

# High-Brightness Microfocus Sources for Chemical Crystallography

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Since its introduction in 2006, the Incoatec Microfocus Source  $\mu\text{S}$  has become the gold standard for home-lab X-ray sources. The  $\mu\text{S}$  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 600 sources sold world-wide, the  $\mu\text{S}$  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  $\mu\text{S}$ , the  $\mu\text{S}$  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  $\mu\text{S}$  in single crystal diffraction.

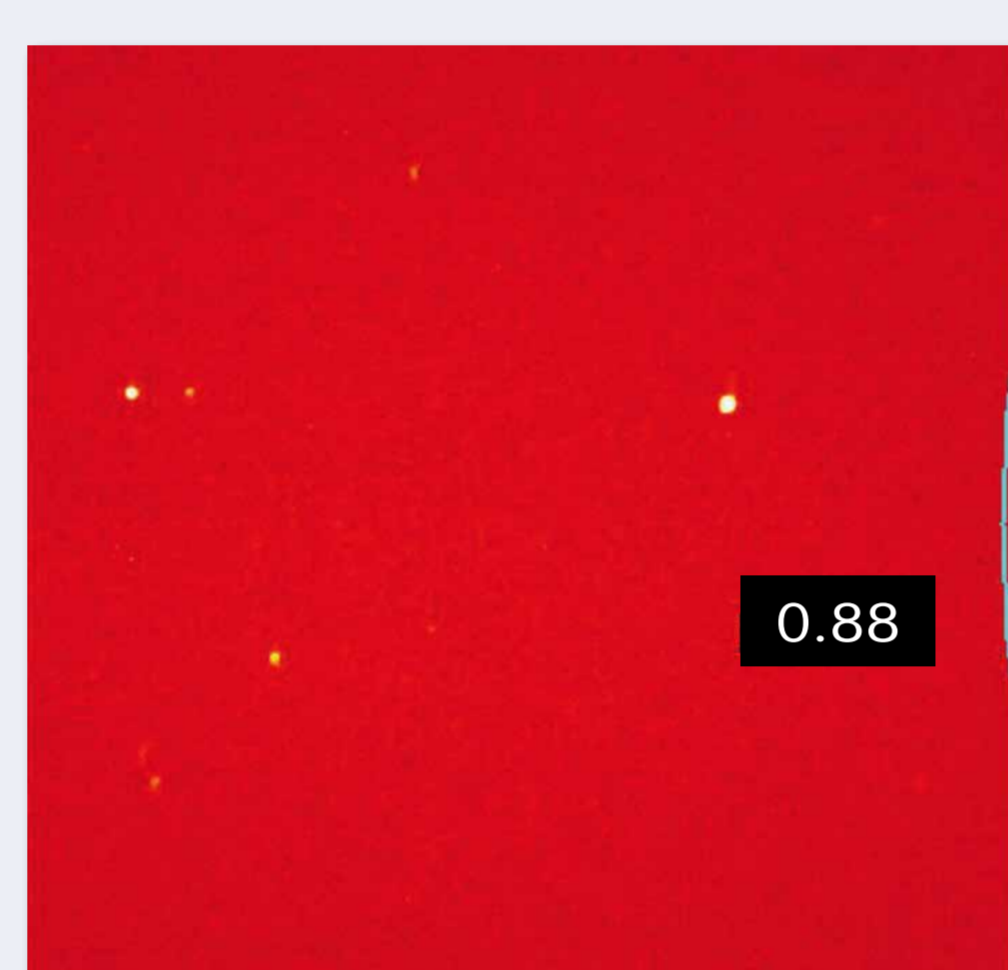
## $\mu\text{S}$ for Cu-K $\alpha$ Radiation

Due to the strong interaction of the Cu radiation with soft matter samples, the  $\mu\text{S}$  for Cu radiation is the ideal home-lab source for most challenging and poorly diffracting organic samples and macromolecular crystallography delivering more than  $2 \times 10^{10}$  ph/s/mm<sup>2</sup>. 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  $10 \times 20 \times 30 \mu\text{m}^3$  was studied on a Bruker D8 equipped with a Mo- $\mu\text{S}$  and a Cu- $\mu\text{S}$ . Very weak diffraction was observed even on a Mo- $\mu\text{S}$ . A mouse click later, however, good quality data could be collected by switching to the Cu- $\mu\text{S}$ . 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

