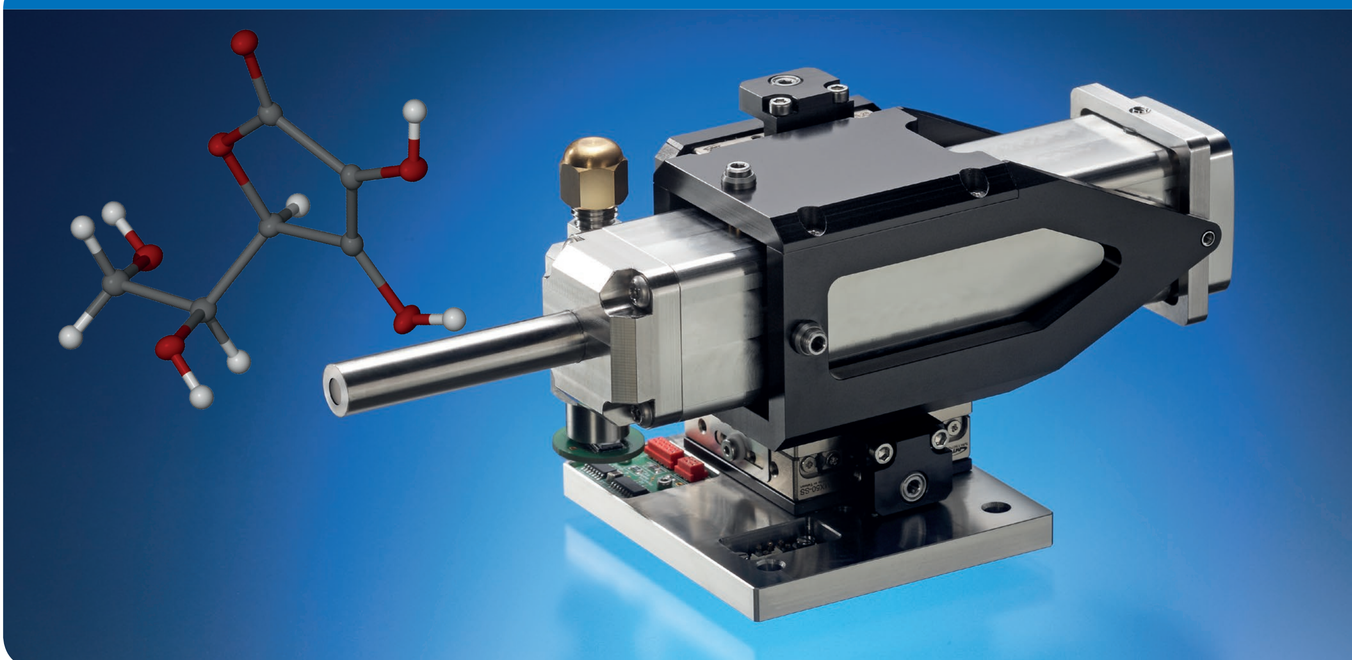

Application Note SC-XRD 519

Best Data Quality From Your Small Organic Sample

- HELIOS EF for IµS 3.0: best ever microfocus sealed tube intensity

Introduction

The need to collect data from smaller, weaker diffracting samples continuously pushes the limits of the developments in X-ray sources, optics and detectors. We designed the new HELIOS EF optics for copper Kα radiation to deliver a highly focused beam with a divergence that matches the typical mosaicity of small and weakly diffracting samples in chemical crystallography. This new optic was developed for the IµS 3.0 microfocus X-ray source, the only micro focus X-ray source optimized for the needs of crystallography.

The HELIOS EF optics delivers more than twice the flux and intensity of the HELIOS MX optics, and the highest flux density of any sealed tube micro-focus source in a small highly-focused beam.

A Small Organic Sample

In order to demonstrate the impact of the highly focusing HELIOS EF optics on the data quality for an organic routine sample, we collected data from a small crystal (0.04 × 0.10 × 0.10 mm^3) of ascorbic acid ($C_6H_8O_6$) using a D8 VENTURE equipped with the IµS 3.0 (Cu) X-ray source. For the comparative data collection, data were first acquired with a regular HELIOS MX optics as a reference data set. The optics was then replaced by a HELIOS EF optics, and the same data collection strategy was applied for maximum comparability.

