Technical Note SC-XRD 11

Characteristics and Relative Performance of Molybdenum X-ray Sources for Chemical Crystallography

Introduction

Although the use of copper radiation is becoming increasingly popular, especially with the advent of dual-wavelength systems, the majority of chemical structures are still solved using Molybdenum radiation. Mo radiation is the principal choice for chemical crystallography due to its combination of relatively high intensity, efficiency*, and relatively low absorption.

Recent developments in X-ray source and optics technology now give the user a number of new options. This Technical Note discusses the characteristics and relative performance of the new X-ray sources now available for Mo radiation including curved-crystal monochromators, microfocus sealed tubes, and microfocus rotating anode generators.

An overview of Mo laboratory sources

The traditional workhorse for chemical crystallography has been the standard Mo sealed tube with a flat-graphite monochromator. More recently, however, curved-crystal monochromators—originally used at synchrotron beamlines—have been optimized for the relatively large source size of a conventional sealed tube. The Bruker TRIUMPH curved-crystal monochromator (Figure 1) delivers a broad beam profile that can easily be optimized to a broad range of sample sizes, and an intensity at the sample up to 8 times higher than that of a classic flat-graphite crystal (see Appendix). The TRIUMPH can easily be added as an upgrade to many existing sealed-tube-based instruments (including the SMART APEX II and D8-based configurations of the KAPPA APEX II).

Another newer option is the microfocus source with multilayer optics. The latest Bruker/Incoatec IμS microfocus sources (Figure 2) can now achieve intensities nearly 20 times that of a classic Mo sealed tube/graphite monochromator. Microfocus sources' performance has steadily improved due to advances in tube and mirror technology, and the IμS has proven its reliability and stability with over 500 installed systems over the nine years since the IμS was introduced. IμS tubes are guaranteed for 3 years and typically deliver approximately 5 years of full-power service.

*Given that a dataset to atomic resolution can most often be collected with a single detector position.

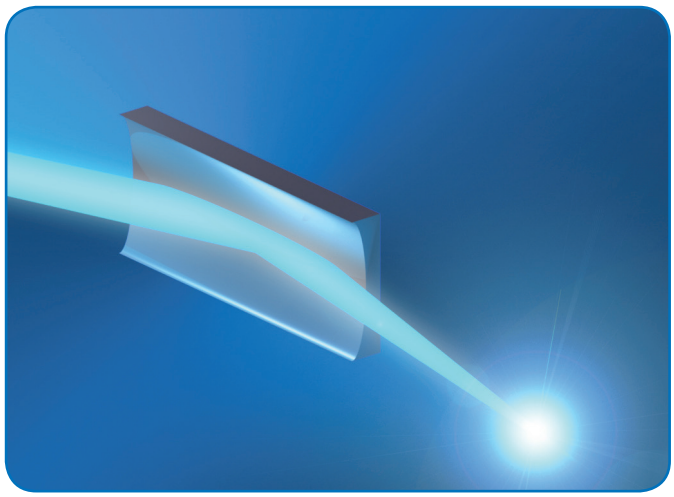


Figure 1: The TRIUMPH monochromator employs a curved crystal to deliver a beam intensity up to 8 times higher than that of a standard flat graphite monochromator.

Microfocus tubes also require significantly less power than sealed tubes (typically < 100 W) and thus require only standard, single-phase power. The IμS also does not require water cooling, eliminating special room preparation and making installation much simpler.

Even higher intensities can be achieved with modern microfocus rotating anode generators; the Bruker High-Brilliance TURBO X-RAY SOURCE (TXS) delivers an intensity more than 100 times that of a classic sealed tube, suitable for the most demanding experiments (Figure 3). However, they do require more routine maintenance than sealed tube or microfocus sources, principally periodic filament changes and anode refurbishment. It should be noted, though, that the maintenance requirements for modern rotating anodes are significantly lower than for older-style conventional rotating anodes due to their lower total power loading and improvements in anode and vacuum seal technology.

The TXS is also the first rotating anode to feature a modern fixed mount with downstream alignment. That is, conventional rotating anodes are fixed to the table, and the entire goniometer must be aligned to the source (using an alignment base).