$a = b = 23.6710(7) \text{ \AA}$ ,  $c = 14.9878(5) \text{ \AA}$ ,  $Z = 8$ ,  $P4_2/n$ ,  
 $[\text{Zn}(\text{C}_{40}\text{H}_{24}\text{N}_8)] \cdot x \text{C}_3\text{H}_7\text{NO} \cdot y \text{H}_2\text{O}$

Size [mm <sup>3</sup> ]	0.03 x 0.02 x 0.01
Source	Cu- $\mu\text{S}$ MX
Power [W]	30
Exposure time [s/0.5°]	60 - 120
Resolution [Å]	0.88 (0.98 - 0.88)
$\langle 1/\sigma \rangle$	16.2 (3.4)
R1, wR2 [%]	5.3, 13.6



Typical diffraction pattern with Cu- $\mu\text{S}$  MX

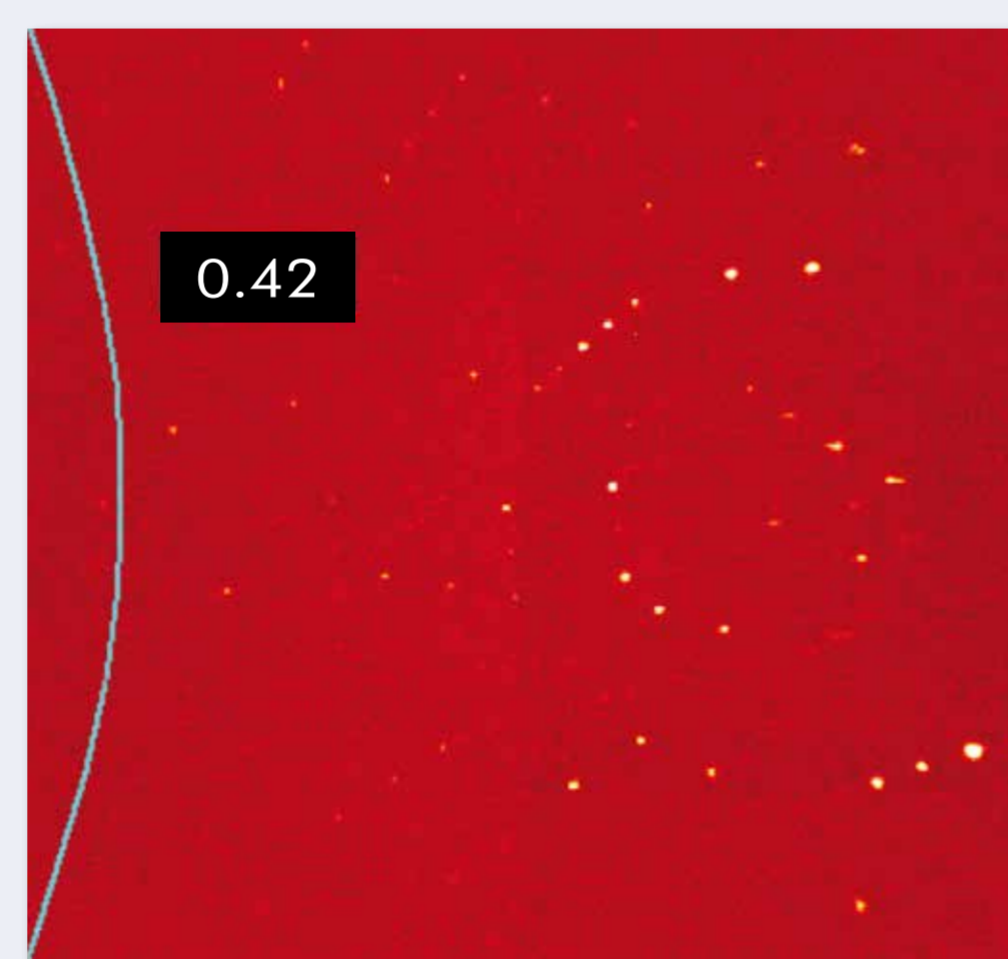
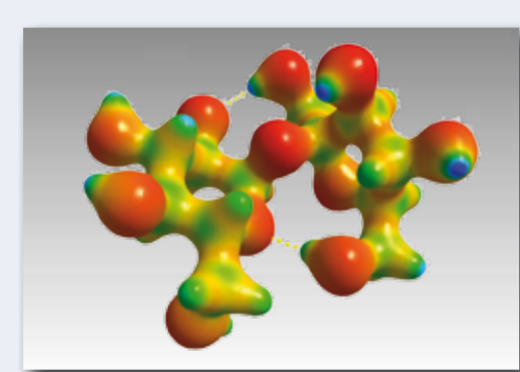
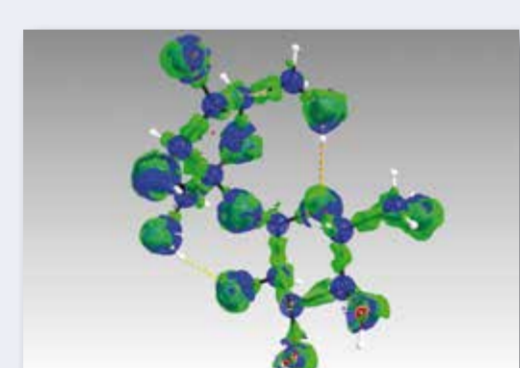
## $\mu\text{S}$ for Mo-K $\alpha$ Radiation

