# Comparative analysis of focusing optics for Mo radiation in small molecule crystallography

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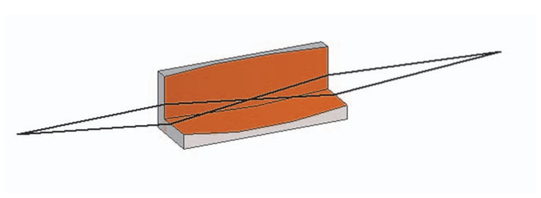
**Abstract**

Montel multilayer optics for small molecule crystallography provide higher brightness compared to standard graphite optics but suffer from lower total flux limiting their applications to small crystal analysis. We will present the advantages of single reflection multilayer optics (FOX2D optics) compared to Montel mirrors when coupled to Mo radiation sources in order to achieve higher flux while keeping the focusing advantages of multilayer optics.
Taking advantage of this optical concept, Xenocs has developed the GeniX Mo HF, a beam delivery system combining a microfocus tube (50W) and a single reflection optic with improved flux properties. In collaboration with STOE, comparative crystallographic data collections have been performed on a IPDS 2T diffractometer on single crystals of different sizes (150 microns and 400 microns). The GeniX Mo HF provides crystallographic performances comparable to standard sealed tube/graphite source optic combination for large single crystal analysis (400 microns) and significant advantages for smaller crystal analysis. The comparative performances and data quality will be detailed.

## Multilayer Focusing Optics for small molecule crystallography

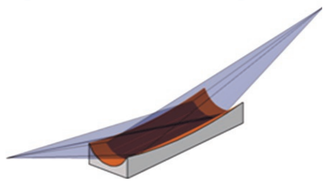
- Montel Mirrors



➔ Two 1D substrates with multilayer coating arranged side by side

- FOX 2D Optics

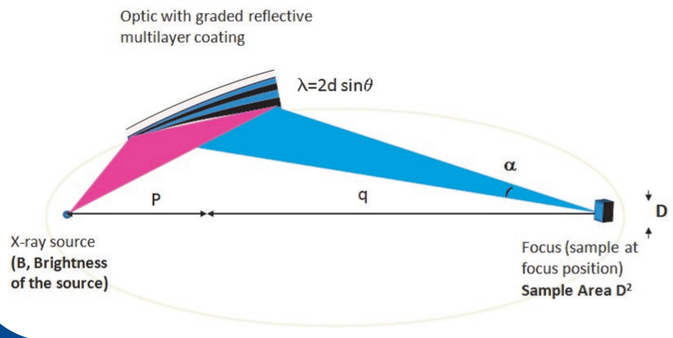
***Single reflection design***



➔ Doubly curved substrate with multilayer coating

## Focusing System performance

- Source-Optic-Sample efficiency




**Useful Flux F within Sample Area :**
**$F = B \times \alpha^2 \times D^2 \times Eff$**
**(for X-ray beam over-illuminating the sample)**

$\alpha^2$ is optic convergence solid angle
$D^2$ is sample area
B is source brightness (ph/s/mm²/sr)
Eff is optic efficiency in the system configuration

**Best compromise is obtained with beam matching sample size**

➔ Optic magnification M=q/p
➔ Optimization of p for coupling efficiency

## GeniX Mo HF test in Small Molecule Crystallography on a STOE IPDS2T system

***Data courtesy of Jens Richter, STOE GmbH, Darmstadt, Germany***

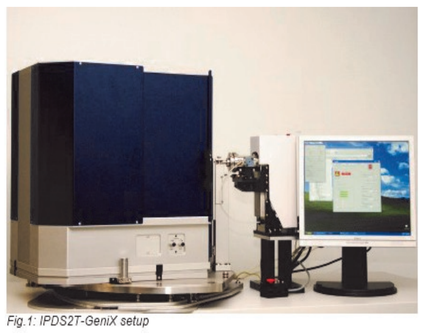


*Fig.1: IPDS2T-GeniX setup*

### Two different Source–Optics Tested

**GeniX Configuration**
50 W Microfocus source
FOX 2D Mo Optic (p=10, q=30)

**Standard Sealed Tube**
2kW (50 kV/40 mA)
Graphite (with 0,5mm capillary)

### On two crystals of different sizes and types

**Large Crystal A:**

| | |
|---|---|
| Crystal type: | Calcium tartrate |
| Crystal System: | orthorhombic |
| Space group: | P 21 21 21 |
| a/Å | 9.2056(5) |
| b/Å | 9.6154(5) |
| c/Å | 10.5550(6) |
| Crystal Size (mm³) : | 0.40 x 0.40 x 0.30 |