Figure 2: The IμS 3.0 microfocus source offers the highest available intensity in a sealed tube, up to 20 times that of a standard sealed tube with graphite monochromator.

In contrast, the TXS is mounted on an alignment stage which is fixed to the goniometer to allow the source to be accurately aligned to the sample (just like sealed tube and microfocus sources). This configuration is much more mechanically stable than the conventional approach, and it is also much easier and quicker to align—which means much smaller standard deviations in the final structure.

The TXS also features pre-aligned, pre-conditioned, long-life filament cassettes for easy and quick filament changes with minimal realignment.

Air cooling versus water cooling: which is better?

As noted above, the relatively low power of the IμS 3.0 microfocus source allows it to be air cooled. This provides the user with a number of benefits including ease of installation, lower operating costs, and lower noise.

Of course, it is more challenging to design an air-cooled tube as the tube must incorporate sophisticated feedback mechanisms to maintain constant temperature in the face of variations in the external air temperature. The IμS is currently the only source to have mastered this technology: a tube with a highly stable output and very long tube lifetime, superior to the best water-cooled designs.

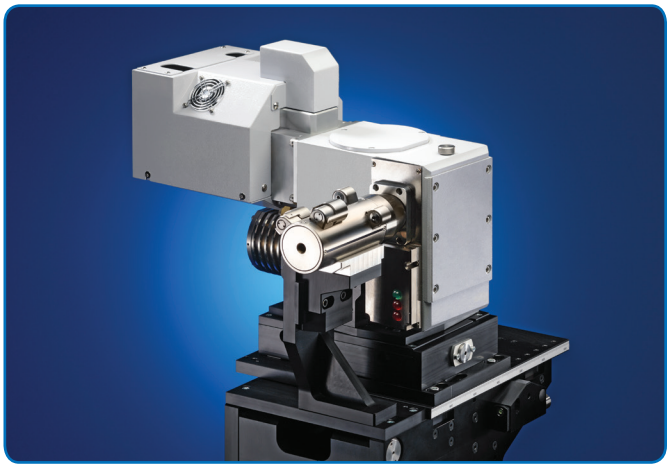


Figure 3: The TXS offers the most intense Mo beam available, up to 100 times higher than a sealed tube, suitable for the most demanding applications.

It is, of course, much easier to design a tube cooling system using water because the water temperature is externally stabilized. However, water chilling has a number of disadvantages. Firstly, the need for a water chiller obviously increases installation and operating costs, requires routine maintenance, and also increases background noise in the laboratory. Secondly, and more importantly, if the cooling water is not properly deionized, ions in the water can attack the cooled anode surface and lead to the buildup of a thermally-insulating corrosion layer. In principle, this can occur in any water-cooled tube, but it is especially a danger for microfocus tubes that require, in contrast to standard fine-focus sealed tubes, cooling of an extremely small, localized spot on the anode. This corrosion compromises the cooling of the tube and leads to decreased tube lifetimes.

Higher-power sources like the TXS microfocus rotating anode do require water cooling, since the heat load is too high to be handled by air cooling. As noted above, water cooling of a microfocus source requires excellent water quality, so the TXS incorporates a patented water deionization system to maximize the anode's lifetime and prevent corrosion of the anode's surface.

In short, for low-power sources, air cooling offers a number of compelling advantages including higher reliability, lower costs, and lower noise. High-power sources absolutely require water cooling, so in this case, it is crucial to maintain the hygiene of the cooling water to prevent damage to the source.

IμS: the importance of advanced tube design

Thanks to our partners at Incoatec, Bruker has long enjoyed the highest-quality X-ray optics. Incoatec optics feature the highest reflectivities, the lowest figure errors, and the largest collection solid angle available anywhere.

Today, the IμS also benefits from having the only microfocus tube on the market specifically designed and optimized for X-ray diffraction applications. The tubes used in other microfocus sources were designed for Non-Destructive Testing, and thus these tubes are not optimized for X-ray diffraction.

Bruker's IμS 3.0 therefore achieves by far the highest brilliance as well as an intensity fully twice as high (for Mo radiation) as the original IμS—along with legendary long lifetimes and reliability.

Microfocus source performance: is it only about the power?

Microfocus sources are often specified only by their primary power. For example, one will see a microfocus source specified as "50 W". What does this mean? Are all 50 W sources the same? Is 50 W better than 30 W?

