The Rigaku Journal
Vol. 17/ number 1/ 2000

Product Information

Data Quality Improvement in the Home Lab: Can the FR-D Help?

Abstract

We present a preliminary comparison for the RU-H2R rotating anode generator with MSC Confocal Blue-3 optic and a FR-D high brilliance generator with both MSC Confocal Blue-1.5 and Purple-1.5 optics. We show the results of characterization of the physical properties of the X-ray beam and data sets collected on the same small frozen lysozyme crystal from these systems. Across all measurements, we observe the FR-D Blue-1.5 and Purple-1.5 systems perform 2 to 5 times better than the laboratory reference RU-H2R Blue-3.

Introduction

The FR-D high brilliance rotating anode generator was designed as a successor to the FR-C generator. It provides a high brilliance focal spot, 3.5 kW at 0.1×0.1 mm or 5.0 kW at 0.15×0.15mm, while retaining the ease of maintenance associated with the Rigaku RU series of generators. This generator has been coupled with Osmic confocal optics to provide the most powerful home laboratory source available, Figure 1. The configuration shown is that installed at the Scripps Research Institute, La Jolla, California, in the laboratory of Professor Raymond Stevens.

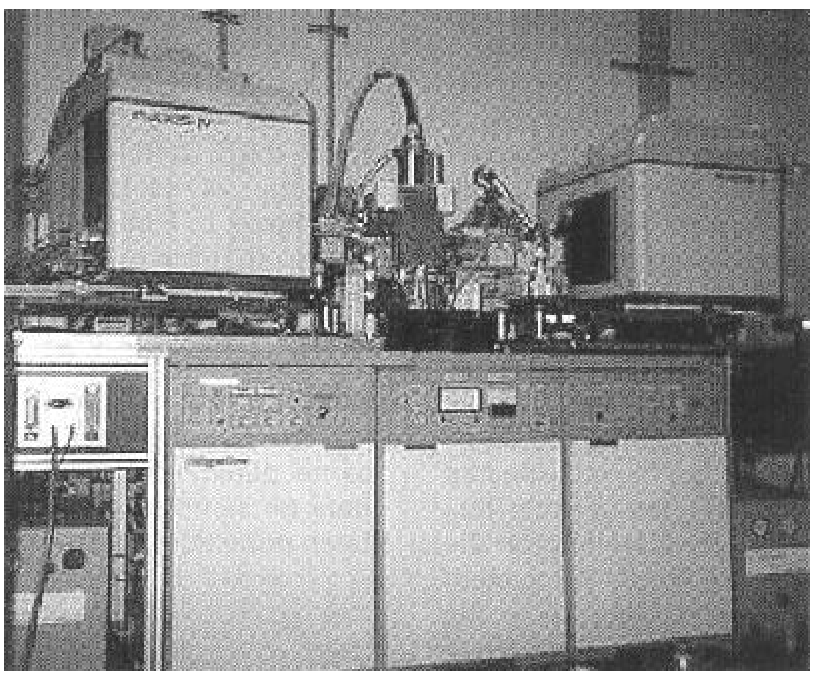

Fig. 1. The FR-D with Blue-1.5 (left port) and Purple-1.5 Optics as installed at the Scripps Research Institute.

Experimental

The RU-H2R and MSC Confocal Blue-3 system has been described elsewhere.[1] Table 1 shows these configuration parameters as well as the relevant configuration information for the FR-D with the Blue-1.5 and Purple-1.5 optics studied in this report. There are small differences in the Blue-3 and Blue-1.5 configurations since the Blue-3 optics were designed nearly 2 years ago. However, these small differences do not impact the performance of either system.

Results and Discussion

Spectral Purity: The spectral purity of the three systems is shown in Table 2. As expected for a monochromator system, the spectral purity is quite good for all three. High spectral purity benefits the diffraction experiment by reducing the X-ray scatter, improving signal-tonoise, and reducing the crystal decay from noncharacteristic radiation.

Useable Flux: The useable flux for each system is shown in Table 3 for 0.5, 0.3, 0.2 and 0.1 mm apertures. As the aperture size decreases, and presumably as the sample size decreases, the FR-D Blue-1.5 system provides 2.6 to 4.9 times more flux than the RU-H2R. The FRD Purple-1.5 provides similar gains from 1.9 to 3.9. Even though the FR-D was run at 90% of the power level of the RU-H2R, the smaller focal spot, hence smaller X-ray beam, of the FR-D creates an increase in useable flux. The overall increase in useable flux for all sample sizes with the FR-D indicates that performance improvement is not limited to just small samples.

