{40}H_{24}N_8)] \cdot x\, C_3H_7NO \cdot y\, H_2O$

Size [mm^3]	0.03 x 0.02 x 0.01
Source	Cu-IμS MX
Power [W]	30
Exposure time [s/0.5°]	60 - 120
Resolution [Å]	0.88 (0.98 - 0.88)
$\langle I/\sigma \rangle$	16.2 (3.4)
*R*1, *wR*2 [%]	5.3, 13.6



Typical diffraction pattern with Cu-IμS MX

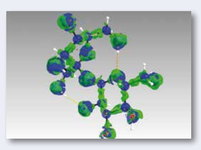
IμS for Mo-K_α Radiation

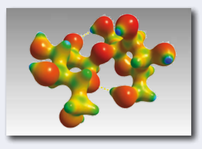
Data collections to a resolution beyond 0.8 Å, which is the typical resolution limit of Cu sources, require the use of sources with a shorter wavelength. The Mo-IμS delivers more than 2 x 10^9 ph/s/mm^2 and enables the collection of high quality data beyond 0.40 Å within a reasonable amount of time. This allows not only a more accurate modelling of the electron density by using aspherical scattering factors, but also enables a reliable determination of the absolute structure, despite the significantly lower anomalous signal obtained with Mo-K_α radiation. The figure below shows the electron density distribution and the data statistics after a multipolar refinement of a sucrose crystals.

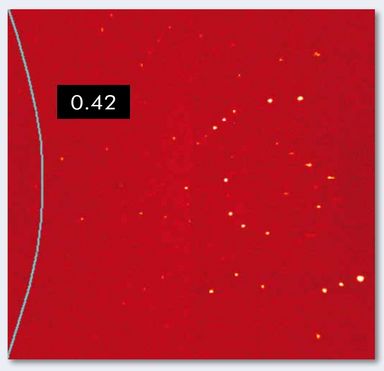
Charge density and absolute structure determination of a sucrose crystal after multipole refinement

$a = 7.7189(2)$ Å, $b = 8.6628(2)$ Å, $c = 10.8085(3)$ Å, $\beta = 102.999(1)°$, $Z = 2$, $P2_1$, $C_{12}H_{22}O_{11}$

Size [mm^3]	0.25 x 0.22 x 0.18
Source	Mo-IμS$^{\text{High Brilliance}}$
Total time [d]	3
Exposure time [s/°]	6 - 100
Resolution [Å]	0.41 (0.51 - 0.41)
Multiplicity	7.7 (5.1)
$\langle I/\sigma \rangle$	47.2 (18.3)
*R*1, *wR*2 [%] (IAM) *R*1, *wR*2 [%] (Multipole)	2.47, 6.63 1.48, 2.28
Flack *x(u)* (IAM) Flack *x(u)* (Multipole) Parsons *z(w)*	0.00(15) 0.02(5) 0.03(4)







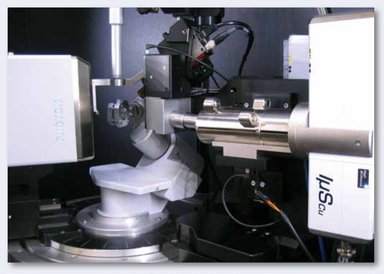
Typical diffraction pattern of the sucrose crystal, recorded with Mo-IμS HB (right), electron density and residual electron density (after IAM refinement,left, above) and electrostatic potential (left, below) of sucrose.

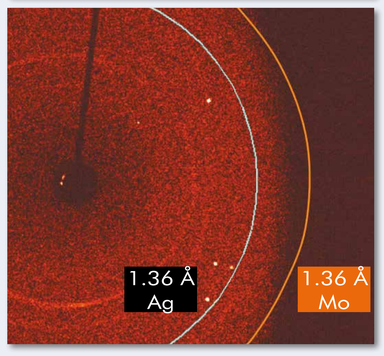
Sucrose ($\langle |\Delta F^2_{ano}| \rangle / \langle F^2 \rangle$ = 0.06% (Mo), 0.30% (Cu))

IμS for Ag-K_α Radiation

Ag-K radiation is the optimum X-ray wavelength for studying absorbing materials and for high pressure experiments in the home lab. The reduced absorption and extinction minimizes the bias of the structure model by systematic errors. High-pressure X-ray diffraction experiments using a diamond anvil cell (DAC) benefit from the compression of the q-space.