The truth is that the primary power can be misleading. From Liouville's theorem, we know that the intensity at the sample is proportional to the brilliance of the source, not the power of the source. Further, the brilliance is proportional to the power density. So, a 30 W tube with a 10 micron focus is fully 1000 times more brilliant than a 3000 W tube with a 1 mm focus!

Another important aspect of the source's performance is the design and quality of the optics. In particular, for microfocus performance, low figure errors (essentially low "waviness") are crucial to the final intensity. For example, for a 20 micron anode focus, Bruker low-figure-error mirrors with 2 mrad arcsecond figure errors are up to 3 times more efficient than equivalent conventional mirrors with 10 arcsecond errors.

So, with all these complicated variables, how should one compare sources? The best way is to consider the intensity at the sample for a given divergence. This takes into account all of the important parameters that determine the source's performance, including brilliance and optics performance.

IμS microfocus source versus the TRIUMPH curved-crystal monochromator: which is better?

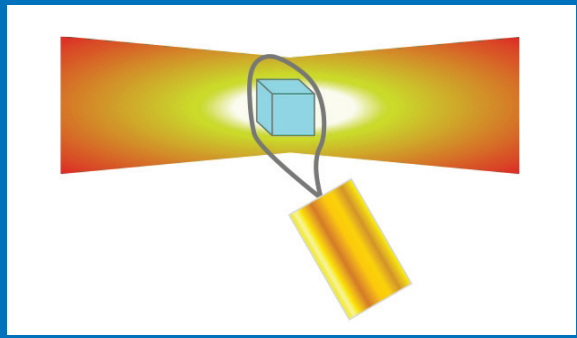
The answer to this simple question is not so simple: it depends on your samples.

It is well known that, in general, the best data for a given sample can be achieved by matching the size of the beam to the sample. If the beam is larger than the sample (as in Figure 4a), then the X-rays which do not hit the sample do not contribute to the diffracted signal. Worse, they do contribute to the scattered X-ray background and thus the overall signal-to-noise (I/sigma) is lower.

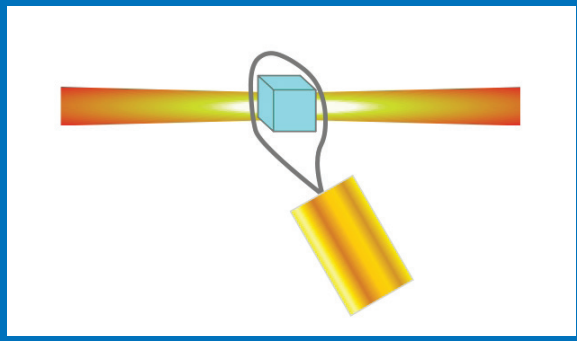
Conversely, if the beam is significantly smaller than the sample (as in Figure 4b), then the diffracted signal is also smaller due to the smaller interaction volume. Also, the variations in the interaction volume with crystal orientation lead to larger absorption errors (strictly speaking, these changes in diffraction intensity are not really "absorption" errors but the terminology is traditionally applied to this differential volume effect as the apparent changes mimic true absorption effects and are also corrected in the same way).

Again, the most favorable case is to match the size of the beam to the sample. This can be achieved by choosing a collimator which matches the size of the sample (as in Figure 4c). This optimizes the diffracted intensity, minimizes the scattered X-ray background, and also minimizes the absorption errors.

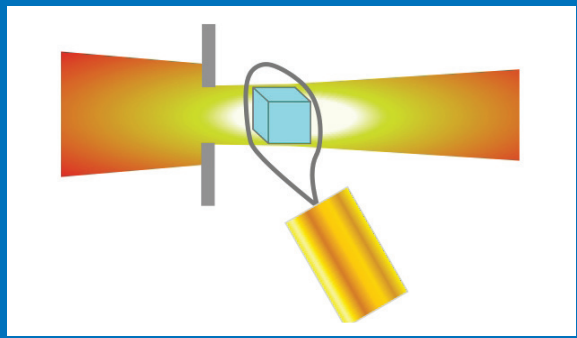
Matching the beam size to the sample size



(a) Beams larger than the sample are inefficient as most of the X-rays do not contribute to the diffracted signal but only produce scattered background.



b) Beams smaller than the sample are also inefficient as there is less interaction volume to contribute to the diffracted signal and absorption errors are higher.



