Conventional small molecule diffractometers employing Mo radiation from fixed anode sources with modern area-detectors have proved to be excellent workhorses for the determination of the vast majority of small molecule single crystal structures. There are however a number of times when these techniques prove to be inadequate due to either the crystal being too small or the unit cell too large. Previously we have used the EPSRC synchrotron radiation (SR) source at Daresbury to provide the extra brightness needed to study such crystals.

As an alternative to using SR we have installed an in-house diffractometer fitted with a rotating anode generator with a Cu target. Beam-focusing mirror optics are employed in to maximize X-ray flux at the sample. A large format low demagnification CCD area detector is used for data collection. Results from this machine have been compared to those from a more conventional CCD diffractometer fitted with a fixed Mo target, graphite monochromator and collimator. These show that, for small crystals, the high brightness machine provides data that are an order of magnitude better than those from the conventional machine. Similarly excellent results are obtained for the structure determination of mesomolecular compounds with large unit cells that are difficult or impossible to study using Mo radiation. The determination of absolute structure for organic compounds is made routine. Our results show that the majority of small molecule samples that might otherwise be earmarked for synchrotron study can be successfully studied on a machine of this type.

**Keywords:** SINGLE CRYSTAL MIRROR OPTICS

**DIFFRACTOMETER**

---

A method of time-resolved X-ray diffraction has been proposed. Counts of diffracted X-rays are successively recorded for each time step during a time interval. For each goniometer angle the counting is repeated. It can be employed if the events are repeatable or periodic. Measuring time by the method does not depend on the time step but the ratio 1/(step number). The method realizes precise measurements because of angular scan and is widely applicable not only rocking curves but also line profiles. Rocking curves of pulse heated LiF have been measured by the method. A LiF (222) single crystal has been heated for 5 to 50 msec (3.2 to 32 J/cm²) by a 3 micron nichrom foil. The time step and the time interval were 2 and 2000 msec, respectively and angular step of 2 arcsec. Change of the profiles clearly showed the time evolution of temperature distribution in the crystal.

**Keywords:** TIME RESOLVED, DEPTH RESOLVED, PULSE HEATED LiF

---

Recently Rigaku released a new laboratory X-ray source by combining a micro focus rotating anode X-ray generator (RA-Micro7) and a multilayer mirror, Confocal Max-Flux (CMF) from Osmic. It's focal size is 0.07mm. This generator was developed originally for Cu radiation for protein crystallography but we found this generator is optimal also for Mo radiation when combined with a CMF for Mo. The system was expected to create a highly brilliant Mo radiation converging to 0.1mm in diameter at the focal position. In order to evaluate the actual performance, we attempted to evaluate the overall performance of this system. First, we evaluated the quality of the beam such as the shape, flux, brilliance and energy distribution of the beam. Then using a CCD detector, we collected some data sets with a 0.05mm organic crystal. As a reference, we also collected data on our conventional rotating anode x-ray generator UltraX18. The beam shape produced by an RA-Micro7 and a CMF (MicroMax007) was perfectly round and its brilliance at the focal point was outstanding. Though the exposure time employed during data collection on a MicroMax007 was a half of that on an UltraX18, the resulting data quality was comparable or better. More detailed results will be presented at the meeting.

**KEYWORDS:** MICRO FOCUS LABORATORY SOURCE SMALL MOLECULE

---

A small angle scattering optics of CMF multilayer mirror

Y. Iwasaki K Hoshino T Taguchi

Rigaku Corporation X-Ray Research Laboratory 3-9-12 Matsubara-Akimaga Tokyo 196-8666

We developed small angle scattering optics of the convergence optics which used confocal max flux(cmf) multilayer mirror for incident monochromator. We can get monochromatic x-ray(cu k alpha) of strong intensity with this optics. I show data of x-ray small angle scattering of the ion exchange film which is a material of the fuel cell which we measured with this optics. We discuss data of small angle scattering by a change of construction by the temperature that we were not able to observe with conventional optics.

**Keywords:** SMALL ANGLE SCATTERING POLYMER FUEL CELL