# High-Brilliance Home-Lab X-Ray Sources: Status and Future


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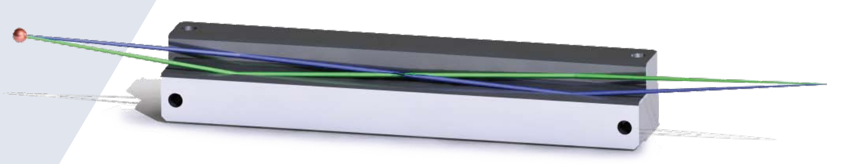
## Introduction

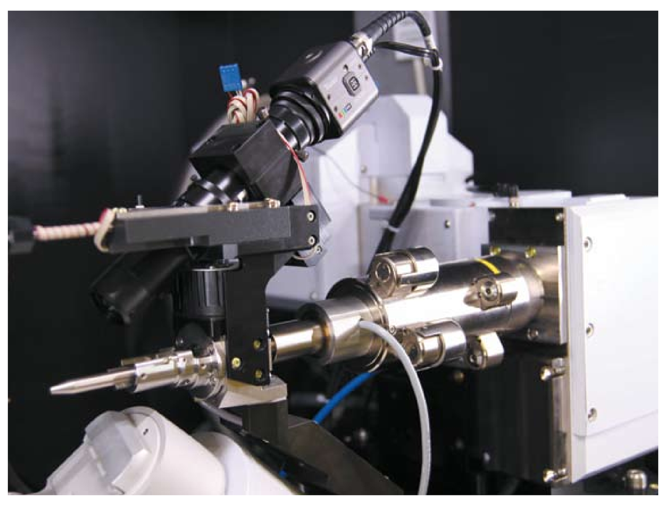
Modern microfocus X-ray sources define the state-of-the-art for a number of applications such as protein crystallography and small-angle scattering in the home lab. These sources have anode spot sizes of 100 μm or smaller. They are usually combined with Montel multilayer optics as beam-shaping devices that image the source spot onto the position of the sample, magnifiying the beam to a suitable size. Multilayer optics deliver a parallel or focused monochromatic beam.



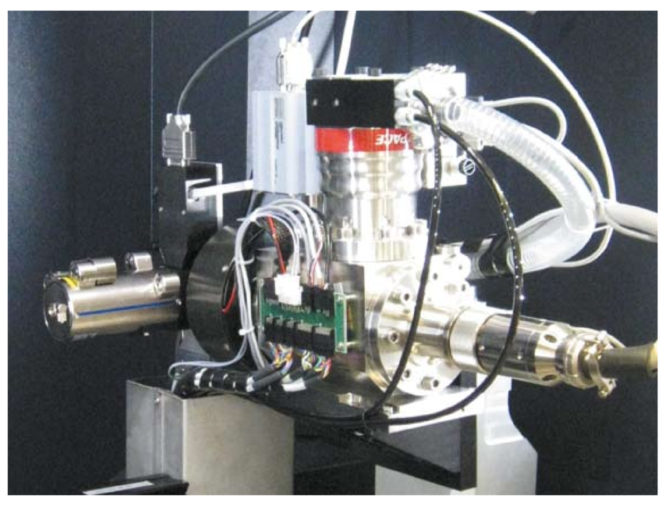
Optical scheme of a Montel multilayer optics (Incoatec GmbH). Brand names such as Helios, Quazar, or MX are also in use.

Below results of three different microfocus sources are shown: a sealed tube source, a rotating anode source, and a liquid metal jet X-ray source. The power densities of these three X-ray sources range from several kW/mm² for the sealed tube source, to about 20 kW/mm² for the rotating anode source, and to more than 500 kW/mm² for the liquid metal jet X-ray source.

Sealed tube microfocus source IμS (Incoatec GmbH, Cu anode, 50 μm focal spot, 30 W), equipped with a Quazar multilayer optics.



Microfocus rotating anode X-ray generator TXS (Bruker AXS, Cu anode, 100 μm focal spot, 2.5 kW), equipped with a Helios MX multilayer optics.



Ga liquid metal jet X-ray source (Excillum AB, 20 μm focal spot, 200 W), equipped with a dedicated Montel optics for Ga radiation (9250 eV).