Due to the unique Quick-lock concept of the IµS 3.0, the exchange of the optics can be accomplished in just a few minutes. The precise mechanics of the entire source guarantees a very high reliability and assures that the replacment optics perfectly mounts into the correct position. This makes a realignment of the instrument obsolete. As part of the Davinci design every optics comes with its own sensor for an automated component recognition, and individual calibration parameters for the modified setup are automatically applied to the instrument.

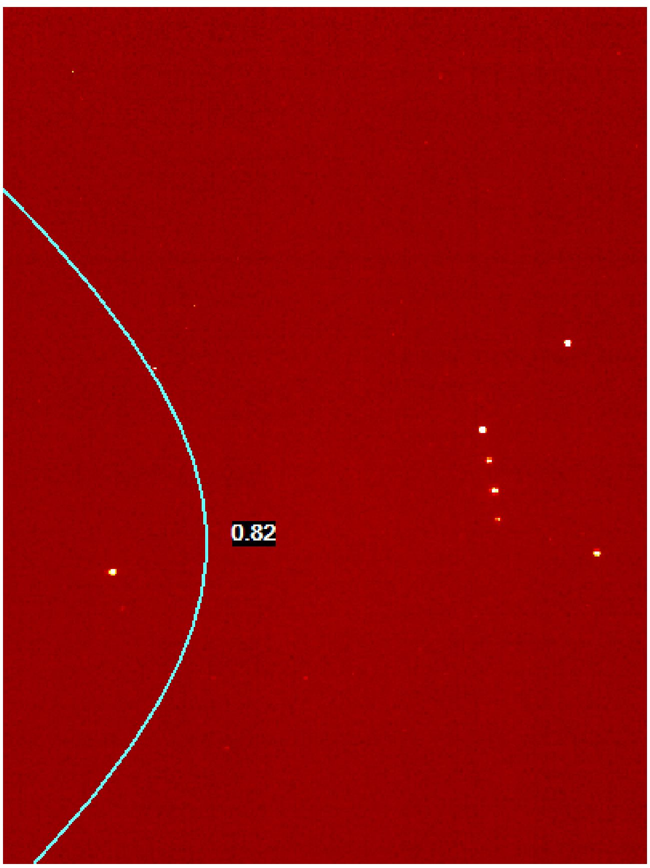

Figure 1: Typical diffraction pattern of a small Vitamin C crystal (0.04× 0.10 × 0.10 mm^3, T = 100 K, $P2_1$, a = 6.396 Å, b = 6.316 Å, c = 17.099 Å, β= 99.34°, IµS 3.0 (Cu) with HELIOS EF optics)

Table 1. Crystallographic data obtained from a small crystal of ascorbic acid

IµS 3.0 (Cu)	HELIOS MX	HELIOS EF
Exposure time [s/0.5°]	2 / 4	2 / 4
Relative normalized intensity	1	2.14
$<I/\sigma(I)>$	27.1	37.0
R(int) [%]	4.91	3.75
$R1$(Fo > 4σ(Fo))	3.27	2.59
w$R2$ [%]	7.51	6.65
d(C-C) [Å]	1.526(5)	1.527(3)

As shown in Table 1, the higher flux and the higher flux density increase the intensities of the integrated reflections by more than a factor of 2. This leads to a significant reduction of all R values and an improvement in the structural model, as can be seen in the decrease of the standard deviations. It should also be noted that, for moderate diffracting samples, the increase in intensity from the HELIOS EF optics can, of course, be used to reduce the overall experimental time by a factor of 2 by simply halving the exposure time per image.

Summary

The HELIOS EF optics was designed by matching the beam divergence to the typical mosaicity of weak diffracting samples in chemical crystallography delivering more than twice the intensity of the regular HELIOS MX. It clearly improves the data quality for weakly diffracting samples and helps to reduce the overall experiment time.

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