(c) The ideal case is to match the beam size to the sample as this maximizes the diffracted signal (for a given beam intensity) while minimizing the absorption errors.

Figure 4: Influence of the beam size on data quality.

Influence of Beam Size

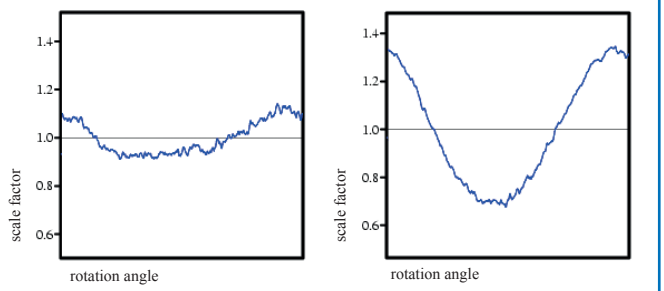


Figure 5: Absorption errors for a large crystal (120 × 320 × 400 μm^3 ascorbic acid) in a large beam (360 microns, left), and a small beam (120 microns, right). A beam smaller than the sample produces larger absorption errors. However, these errors can often be accurately corrected by collecting a highly-redundant data set and using a multi-scan scaling program such as SADABS.

So, for general-purpose crystallography where samples might range between 100 microns and 500 microns, the TRIUMPH monochromator offers a significant advantage as it features a very intense beam (up to 8 times the intensity of a standard sealed tube, which is very comparable to most microfocus sources) with a large beam size that can be easily adjusted from 100 microns up to 500 microns via appropriate collimator selection (as in Figure 4c).

On the other hand, if one is working with smaller crystals (for example, less than 150 microns), then the microfocus source has a significant advantage due to its higher intensity: almost 20 times that of a classic sealed tube.

Also, it should be understood that a microfocus source can certainly be used with samples larger than the beam (as in Figure 4b) but in this case the absorption errors will be larger (as shown in Figure 5). Thus, it is important to collect highly-redundant data in order to facilitate accurate multiscan-type absorption corrections, e.g., with SADABS.

In short, the TRIUMPH monochromator offers the best combination of intensity and flexibility for general-purpose crystallography with a broad range of crystal sizes (100-500 microns), while the IμS microfocus source offers superior performance for small crystals (less than 150 microns).

Conclusions

Three improved types of Mo X-ray sources are now available for laboratory chemical crystallography systems. They differ in performance as summarized in Table 1.

The TRIUMPH sealed tube is the most economical option and delivers the best compromise between high intensity and flexibility for a broad range of sample sizes (in the range of 100-500 microns). It is often the best choice for a general multi-purpose system.

The IμS 3.0 microfocus source offers the highest intensity from a sealed tube for optimal performance with smaller samples, together with the convenience of air cooling and single-phase power. It is ideal for smaller, more challenging samples.

The TXS rotating anode offers the highest intensity available from a laboratory source, and delivers the ultimate performance for the most demanding applications including micron-sized samples, charge density, diffuse scattering, etc.

Table 1: Comparison of the typical performance of Bruker Mo sources (sealed tube, microfocus, and rotating anode) for chemical crystallography. All values are as specified by the respective manufacturers as of August 2015. Values are for comparison only and are subject to change without notice.

Bruker Molybdenum Sources	Power (W)	Cooling	Typical tube lifetime* (years)	Beam size at focus (μm)	Max diver-gence (mrad)	Relative Intensity
Sealed tube (fine focus), graphite monochromator	2000	water	2-3	500	7	1
Sealed tube (fine focus), TRIUMPH monochromator	2000	water	2-3	500	9	8
Microfocus source, IμS 3.0	70	air	5	110	4.9	20
Microfocus rotating anode, TXS	2500	water	1**	180	4.9	100

* Typical at full power, continuous operation
** Refurbish (clean, polish) anode once per year.

Bruker AXS GmbH

Karlsruhe · Germany
Phone +49 721 50997-0
Fax +49 721 50997-5654
info.baxs@bruker.com

www.bruker.com