**Small Crystal B:**

| | |
|---|---|
| Crystal type: | Potassium tartrate |
| Crystal System: | orthorhombic |
| Space group: | P 21 21 21 |
| a/Å | 7.6022(4) |
| b/Å | 7.7781(4) |
| c/Å | 10.6426(5) |
| Crystal Size (mm³) : | 0.15 x 0.10 x 0.02 |

### Large VS Small crystal : comparative crystallographic data collection quality

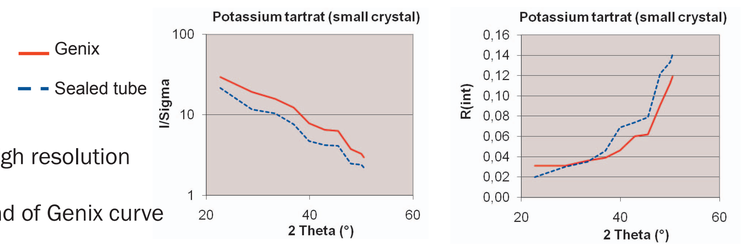
➔ I/Sigma improved by 50% with Genix for small crystal, with no loss of quality for large crystal (I/sigma already large)

| Crystal A: 400*400*300µm³ | GeniX Mo HF | Sealed Tube (0.5 mm collimator) |
|---|---|---|
| Completness of data set [%] | 100 | 100 |
| Redundancy | 9 | 10.3 |
| R(int) | 0.0526 (15387 contr.) | 0.0526 (17567 contr.) |
| R(sigma) | 0.0256 | 0.0177 |
| Avg. I/Sigma (all data) | 32,76 | 44.55 |
| R1 | 0.019 | 0.016 |
| R1(all) | 0.043 | 0.038 |
| wR2 | 0.021 | 0.017 |
| wR2(all) | 0.043 | 0.028 |
| GooF | 0.957 | 1.005 |

| Crystal B: 150*100*20µm³ | GeniX Mo HF | Sealed Tube (0.5 mm collimator) |
|---|---|---|
| Completness of data set [%] | 100 | 100 |
| Redundancy | 13.44 | 13.79 |
| R(int) | 0.0417 (15505 contr.) | 0.0442 (15 978 contr.) |
| R(sigma) | 0.0759 | 0.1263 |
| Avg. I/Sigma (all data) | 11.13 | 7.4 |
| R1 | 0.019 | 0.017 |
| R1(all) | 0.030 | 0.026 |
| wR2 | 0.025 | 0.030 |
| wR2(all) | 0.030 | 0.028 |
| GooF | 0.581 | 0.385 |

### Evolution of crystallographic parameters for the small crystal (crystal B)

Conditions of experiment: Detector @ 100 mm from the crystal. Exposure time 10 min / frame , ω increment = 2°




➔ I/Sigma improved with Genix, even at high resolution

➔ R(int) remains very low (<0.15) and trend of Genix curve leads to higher resolution expectations

## Multilayer optics for Mo radiation

- Multilayer optics coupling efficiency

**Resulting flux outcome from the optic is a convolution of Source intensity distribution (Ss) and acceptance size (Sm) by the multilayer optic**

*$S_m$ is a function of $\Delta\theta$ (width of Bragg reflection of the multilayer optic) and p (source-optic distance) and is expressed as $S_m = p.\Delta\theta$*

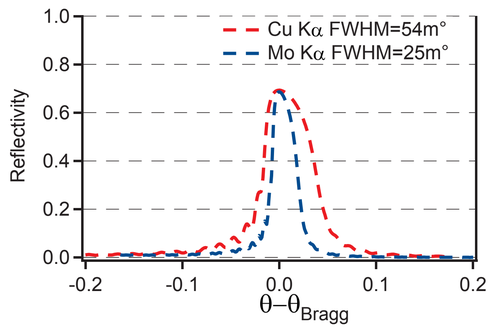
*Sr is resulting from the convolution of both size distribution*

