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SAXSess – An Analytical Tool for Nanostructured Materials

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Small-angle X-ray scattering (SAXS) is a well established method for structural investigations in the size regime of 1 nm to 50 nm. With the new laboratory instrument, SAXSess, structural informations can be acquired, such as

(1) Size distribution
(2) Particle shape and internal structure
(3) Surface-to-volume ratio
(4) Degree of crystallinity

One unique feature of this instrument is its ability to simultaneously measure up to wide-angles (of 40°) without the need for realignment works. With that it takes just one experiment to know the particle structure and the phase state of its constituents. Thus, a huge variety of applications can be addressed in quality control and research.

In this presentation we show a few examples of the above mentioned application areas.

Keywords: characterization of materials, macromolecules, aggregates

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Illuminating the Role of Brightness in X-ray Diffraction

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Single crystal diffraction experiments require intense X-ray beams. In a given geometry the photon flux is determined by the beam properties and the crystal properties. Ideally the beam diameter should only be a little bigger than the sample. The maximum divergence is reached when reflections, due to large mosaicity and large lattice constants, start to overlap.

The Brightness (B) of an X-ray source is linked to the divergence (Ω), beam size (A) and flux (Φ) by: $B = \Phi/A\Omega$. This brightness is a constant and can't be changed according to Liouville's theorem. No optic will be able to increase the brightness of the source; it can only lower the brightness by having an efficiency lower than 100%. Today's best quality multilayer optics have an efficiency of about 70%. Thus, given the sample properties and the efficiency of the optic, the only way to increase the flux on the crystal is by increasing the brightness of the source.

Rotating anode generators are the logical X-ray source choice for Structural Biology home laboratories. These generators offer a much higher brightness than sealed tube X-ray sources due to the larger power density applied on the anode. In this paper we show the results of brightness measurements on rotating anode sources with various focal sizes and optics with different efficiency. We give examples of how the electron spot on the anode influences the number of photons on a sample and how this affects the quality of the diffraction experiment.

Keywords: intensity measurement, brightness, X-ray optics

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A Holder for Diffracting Crystals and *Mesophases* Straight from Crystallization Plates

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High throughput crystallization entails miniaturization of the protein+precipitant solution mixture reaching drops of less than 100 nanoliters. Crystals growing in such small volume are difficult to handle and quite often most of crystals grown together in the same drop get injured when scrambling with the loop trying to fish a single one for mounting. This situation is critical when, for example, we just want to check crystals under x-rays to distinguish proteins from salts. Things gets even worse when the crystallization experiment is in *mesophase* where the growing medium is much more viscous than all solutions used in vapour diffusion methods. In order to avoid crystal handling and mounting we designed and constructed a holder to put the plate directly into the diffractometer. The holder is fixed to a x-y-z standard goniometer head and -when mounted on a MAR345 Image Plate detector- a 96-well plate can rotate about 30 degrees. This attachment can hold standard hanging, sitting and micro batch 96-well plates and was proved to be useful for checking crystals directly inside the growing solution. This holder was also useful for screening precipitant solutions that destabilize monoolein-based cubic phases when setting up for membrane protein crystallization assays using the micro-batch method.

Keywords: mesophase, X-ray diffraction, crystallization plates

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Applications of Bragg Backscattering from Crystalline Quartz

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The backscattering silicon single crystals normally used for energy analysis in hard X-ray inelastic scattering suffer from parasitic reflections and gaps in photon energy where no backscattering reflection exists. Sapphire has been proposed as an alternative because its trigonal lattice has lower symmetry than silicon's fcc lattice. The lower symmetry means both that fewer reflections are forbidden and that multi-beam cases are less likely to arise when one approaches a backscattering Bragg reflection. However, crystal quartz, which is also trigonal, has a larger number of backscattering reflections predicted to have energy widths of 6 meV at photon energies between 5 and 12.5 keV, and has peak reflectivities comparable to those of sapphire. Such photons have less energy than those now normally used in X-ray inelastic scattering, but using them would allow scattering at smaller momentum transfers to be explored. Furthermore, some new synchrotron sources are optimized for 10-12 keV photons, for which silicon backscattering analyzers cannot provide energy resolutions below 5 meV.

At present it is not certain if quartz crystals with sufficiently low distortion can be found for use as backscattering analyzers. Therefore, we have measured the energy width of several backscattering reflections in quartz, and have performed X-ray topography on several samples. Though the results do not match those predicted for perfect crystals, meV energy resolutions were attained.

Keywords: high-resolution X-ray diffraction, quartz, inelastic X-ray scattering

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Development of a Real-time Timing-shutter Delay Monitor

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The Structural Biology Center, Sector 19 at the Advanced Photon Source, is a dedicated protein crystallography facility. Conducting successful experiments on low mosaic samples requires attention to all aspects of the experiment, including accurate shutter timing and synchronization. Signalling the timing-shutter to open or close can be synchronized with the scanning motor encoder, but knowing when the shutter actually opens or closes depends upon the delays inherent with the specific shutter. Because timing-shutters may be exercised in excess of 1-2 million cycles during their lifetime, delay times may change as shutter components age.

In order to accurately monitor the opening and closing delay times, we have designed a pin diode array in combination with fluorescence off the timing-shutter blade to monitor shutter delay