Data collections to a resolution beyond  $0.8 \text{ \AA}$ , which is the typical resolution limit of Cu sources, require the use of sources with a shorter wavelength. The Mo- $\mu\text{S}$  delivers more than  $2 \times 10^9$  ph/s/mm<sup>2</sup> and enables the collection of high quality data beyond  $0.40 \text{ \AA}$  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 $\alpha$  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

$a = 7.7189(2) \text{ \AA}$ ,  $b = 8.6628(2) \text{ \AA}$ ,  $c = 10.8085(3) \text{ \AA}$ ,  
 $\beta = 102.999(1)^\circ$ ,  $Z = 2$ ,  $P2_1$ ,  $\text{C}_{12}\text{H}_{22}\text{O}_{11}$

Size [mm <sup>3</sup> ]	0.25 x 0.22 x 0.18
Source	Mo- $\mu\text{S}$ High Brilliance
Total time [d]	3
Exposure time [s/°]	6 - 100
Resolution [Å]	0.41 (0.51 - 0.41)
Multiplicity	7.7 (5.1)
$\langle 1/\sigma \rangle$	47.2 (18.3)
R1, wR2 [%] (IAM)	2.47, 6.63
R1, wR2 [%] (Multipole)	1.48, 2.28
Flack x(u) (IAM)	0.00(15)
Flack x(u) (Multipole)	0.02(5)
Parsons z(w)	0.03(4)



