# Focussing Optics for Molybdenum Radiation: An Intense Home Laboratory Source for Small Molecule Crystallography

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## The Problem

Recent developments in synchrotron science have led to small molecule crystallographers being able to study increasingly small crystals at dedicated centralised facilities, such as Station 9.8. The chemical sciences have benefited greatly from these advances in instrumentation, which have in many cases enabled studies to be performed that otherwise would have been impossible. This has led to a large difference in the capabilities of the home laboratory when compared to synchrotron facilities. In addition these centralised facilities are exceedingly expensive to run and maintain and in some cases access to them can be difficult and time consuming to obtain.

It is clear that the small molecule crystallography community requires advances in instrumentation in order to reduce the workload on oversubscribed synchrotron facilities and better screen candidate samples.

## The Solution

The answer is to focus the radiation from the source rather than throw most of it away by using pin holes or a collimator!

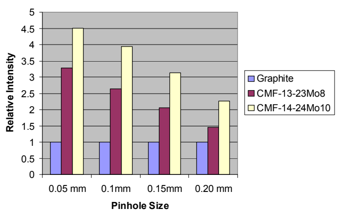
Protein crystallographers do this, but the requirements of small molecule work are different in that a shorter wavelength radiation is necessary (Mo-kα as opposed to Cu-kα). Mo-kα has a lower scattering power than Cu-kα and a much worse efficiency (from the source) than Cu-kα. Moreover, the required Bragg angles are half that of Cu-kα, which reduces the capture angle of the x-rays from the source. Until recently the technology to address this issue has not been available.

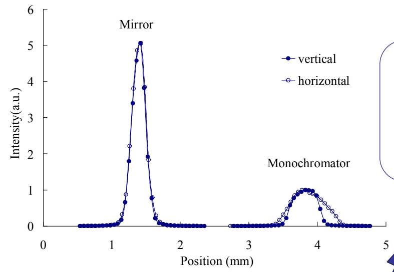
This poster reports the performance of a graded multilayer focussing optic arranged in a side by side geometry and designed specifically for a rotating anode generator with a molybdenum target.

## Design

Ray tracing simulations were performed and indicated that 100mm mirrors were optimum for Mo radiation. A compromise of 0.1mm beam size was decieded on for practicality reasons.

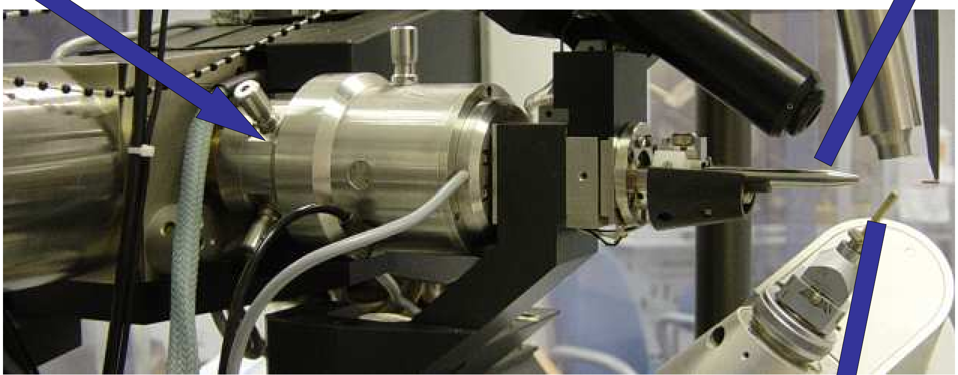
| | 80mm Mirror | 100mm Mirror |
|---|---|---|
| FWHM at focus (mm) | 0.07 | 0.07 |
| FWHM at pinhole (mm) | 0.13 | 0.14 |
| FWHM at sample (mm) | 0.07 | 0.07 |
| FWHM at detector (mm) | 0.26 | 0.33 |
| System effective efficiency | 1.13E-07 | 1.64E-07 |
| Photons / second | 2.82E+07 | 4.11E+07 |
| Divergence (degrees) | 0.24 | 0.31 |

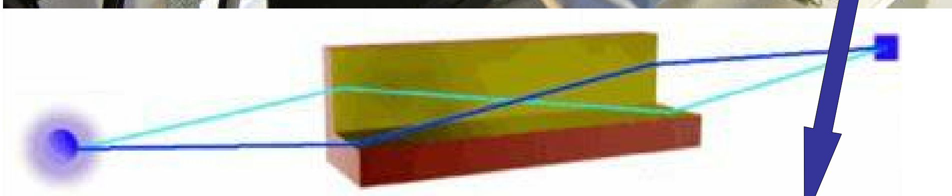


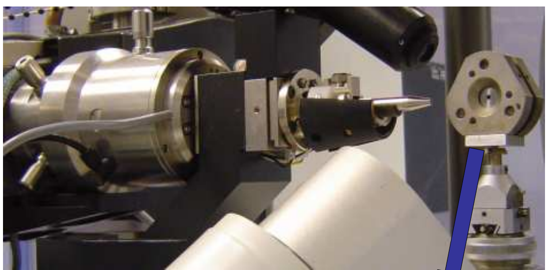




## Beam Profile

'Raw beam' measurements show a five times increase in intensity and a considerable reduction in divergence.





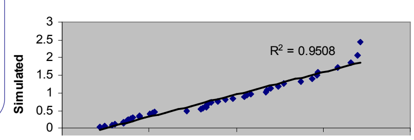


## Comparison Data Collections

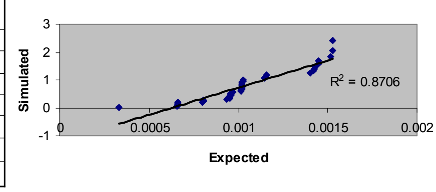
Data collected after the installation of the mirrors shows a marked increase in quality over that collected on the same crystal with a graphite monochromator.

| | Graphite Monochromator | 100mm Confocal Mirrors |
|---|---|---|
| Angular range (degrees) | 343 | 356 |
| Max. theta (degrees) | 27.48 | 27.39 |
| Measured reflections | 19655 | 17853 |
| Independent reflections | 6924 | 6374 |
| Completeness | 95.8% | 88.4% |
| $R_{int}$ | 0.0978 | 0.0464 |
| $R_1$ & $wR_2$ (obs) | 0.0598 / 0.0919 | 0.0388 / 0.0982 |
| $R_1$ & $wR_2$ (all data) | 0.1382 / 0.1098 | 0.0461 / 0.1104 |

Half Normal Probability Plot for Intensity Data

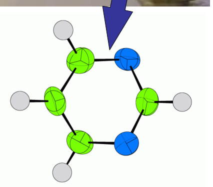



Half Normal Probability Plot for Derived Geometry




## Applications

This apparatus is also allowing us to carry out non routine investigations that are difficult or impossible to perform on a 'normal lab instrument e.g. studies under high pressure.



## Conclusion

Focussing optics are now enabling us to investigate crystals in the home laboratory with sizes of the order of 10-20microns and provide a much more effective screening mechanism for synchrotron candidates. Moreover, for small crystals of slightly more routine dimensions (ca 50-100microns), a 5-fold increase in intensity has a concomitant decrease in collection time, thus increasing the throughput of the instrument.

## Acknowledgements

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