Profile: Table 4 shows the full width at half maximum of the direct beam at the crystal position for the three systems. The value for the RUH2R Blue-3 system was determined from intensity measurements through a 5ym pinhole scanned across the beam. The beam for the FRD systems was profiled in the vertical and horizontal directions using the moving slit method. The reduced FWHM for the 0.15 mm source as compared to the 0.30 mm source is expected and is consistent with the higher useable flux observed, given the same power loading on the anode. One might expect the Purple-1.5 system to provide the highest useable flux since the beam size is smallest at the focal point. However, this optic has a smaller capture angle as compared to the Blue-1.5 resulting in less flux.

Divergence: Table 4 also provides the divergence data, as measured by determining the FWHM using an R-AXIS IV or R-AXIS IV++ detector at various crystal-to-detector distances. The divergence data for the Blue-3 system is described in the horizontal and vertical direction by two values each, which are different before and after the focal point. Prior to the focal point the divergence is about 1 mR and after the focal point the divergence is about 3 mR.

The FR-D Blue-1.5 and Purple-1.5 systems present divergence plots that match the theoretically calculated quadratic expected for a confocal optic. For these two cases experimental data were fit to a quadratic and the divergence at any point is determined by the derivative of this curve; these formulae are provided in Table 4. A mean divergence is provided for clarity. The Purple-1.5 optic was designed with a shorter focal length, so the observed divergence is higher. The Purple-1.5 system is designed specifically to extract the most data from small samples of relatively short unit cell length (<300 D). The Blue-1.5 configuration is designed to accommodate a wider range of samples, both in size and unit cell length.

Data Collection: In order to compare the performance of the Blue-3 and Purple-1.5 systems described in Table 1, a data set on each was collected on the same frozen lysozyme crystal of dimensions 0.1 mm × 0.l mm × 0.l mm. The crystal was frozen and handled using standard techniques. The data were collected as 180, 0.5°oscillations of 1 minute duration on an R-AXIS IV++ detector.

The overall R(merge) for the two data sets were 0.072 and 0.051, respectively. Plots of R(merge) vs resolution and <I/σ(I)> vs resolution are shown in Figs. 2 and 3. It is clear from both figures that the overall data quality is improved dramatically with the increase in flux on the small sample. In the highest resolution shells the R(merge) drops from 0.23 to 0.17 and the increases from 4.7 to 8.4. This will translate to shorter data collection times for the same quality data set or the ability to collect data on samples that up until now would have been done at synchrotrons.

[1] Yang, C., Courville, A., Ferrara, J. (1999) Acta Crystallographica, **D55**,1681-1689.

Table 1. Configuration Parameters for the systems under study.

System Parameter	RU-H2R Blue-3	FR-D Blue-1.5	FR-D Purple-1.5
Focal spot (mm)	0.3 x 3.0	0.15 x 1.5	0.5 x 1.5
Take off angle (°)	6.0	6.0	6.0
Power (kW)	5.0	4.5	4.5
Source-to-optic distance (mm)	120	120	200
Aperture 1 size (mm)	1.2	0.6	0.6
Source-to-aperture 1 distance (mm)	230	260	340
Aperture 2 size (mm)	0.5	0.5	0.5
Source-to-aperture 1 distance (mm)	370	360	440
Source-to-sample distance (mm)	380	370	450
Source-to-focus distance (mm)	500	500	480

Table 2. Observed spectral purity for the systems under study.

System Radiation Type	RU-H2R Blue-3	FR-D Blue-1.5	FR-D Purple-1.5
CuKα	97.68	98.93	98.20
CuKβ	Not measurable	Not measurable	Not measurable
FeKα	0.22	0.23	0.23
White Radiation	2.10	0.84	1.57

Table 3. Pin-diode readings, corrected for spectral purity, for the system under study through apertures approximating crystals of different sizes.

System Aperture Size (mm)	RU-H2R Blue-3	FR-D Blue-1.5	FR-D Purple-1.5
0.5	12.41	31.95	23.37
0.3	5.69	17.51	16.89
0.2	2.43	9.00	10.01
0.1	0.63	3.07	2.46

Table 4. Observed FWHM, divergence and spectral purity for the direct beam for the systems under study.