**Efficiency of the coupling is therefore expressed as (gaussian hypothesis)**

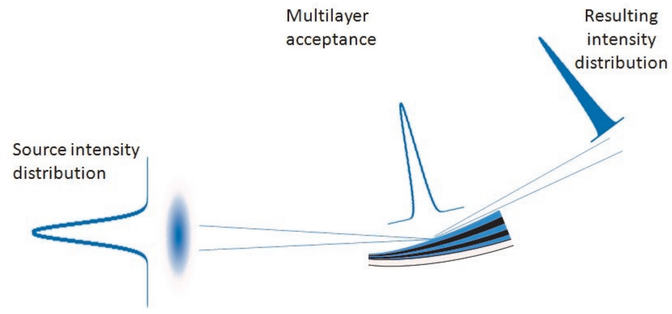
$$Ce = \frac{Sm}{\sqrt{Ss^2 + Sm^2}} \qquad \text{Eq.1}$$

- Comparison of Cu VS Mo rocking curve width

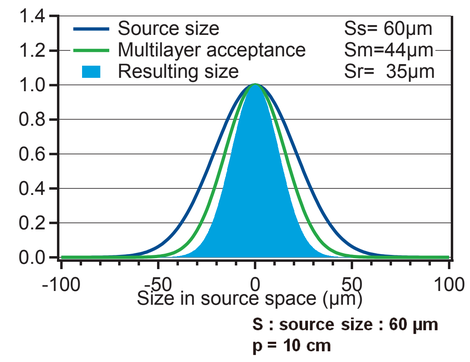
➔ Reduced FWHM for shorter radiation




- Illustration of coupling source, multilayer and resulting intensity distribution




- Intensity after optic depends on source intensity distribution and multilayer acceptance




**S : source size : 60 µm**
**p = 10 cm**

- Comparative coupling efficiency: Montel VS FOX 2D

(simulation for source 60µm, source to optic p= 10cm)

**Montel mirrors**

➔ Source intensity distribution Ss = 60µm
➔ Reflectivity peak: 70 %
➔ With **2 reflections**, equ. 1 needs to be used at **each reflection**
1st reflection: Ss= 60µm, Sm = 44µm
$C_{eff1}$=58%
2nd reflection (here Ss= Sr from 1st reflection)
Ss= 35µm, Sm = 44µm
$C_{eff2}$=78%
➔ Calculated optic efficiency (1st approximation) :
$Eff \approx C_{eff1} * R * C_{eff2} * R$ = 22 %
➔ Calculated optic efficiency (mirror length correction) :
Eff = 15 %

**FOX 2D optics**

➔ Source intensity distribution Ss = 60µm
➔ Reflectivity peak: 70 %
➔ **Only one** reflection
Ss= 60µm, Sm = 44µm
$C_{eff}$=58%
➔ Calculated optic efficiency (1st approximation) :
$Eff \approx C_{eff} * R$=41 %
➔ Calculated optic efficiency (mirror length correction) :
Eff = 32 %

***Single Reflection multilayer optics provide 110% higher flux compared to Montel mirrors for similar geometry and source size***

## X-ray beam characteristics of Mo GeniX solutions

| Source –Optic configuration | Applications | Typical Flux [photons/sec] | FOX 2D p, q [cm] | Focal Spot Size FWHM [µm²] | Divergence mrad² |
|---|---|---|---|---|---|
| **GeniX Mo High Flux** | Small Molecule Crystallography | > 30 x $10^6$ | 10,30 | 250 | 2.8 x 2.4 |
| **GeniX Mo Small spot** | Microdiffraction | > 5 x $10^6$ | 25,25 | 80 x 80 | 4 x 5.3 |

## X-ray beam delivery systems :

- Low power/High brilliance microfocus sources
- efficiently coupled to X-ray multilayer optics

**GeniX System**

## Applications currently covered :

- Protein Crystallography
- Small Molecule
- SAXS
- High Resolution
- High pressure diffraction
- Powder diffraction
- Reflectometry
- And many others …

- **Experimental comparison of Genix Mo High Flux VS standard sealed tube shows 50% improvement of data quality on small crystals, with no sacrifice for large crystals**
- **Comparison of Xenocs FOX2D single reflection VS Montel-type shows X2 flux increase**

**Genix Mo High Flux is therefore ideal for high throughput Small Molecule crystallography**

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