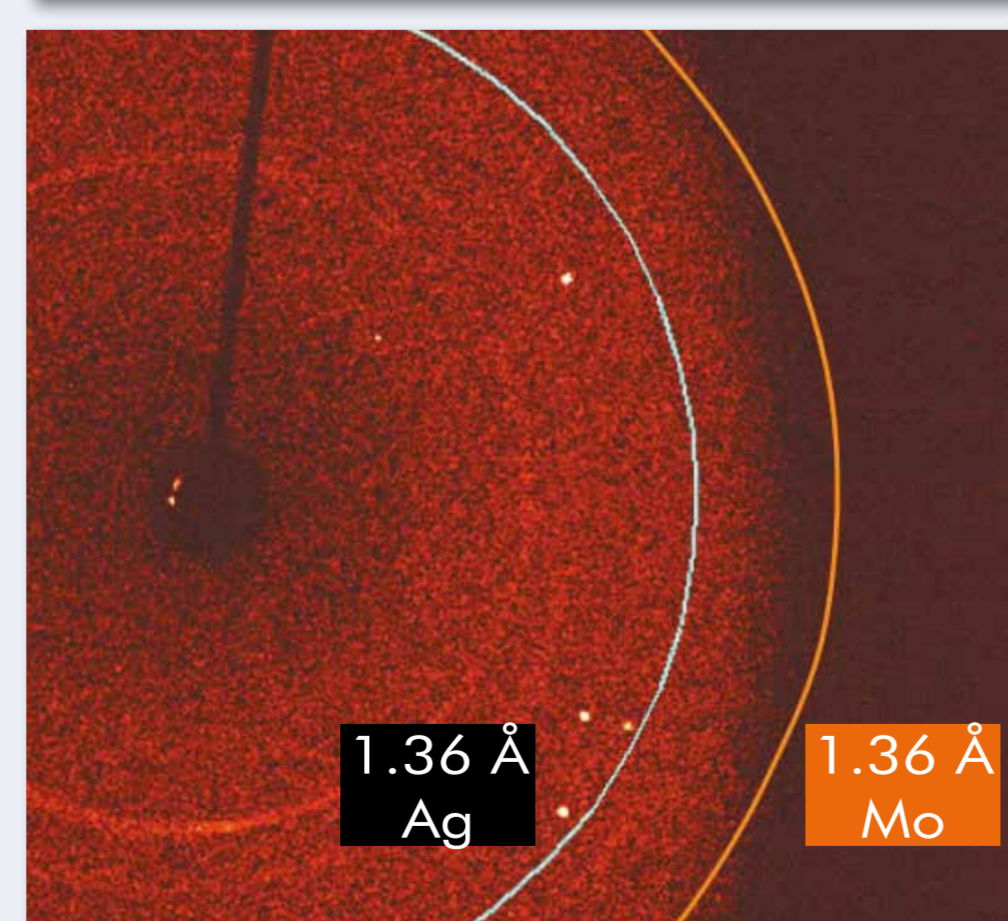
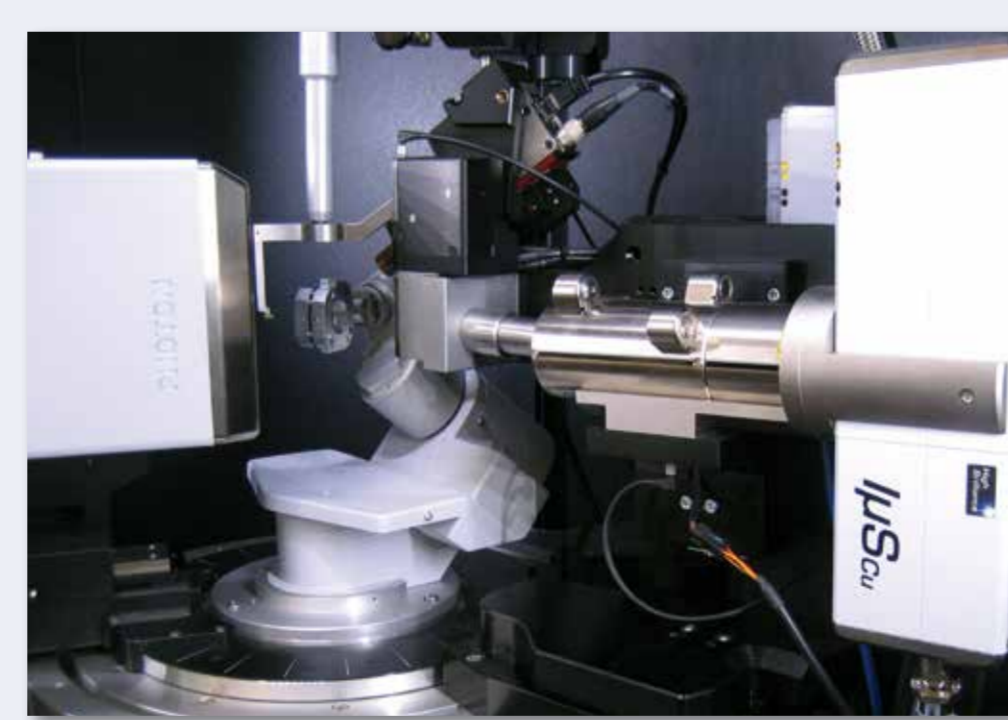
Typical diffraction pattern of the sucrose crystal, recorded with Mo- $\mu\text{S}$  HB (right), electron density and residual electron density (after IAM refinement, left, above) and electrostatic potential (left, below) of sucrose.