System Parameter	RU-H2R Blue-3	FR-D Blue-1.5	FR-D Purple-1.5
Horizontal FWHM (mm)	0.46(1)	0.26(1)	0.19(1)
Vertical FWHM (mm)	0.38(1)	0.28(1)	0.23(1)
Horizontal Divergence (mR)	XTD < 174:1.1(1) XTD > 174: 2.7(1)	$1000*atan(3.8*10^{-6}*XTD+1.4*10^{-3})$ Mean = 2.36(4)	$1000*atan(1.3*10^{-5}*XTD+7.8*10^{-4})$ Mean = 3.8(2)
Vertical Divergence (mR)	XTD < 173: 1.3(1) XTD > 173: 3.1(1)	$1000*atan(3.5*10^{-6}*XTD+1.6*10^{-3})$ Mean = 2.52(5)	$1000*atan(9.6*10^{-6}*XTD+1.8*10^{-3})$ Mean = 4.0(1)

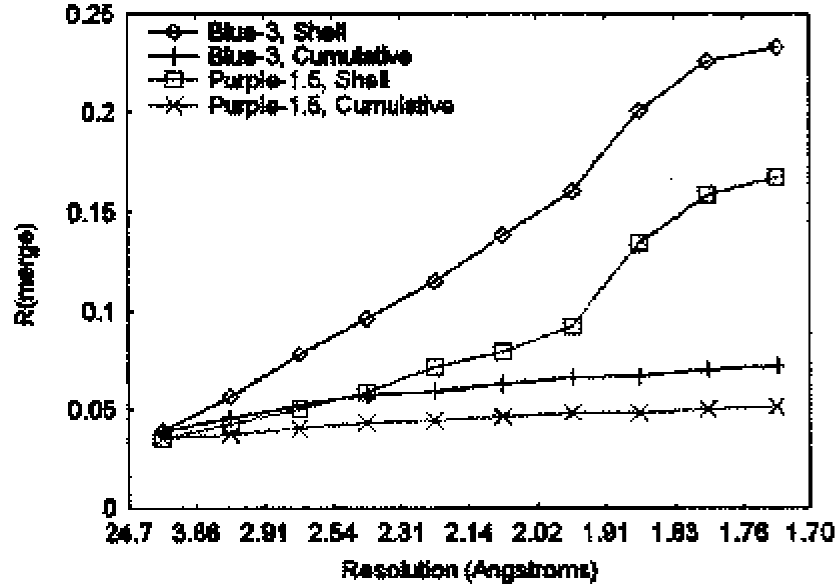

Fig. 2. R(merge) versus resolution for the lysozyme data set for the Blue-3 and Purple-1.5 systems

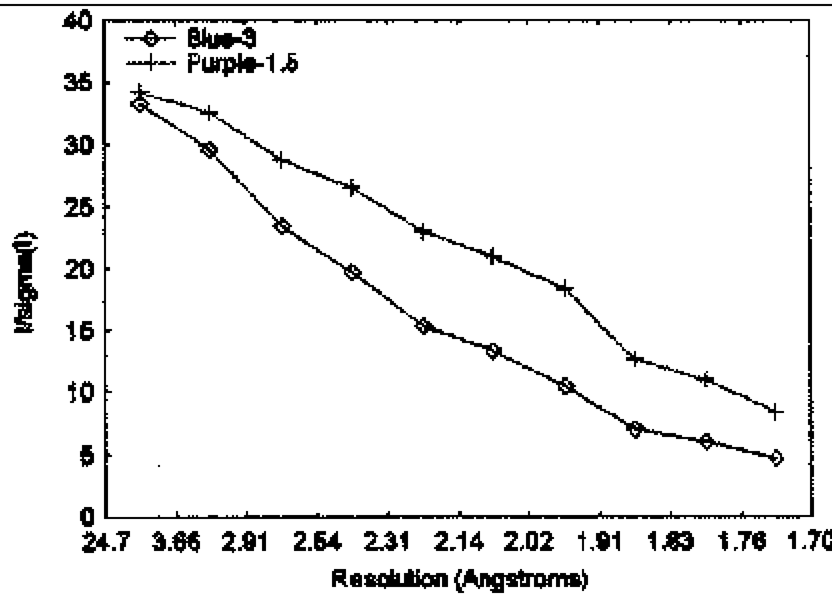

Fig 3. I/σ(I) vs. resolution for the lysozyme data sets for the Blue-3 and Purple-1.5 systems

Conclusion

The FR-D generator provides a significant improvement in beam properties over a standard rotating anode source. The beam size is smaller and more intense allowing data collection on ever smaller samples. The divergence and spectral purity are similar to that observed for the RU-H2R Blue-3 system.

Data collection on a small lysozyme sample is a further indication of the improvement in performance as seen in the downward trend in R(merge), upward trend in I/σ(I) for both the cumulative values and shell values for these metrics.