Small Molecule
GeniX Mo High Flux

Application Note n° AN-G7

Abstract

The traditional solution for diffraction applications requiring Molybdenum radiation is based on 2KW sealed tube with a graphite monochromator and monocapillary optics. The GeniX Mo High Flux beam delivery system constitutes a new approach based on a high reflectivity single reflection optic coupled to a high-brightness microfocus X-ray source.

At STOE GmbH a traditional sealed tube system and a GeniX Mo High Flux system were tested with the IPDS2T for small molecule, high pressure, and powder diffraction applications. Results of these tests show that the GeniX Mo High Flux system leads to a significant improvement in spot resolution and a lower background level compared to 2KW sealed tube solution.

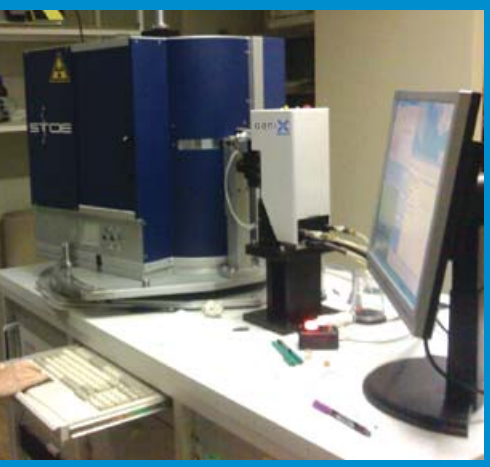

Fig. 1 : The combined GeniX-IPDS2T setup

GeniX Mo High Flux tested on a STOE IPDS2T system for Small Molecule and Powder Diffraction

Data courtesy of Dr. Jens Richter, STOE GmbH, Darmstadt, Germany.

Introduction

Following successful tests with the GeniX Cu High Flux micro-beam generator, the Mo version was tested on a STOE IPDS2T System to evaluate its performance for applications requiring Mo Kα radiation.

Experiment

The first test consisted of measuring an organometallic compound on an IPDS2T system initially equipped with a sealed tube and planar graphite crystal monochromator and subsequently with a GeniX Mo High Flux system. The data acquired using the GeniX showed a significant improvement in spot resolution and a lower background level. In addition, the GeniX delivered slightly higher intensity compared to the sealed tube system.

To perform a more rigorous comparison between the two systems, two measurements with the same conditions (other than generator settings) were conducted. The imaging plate was positioned at a distance of 100 mm from the crystal, which resulted in a maximum 2Θ angle of nearly 60° at full radius (170mm) readout. The exposure time was set to 2 min / frame, the omega increment to 1°, and a full omega range from 0° to 180° was collected. The results, reported in Table I, indicate that the data collected with the GeniX are slightly better.

Table I : Comparison of the results for the single crystal test measurements

	GENIX	Sealed Tube 0.5 mm Collimator
kV / mA	50 / 1	50 / 40
Max 2θ	58.29	58.40
Mean I/σ	8.46	5.40
Nr. of Reflections	26003	25743
Nr. of equiv. Reflect.	3634	3615
R(int) SHELX	0.0521	0.0616
Rσ SHELX	0.0397	0.0611
R1 (>4σ)	0.0272	0.0283
R1 (all)	0.0521	0.0773
wR2	0.0642	0.0542
GooF	0.850	0.765
Highest Peak	0.20	0.23
Deepest Hole	-0.31	-0.32