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

## $\mu\text{S}$ for Ag-K $\alpha$ 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- $\mu\text{S}$  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.

## $\mu\text{S}$ 3.0 - The new Microfocus X-ray Tube with IXT

The latest generation of the  $\mu\text{S}$ , the air-cooled  $\mu\text{S}$  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  $\mu\text{S}$  3.0 the most user-friendly, yet most powerful microfocus X-ray source ever.

## $\mu\text{S}$ 3.0

Incoatec Microfocus Source

- 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

**NEW**  
with IXT

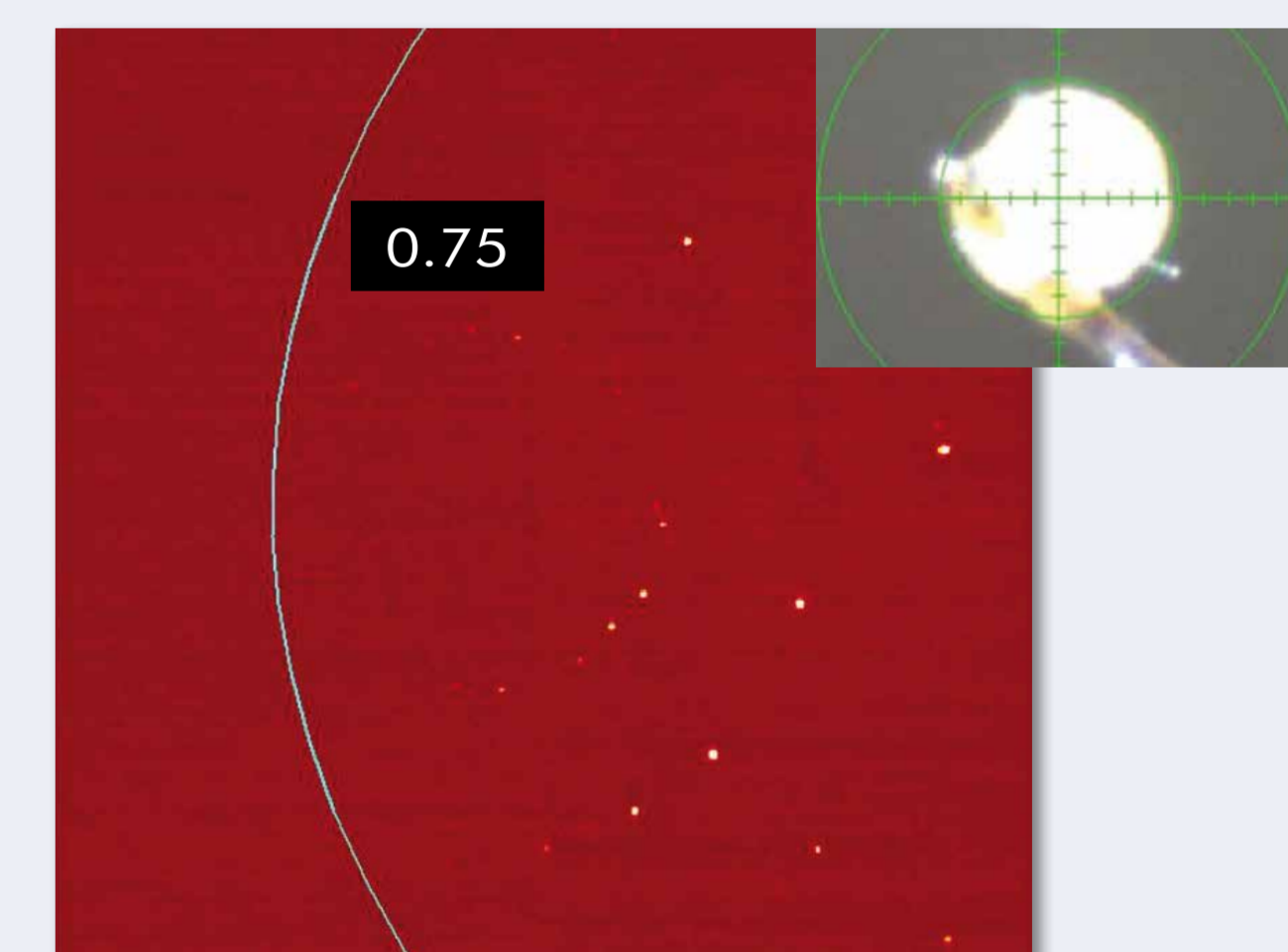
**30%**  
more  
intensity



Structure determination on a Micro-Ylid crystal

$a = 5.9691(3) \text{ \AA}$ ,  $b = 9.0265(5) \text{ \AA}$ ,  $c = 18.3725(10) \text{ \AA}$   
 $Z = 4$ ,  $P2_12_12_1$ ,  $\text{C}_{11}\text{H}_{10}\text{O}_2\text{S}$

Size [mm <sup>3</sup> ]	0.12 x 0.10 x 0.09	
Source	Mo- $\mu\text{S}$ High Brilliance	Mo- $\mu\text{S}$ 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)
$\langle 1/\sigma \rangle$	27.0 (5.4)	32.0 (7.6)