## Incoatec Microfocus Source IμS

The IμS is a low-maintenance, high-brilliance sealed tube X-ray source. When combined with a dedicated multilayer optics, such as the Quazar (FWHM = 0.25 mm, 5.1 mrad) or the Quazar MX (FWHM = 0.10 mm, 7.6 mrad), the IμS delivers a flux density of up to $10^{10}$ photons/(s mm²) in a 2D focused beam. The latest version of the IμS, the new IμS$^{\text{High Brilliance}}$, has an improved X-ray optical design that even leads to a flux density of up to $2 * 10^{10}$ photons/(s mm²). The IμS gives, therefore, intensities comparable to older microfocus rotating anode generators. This makes the IμS ideal for screening and structure determination of most protein crystals, including SAD phasing experiments.

### Data Collection on a Small and Poorly Diffracting Thrombin Crystal (Quazar Optics)

| | |
|---|---|
| crystal size | 0.10x0.10x0.05 mm³ |
| exposure time | 10 min / 0.5° |
| total time | ~ 69 h |
| resolution | 19 - 1.95 Å (2.05 - 1.95 Å) |
| $<I/\sigma>$ | 13.7 (2.8) |
| <redundancy> | 3.7 (3.6) |
| <completeness> | 99.5 % (99.5 %) |
| $R_{int}$ | 0.0807 (0.4175) |
| $R_{p.i.m.}$ | 0.0487 (0.2540) |
| $R1(I > 2\sigma(I)$ ; all) | 0.2107; 0.2693 |

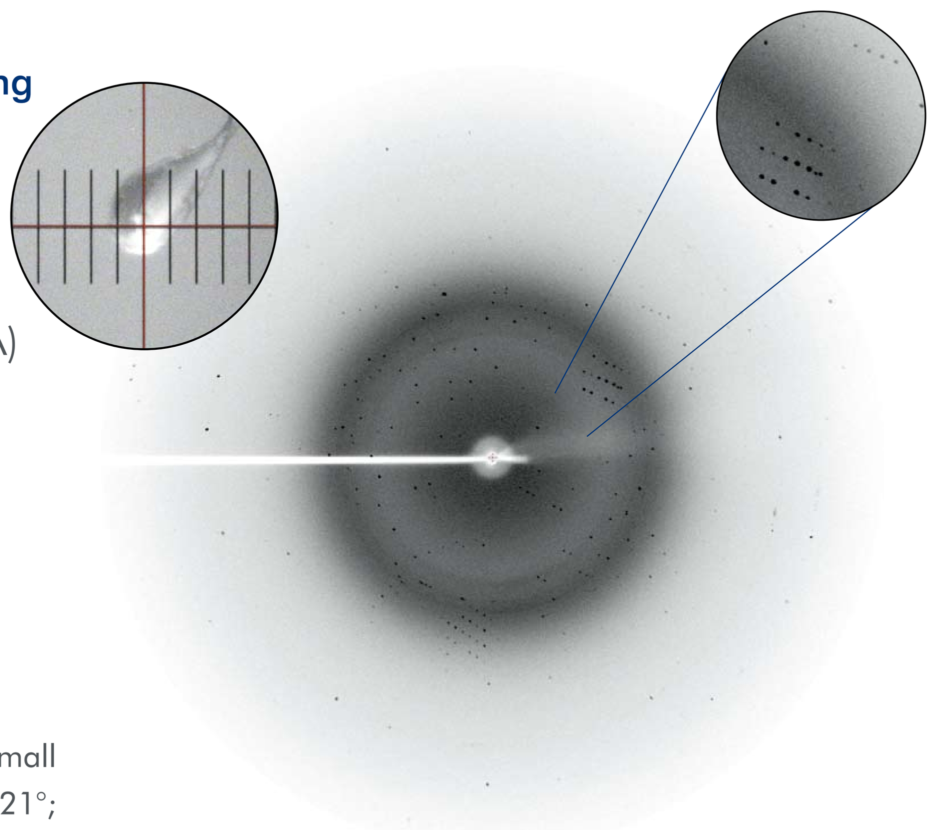


Fig. 1: Data statistics and a typical diffraction pattern of a small thrombin crystal ($a$=70.27Å; $b$=71.58Å; $c$=72.25 Å; $\beta$=100.21°; $C2$; $T$=100 K; 290 amino acids/ASU); measured with a mardtb equipped with the IμS (Quazar optics). The crystal was measured beforehand on a rotating anode system where it showed diffraction down to 2.0 Å (10 min / 0.5°, 4 kW).