As the area of reciprocal space accessible during data collection is primarily restricted by the geometry of the DAC, a larger portion of the reciprocal space is accessible when Ag radiation is used instead of Mo radiation. Thus, using a Ag-IμS for high-pressure experiments increases the resolution and the number of observations as well as the completeness of the data which facilitates the structure solution and refinement. Further, scattering from the gasket is reduced due to the small beam diameter and the harder radiation. This reduces the background and improves the signal-to-noise ratio.





Diffraction pattern of a gabapentin crystal: Illustration of the gain in resolution and completeness with Ag radiation.

IμS 3.0 - The new Micorofocus X-ray Tube with *IXT*

The latest generation of the IμS, the air-cooled IμS 3.0 source, contains the new Incoatec X-ray Tube *IXT* and is the first microfocus X-ray source that is designed for X-ray diffraction. Numerous small improvements make the IμS 3.0 the most user-friendly, yet most powerful microfocus sealed tube X-ray source ever.

IμS 3.0

Incoatec Microfocus Source

NEW *with IXT*

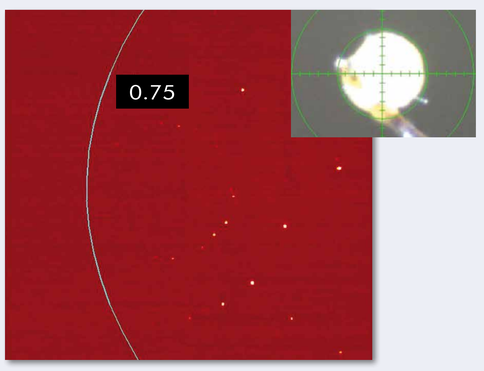
- new X-ray tube *IXT* designed by Incoatec
- first microfocus source optimized for X-ray diffraction
- new beam path concept with true downstream alignment
- He filled optics housing and factory-aligned optics
- swappable optics (Quick-lock)
- improved user-friendliness
- new high performance HV generator
- for Cu, Mo and Ag radiation
- 3 years warranty

30% more intensity

Structure determination on a micro-ylid crystal

$a = 5.9691(3)$ Å, $b = 9.0265(5)$ Å, $c = 18.3725(10)$ Å
$Z = 4$, $P2_12_12_1$, $C_{11}H_{10}O_2S$

Size [mm^3]	0.12 x 0.10 x 0.09	
Source	Mo-IμS$^{\text{High Brilliance}}$	Mo-IμS 3.0
Exposure time [s/°]	4	4
Resolution [Å]	0.80 (0.90 - 0.80)	0.80 (0.90 - 0.80)
Multiplicity	11.9 (10.8)	11.9 (10.8)
$\langle I/\sigma \rangle$	27.0 (5.4)	32.0 (7.6)
*R*1, *wR*2 [%]	3.96, 8.45	3.53, 8.30
Flack *x(u)*	-0.07 (13)	0.03 (12)
Parsons Q*(v)*	-0.02 (4)	0.05 (3)
d(C1-C2)	1.390 (5)	1.392 (4)

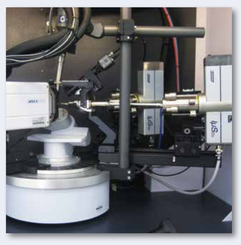


Typical diffraction pattern from a micro-ylid crystal, recorded with a D8 VENTURE and a Mo-IμS 3.0. The 30% gain in intensity leads to an improved fit for the structure refinement and to lower standard deviations for the structural model.

Upgrading Existing Diffractometers with the Microfocus Source IμS

You have a Bruker AXS, Marresearch, Nonius, Rigaku, Huber diffractometer or some other system?
Incoatec supports a full integration of the IμS into all common commercial diffractometers as well as into customized set-ups. Over the last years, we have upgraded more than 60 diffractometers world-wide. An international team of engineers, physicists and chemists with a broad scientific background will find the optimal solution also for your specific application. Contact us and challenge us.

IμS upgrade on a customized setup in Kolkata, India



Bruker APEX II DUO IμS in Düsseldorf, Germany

Replacement of Rigaku RU-200 generator in Boulder, USA

Bruker NANOSTAR (IμS and SCATEX) in Vienna, Austria

Conclusion

The Incoatec Microfocus Source IμS has all the advantages of a sealed tube system and a flux density exceeding that of traditional rotating anode X-ray sources. It is therefore a cost effective and user-friendly X-ray source for diffraction applications in the home lab, bridging the gap between conventional sealed tube sources and high performance microfocus X-ray sources, such as microfocus rotating anodes and liquid metal jet sources.

The new IμS 3.0 is the first microfocus X-ray source that is designed for X-ray diffraction and delivers 30% more intensity than the second generation IμS. This makes the IμS 3.0 the most user-friendly, yet most powerful microfocus sealed tube X-ray source ever.

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