R1, wR2 [%]	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)

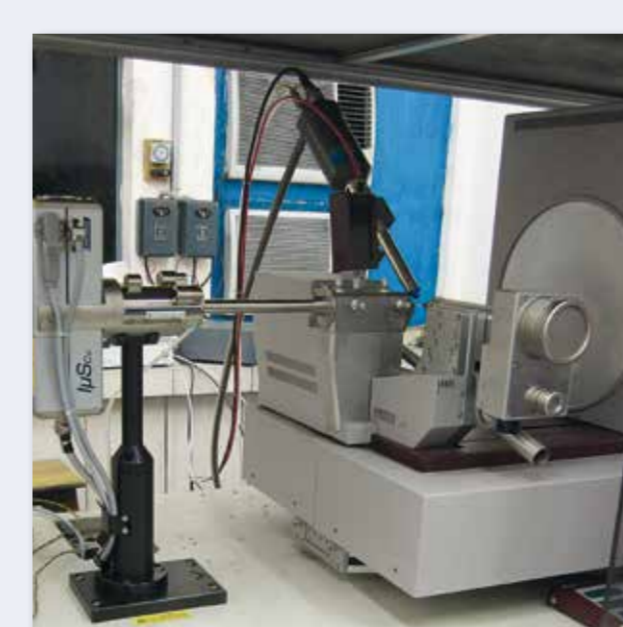


Typical diffraction pattern with Mo- $\mu\text{S}$  3.0, recorded with a D8 VENTURE. The 30% gain in intensity leads to an improved fit for the structure refinement and to lower standard deviations for the structural model.

## Upgrading Existing Diffractometers with the Microfocus Source $\mu\text{S}$

You have a Bruker AXS, Marresearch, Nonius, Rigaku, Huber or some other system?

Incoatec supports a full integration of the  $\mu\text{S}$  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.



$\mu\text{S}$  upgrade on a customized setup in Kolkata, India



Bruker APEX II DUO  $\mu\text{S}$  in Düsseldorf, Germany



Replacement of Rigaku RU-200 generator in Boulder, USA



Bruker NANOSTAR ( $\mu\text{S}$  and SCATEX) in Vienna, Austria

## Conclusion

The Incoatec Microfocus Source  $\mu\text{S}$  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  $\mu\text{S}$  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  $\mu\text{S}$ . This makes the  $\mu\text{S}$  3.0 the most user-friendly, yet most powerful microfocus X-ray source ever.