### SAD Phasing on Glucose Isomerase (Quazar Optics)

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

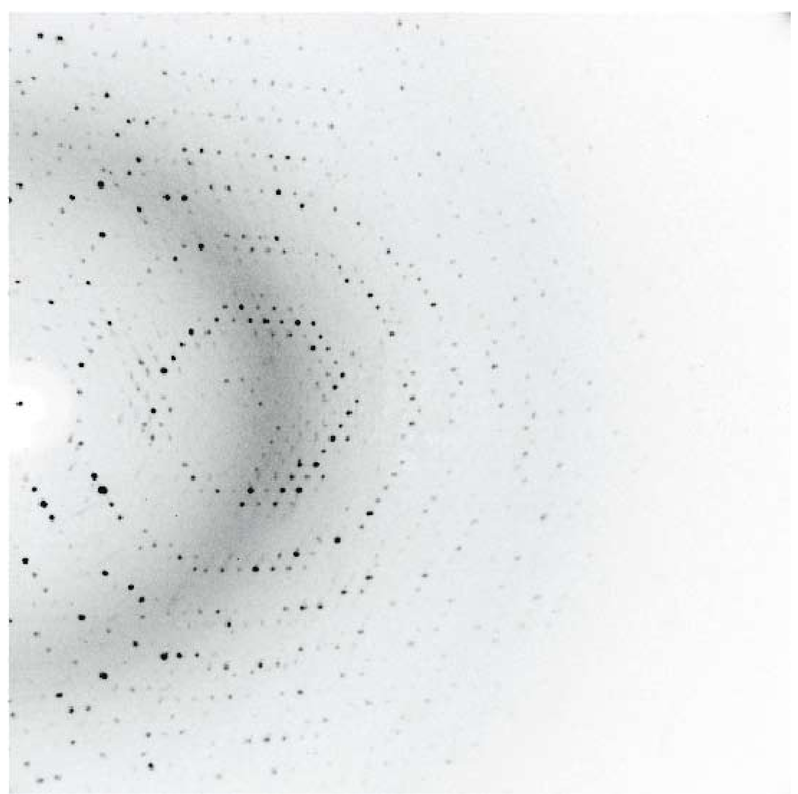


Fig. 2: Data statistics and a typical diffraction pattern of glucose isomerase ($a$ = 93.88 Å, $b$ = 99.68 Å, $c$ = 102.90 Å; $I222$; $T$ = 100 K; 388 amino acids / ASU). $Ca^{2+}$ site has been found in the initial phasing with SHELXD; measured with a Bruker Smart6000 diffractometer equipped with the IμS (Quazar optics).

### Fragment Screening (Quazar MX Optics): Twinned crystal of an enzyme in complex with a small molecule fragment

| | |
|---|---|
| crystal size | 0.45 x 0.07 x 0.04 mm³ |
| exposure time | 15 s / 0.5° |
| total time | 3.5 h |
| resolution | 41.00 – 2.59 Å (2.69 – 2.59 Å) |
| <completeness> | 95.9 % (85.8 %) |
| $<I/\sigma>$ | 18.0 (5.7) |
| $R_{int}$ | 0.0679 (0.1052) |
| $R_{\sigma}$ | 0.0642 (0.1936) |
| $R_{I}$ | 0.1331 |

