



Application Note 4

I μ S for High Resolution Small Molecule Crystallography

Introduction

The concept of using a microfocus X-ray source in combination with multilayer X-ray mirrors for Cu-K α radiation was first pioneered for protein crystallography by U. Arndt in the early 90's. In contrast to multilayer mirrors for Cu sources, the maximum angles of incidence at which a multilayer mirror reflects higher energy radiation, such as Mo-K α or Ag-K α radiation, are significantly smaller. With today's deposition technology, however, high quality multilayer mirrors can be produced that have a small d-spacing and reflect higher energy radiation at larger angles of incidence. These mirrors ensure high flux and make way for new high-brilliance low-power X-ray sources for shorter wavelengths.

Here, we will be comparing the performance of the Incoatec Microfocus Source I μ S for Mo-K α radiation with a fine focus sealed tube system and with a traditional rotating anode.

Experimental set-up

To compare the I μ S with a 2 kW Mo sealed tube system (graphite monochromator, 0.5 mm monocapillary), the

I μ S was attached to a Bruker Smart Apex II goniometer as a second source. The I μ S was constantly operated at 30 W (50 kV, 0.60 mA) for a period of 16 months. The flux was monitored repeatedly and no significant decrease was detected (~ 0.3 % per month).



Fig. 1: The Incoatec Microfocus Source I μ S in combination with a Bruker Smart Apex II (left) and a Nonius Kappa CCD diffractometer (right).

Furthermore, several crystals of organic and inorganic compounds were measured with the I μ S attached to a Nonius Kappa CCD diffractometer. Reference data sets of the same crystals were recorded with a FR591 Mo rotating anode (graphite monochromator, 0.3 mm collimator). The rotating anode generator was operated at 4 kW (50 kV, 80 mA).

Beam Characteristics of the μ S

The Mo- μ S delivers a focused and monochromatic X-ray beam with a FWHM of 0.12 mm in the image focus and a flux density of about 1×10^9 counts/(s mm²) calculated over the central beam area. In contrast to the typical intensity plateau that is produced by sources with a graphite monochromator, the beam profile from the μ S has a symmetrical Gaussian-shaped intensity distribution with high peak intensity. The flux density of the μ S is about four times higher than the flux density of the 2 kW Mo sealed tube system and comparable to that of the 4 kW rotating anode generator. The beam divergence of the μ S is 5 mrad, whereas the two systems with graphite monochromator have a beam divergence of about 8 mrad.

μ S compared to a 2 kW Mo sealed tube

Comparative measurements have been performed on two crystals of the organic compound C₂₄H₂₁N₃O₃ (Fig. 2) under identical conditions. The total gain in intensity achieved with the μ S was more than a factor of 7 for a small crystal and about a factor of 4 for a larger crystal. The signal-to-noise-ratio after scaling is in both cases clearly in favour for the μ S. Table 1 summarizes details of the comparative measurements on C₂₄H₂₁N₃O₃ (T. Schulz et al. *J. Appl. Cryst.* (2009) 42, 885 – 891).



Fig.2: Picture of the molecule C₂₄H₂₁N₃O₃ (P2₁, T = 100 K, H-atoms are omitted for clarity).

Source	μ S	Sealed Tube	μ S	Sealed Tube
Size [mm ³]	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
$\langle I \rangle^\#$	139	19	4763	1286
$\langle I/\sigma \rangle$	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
R _{σ}	0.053	0.090	0.019	0.022
R1	0.039	0.056	0.030	0.031
wR2	0.092	0.105	0.082	0.082
N1-C9 distance[Å]	1.325 (3)	1.325 (4)	1.323 (2)	1.324 (2)

Tab.1: Details of four sets of data collected on C₂₄H₂₁N₃O₃ (# normalized to 1°/min; * values for highest resolution shell 0.85 - 0.75 Å).

μ S compared to a 4 kW rotating anode

Several data sets were measured on crystals of organic and inorganic compounds with the μ S and with a FR591, both attached to a Nonius Kappa CCD. Table 2 shows details for two representative crystals (quartz, ammonium-bitartrate). The data clearly demonstrate the superior performance of the μ S on very small crystals for which an increase in intensity by a factor of about 3 was observed. For larger crystals, the performance is comparable to a 4 kW rotating anode generator with graphite monochromator.

Source	μ S	RAG	μ S	RAG
Sample	SiO ₂		C ₄ H ₉ O ₆ N	
Size [mm ³]	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
$\langle I \rangle$	17.1	5.9	344.1	537.9
$\langle \sigma \rangle$	1.6	1.1	5.1	6.8
R1	0.084	0.090	0.036	0.041
wR2	0.240	0.241	0.082	0.082

Tab.2: Details for sets of data recorded on a smaller quartz crystal and a larger crystal of ammonium-bitartrate.

Conclusion

The μ S for Mo radiation yields excellent results for single crystal diffraction experiments. The focusing multilayer optics deliver maximum flux on the sample and improve the signal-to-noise ratio by minimizing the background. The μ S is ideal for routine and high resolution structure determinations and for special applications, such as high-pressure experiments. For very small crystals, the μ S showed an intensity gain of factor of 3 compared to the 4 kW rotating anode. The data quality from crystals with a diameter of about 0.1 mm was comparable to that from the FR591 rotating anode generator.

The air-cooled μ S has all the advantages of a sealed tube system and is geared up as an upgrade for all existing diffractometers, offering high performance together with low maintenance and low operating costs.

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