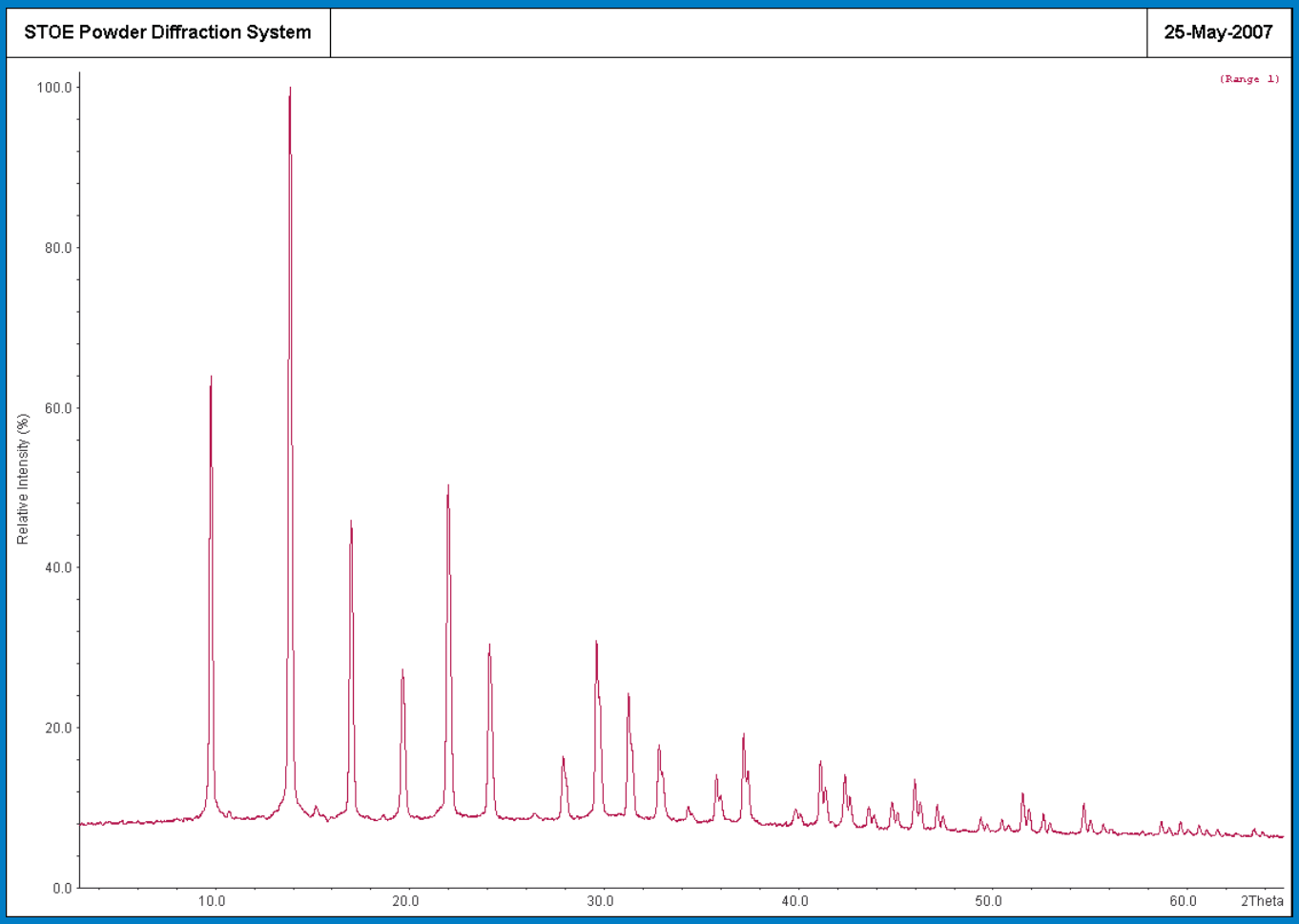

Fig. 3 : Lab6 measurement optimized for resolution

To compare the resolution achievable with the two systems, a powder experiment was performed using LaB6 in a 0.3 mm capillary exposed for 3 minutes with a sample-detector distance of 80 mm. The results, shown in Fig. 2, highlight the improved resolution obtainable using the 50 W GeniX Mo High Flux microbeam delivery system (50 kV, 1 mA) compared to the 2 kW sealed tube (50 kV, 40 mA) with a 0.3 mm collimator.

Due to these promising results a further test became necessary to determine the performance level for powder measurements of the GeniX Mo High Flux in combination with the STOE IPDS2T. A pinhole system was used to lower the divergence of the beam and a measurement of LaB6 powder in a 0.3 mm glass capillary was carried out. The exposure time was 20 min with 200 mm between the sample and the detector. The detector was positioned at 30° 2Θ which yields a total 2Θ value of 70°. The integration of the intensities, using the STOE X-Area software suite, showed a mean FWHM of 0.15°.

Conclusion

In conclusion, three types of experiments were successfully performed on a single diffractometer system with minimal system reconfiguration, demonstrating the versatility of the GeniX Mo High Flux system. Its beam properties may easily be adapted according to sample and experiment requirements.

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