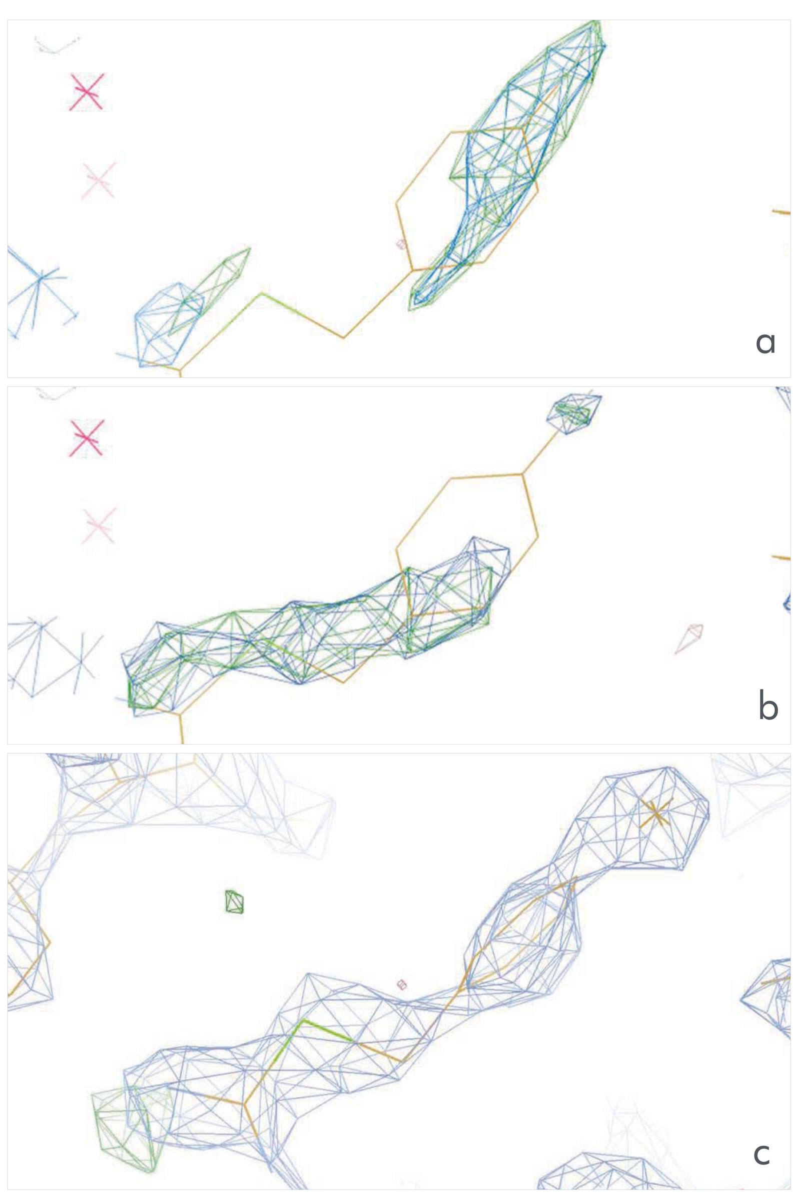



Fig. 3: Electron density map ($F_o$-$F_c$, 1.5σ in blue, $2F_o$-$mF_c$ 3σ in green) of the ligand after rigid body refinement with 60 % completeness (a, after 1 h), 80 % completeness (b, after 1.3 h) and 96 % data completeness (c, after 3.5 h and SHELXL refinement).

## Microfocus Rotating Anode

Modern microfocus rotating anode generators (μRAGs), such as the Microstar MX or the Turbo X-ray Source (TXS), allow up to 10 times higher power loads than microfocus sealed tube sources. In combination with high performance multilayer optics, this type of μRAG delivers intensities that are more than an order of magnitude higher. This allows for diffraction studies on very small and poorly diffracting protein crystals and complete data collection with significantly shorter exposure times.

### 30 s Data Collection on a Lysozyme Crystal

| | |
|---|---|
| crystal size | 0.17 mm diameter |
| exposure time | 0.5 s/° |
| total time | 30 s |
| resolution | 22 - 2.00 Å (2.10 - 2.00) |
| $<I/\sigma>$ | 15.73 (4.24) |
| <redundancy> | 3.68 (1.73) |
| <completeness> | 97.6 % (85.1 %) |
| $R_{int}$ | 6.34 (17.89) |

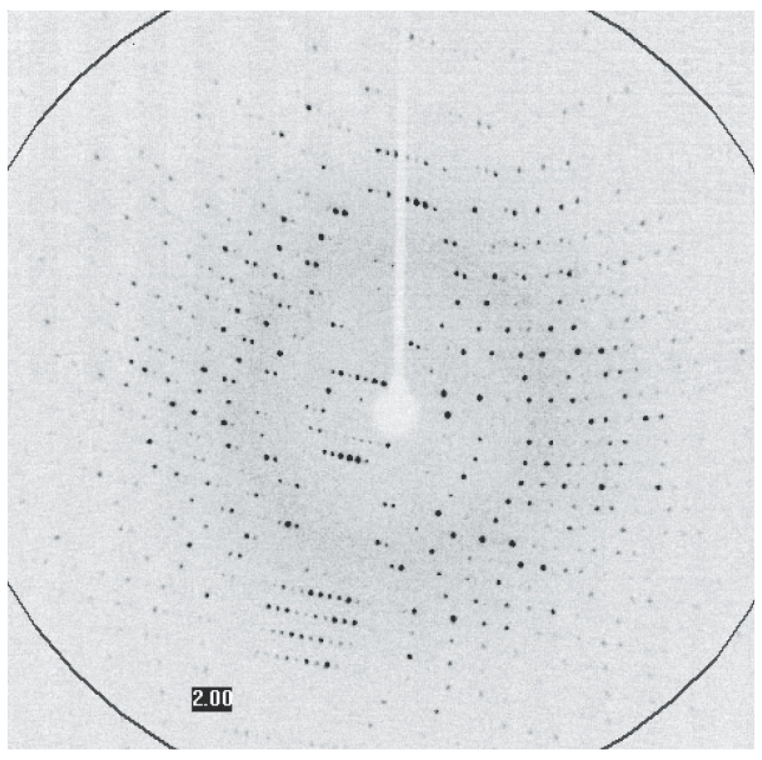



Fig. 4: Data statistics and a typical diffraction pattern of a 30 sec lysozyme data set; measured with a Bruker AXS X8 Proteum system equipped with Helios MX optics.

## Liquid Metal Jet X-ray Source

The maximum power load that can be applied to solid metal anodes is limited primarily by the thermal properties of the anode material and by the heat dissipation mechanism which sets a hard limit for future improvements of X-ray sources based on solid metal targets. In liquid metal jet X-ray sources, however, X-rays are generated by an electron beam that is focused on a jet of a liquid metal melt, such as Ga, In or Sn. An anode made of a liquid melt jet has a higher heat capacity than a solid metal anode and allows for a much higher anode speed. Therefore, liquid metal jet anodes allow for power loads that are an order of magnitude higher. Liquid metal jet X-ray sources represent a promising development for future high brilliant sources with intensities up to $10^{12}$ photons/(s mm²).

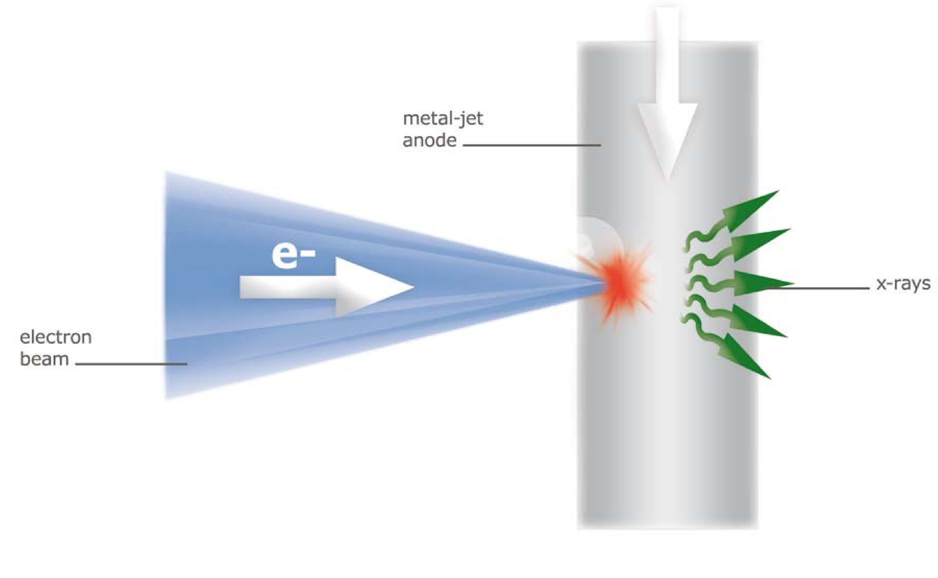
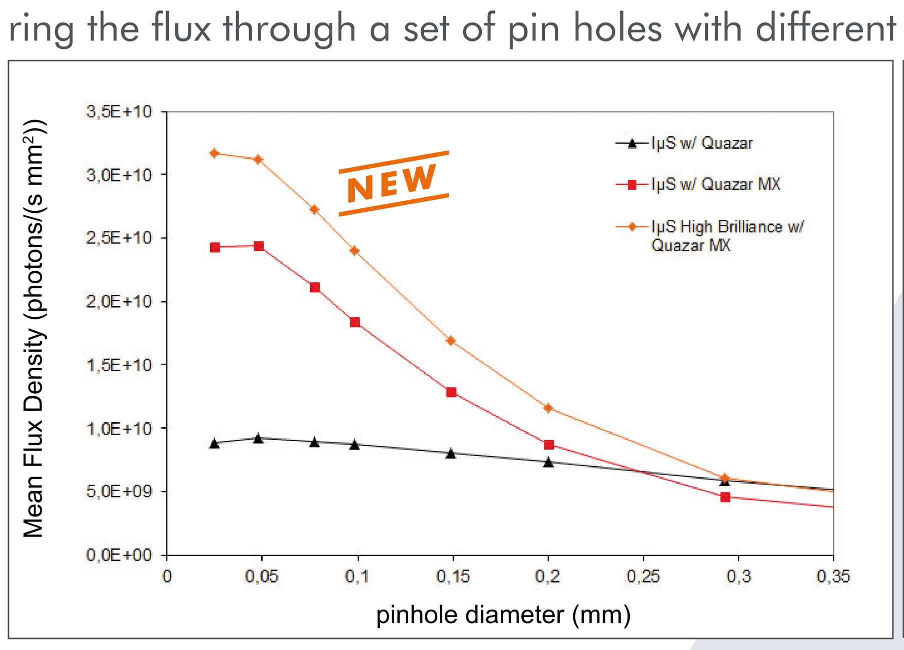


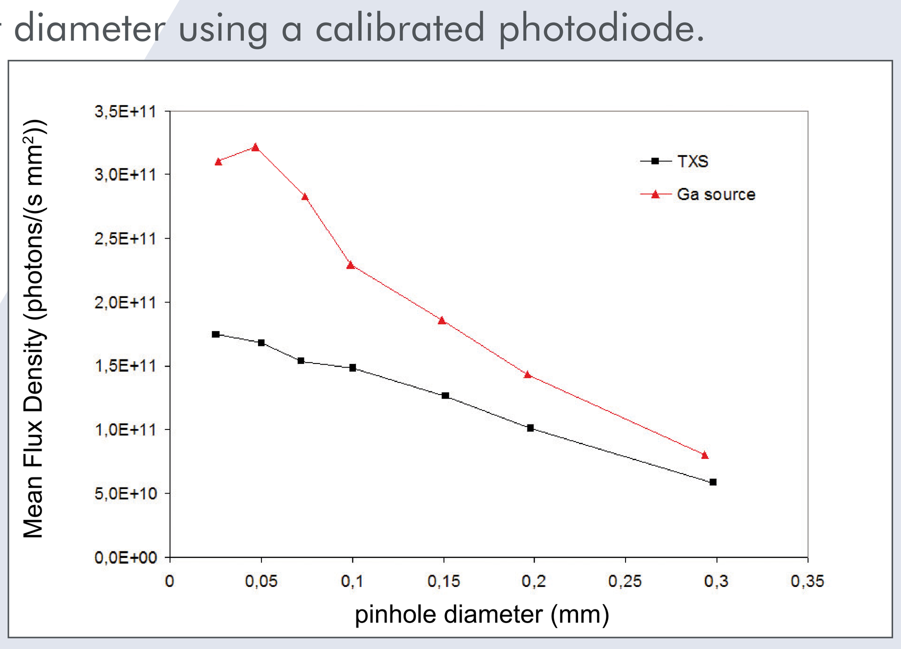

Fig. 5: Working principle of a liquid metal jet anode.

## Flux Density Comparison

A good way to compare the performance of X-ray sources independent of sample properties is measuring the flux through a set of pin holes with different diameter using a calibrated photodiode.




Flux density measurement of the IμS with Quazar optics (5.1 mrad divergence, beam size 0.25 mm FWHM) compared to flux density measurements with the IμS and the new IμS$^{\text{High Brilliance}}$ with Quazar MX optics (7.6 mrad divergence, beam size 0.10 mm FWHM).




Flux density measurement of a TXS rotating anode with Helios MX optics (7.6 mrad divergence, beam size 0.15 mm FWHM) and of a liquid metal jet source (68 wt.% Ga) with a dedicated Montel optics (7.5 mrad divergence, beam size 0.12 mm FWHM).

## Conclusion

Microfocus sealed tube sources, such as the IμS and IμS$^{\text{High Brilliance}}$, have all the advantages of a sealed tube system and a flux density exceeding that of traditional home-lab X-ray sources. Microfocus rotating anode sources provide about 10 times more intensity but their potential for major improvements seem to be exhausted. Liquid metal jet sources use a new technology and have already shown intensities superior to the best mircofocus rotating anodes. These sources have a clear potential for significant improvements in the future.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