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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

times as they occur; the design is based on the existing design for the beam position monitor [1]. We use a Uniblitz x-ray shutter with a PtIr blade located downstream of the beam defining slits. The device is operated in air with He flowing into it from an attached scatter guard.

[1] Alkire R.W., Rosenbaum Gerold, Evans G., *J Sync. Rad.*, 2000, **7**, 61.

Keywords: synchrotron, instrumentation, timing-shutter

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An X-ray Chopper for Time-resolved Crystallography

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A chopper has been developed for time-resolved (pump-probe) x-ray crystallography, with pulse lengths continuously adjustable from DIAMOND single bunch extraction (3µsec) to 45% of rotation time (45msec at 10Hz). Rotation frequency can be DC to 50Hz. This allows access to all time windows from hours to picoseconds. The chopper transmits 0.002% of flux at 25KeV (0.5Å), with improved blocking at lower energy. The chopper can be phase-locked to the synchrotron bunch clock, and includes an on-board digital delay (waveform) generator with 10ns resolution and 16 channels of output for triggering lasers, gating high-speed electronics, etc. The chopper is controlled via an RS232 interface to Windows PC software, with all high-speed electronic processing being performed by a Field Programmable Gate Array (FPGA).

The parameters for chopper design are presented, and the overall mechanical operation, along with the control electronics and logic. Results of initial testing at Daresbury station 9.8 are provided, as well as opening time jitter and accuracy measured to verify single bunch extraction at DIAMOND.

Keywords: time-resolved, pump-probe, synchrotron

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Modeling the IRF of Synchrotron Powder Diffractometers with focusing Optics

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We extend the theory developed by Caglioti, Paoletti, Ricci [1-3] and Sabine [4,5] that provides an analytical description of the Instrumental Resolution Function (IRF) of single crystal and powder spectrometers consisting of collimators and crystals by including the effect of collimating and refocusing mirrors. Two cases are explicitly considered: the case when both collimating and refocusing mirrors are bent to a parabolic shape and the case when the collimating mirror is bent to a parabolic shape and the refocusing mirror is flat. The effect of isotropic sample size is also considered as a possible additional contribution to the peak width.

Experimental IRFs collected at the Swiss Light Source Materials Science beamline powder diffractometer at different photon energies and in both optical configurations are modeled by our analytical expressions and the agreement found is good. All experimental tests are performed using the $Na_2Ca_3Al_2F_{14}$ (NAC) standard powder.

[1] Caglioti G., Paoletti A., Ricci F. P., *Nucl. Instrum.*, 1958, **3**, 223. [2] Caglioti G., Paoletti A., Ricci F. P., *Nucl. Instrum. Methods*, 1960, **9**, 195. [3] Caglioti G., Paoletti A., Ricci F. P., *Nucl. Instrum. Methods*, 1962, **15**, 155. [4] Sabine T. M., *J. Appl. Cryst*., 1987, **20**, 23. [5] Sabine T. M., *J. Appl. Cryst.*, 1987, **20**, 173.

Keywords: high-resolution X-ray powder diffraction, synchrotron X-ray instrumentation, modeling

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Microcrystal X-ray Analyses using LTV Camera at SPring-8 BL02B1 Beamline

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Single crystal X-ray analysis is conventional and powerful tool in order to explore new functional materials and understand those chemical and/or physical properties. However, the single crystal analyses are frequently encountered difficulties because of extremely small sizes of the crystal specimens.

High intense synchrotron radiation should enable us to perform the crystal structure analyses of such very small crystals with several micrometers. As the first step of developing the technique of microcrystal structure analysis, we tried to do the single crystal structure analyses for small organic compounds using the low-temperature vacuum X-ray camera installed at SPring-8 BL02B1 beamline.

Cytidine crystal with dimensions 6.7 x 5.0 x 3.3 μm^3 was used for measurement. Although the crystal structure could be solved by the direct method, all the non-hydrogen atoms were refined isotropically and hydrogen atoms located geometrically.

The structure analysis of chiral C60-fullerene *cis*-3 bisadducts crystal with dimensions 100 x 75 x 2 μm^3 was also curried out. The crystal structure could be solved by the direct method and all the non-hydrogen atoms were refined isotropically. The absolute configurations were first established by combination of this study and CD studies.

Keywords: single crystal, small crystals, synchrotron X-ray diffraction

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The Phase 1 Macromolecular Crystallography Beamlines on Diamond

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Diamond is the new 3rd generation synchrotron source being built in the UK just south of Oxford. Seven beamlines are funded as part of Phase 1 which includes 3 beamlines for macromolecular crystallography (MX). The beamlines will used radiation from 3 in-vacuum undulators; each will be canted to allow a second undulator to be placed in the straight to provide radiation for a side station.

The beamlines will provide radiation tunable over a wavelength range of 0.5A – 2.5A. Significant emphasis is being placed on the automation of the beamline in terms of both hardware and software. Each of the beamlines will have diagnostics to allow the beam to be monitored remotely. The experimental station will be equipped with high quality crystal viewing systems and robotic sample changers to enable automated mounting for both cryocooled and room temperature capillary mounted samples. One of the beamlines will also include facilities for biological containment at Category 3 level at the experiment. This facility will open up opportunities for carrying out exciting structural work that it is currently very difficult to carry out due to the restrictions imposed by the safety requirements associated with working with biological pathogens.

These beamlines are scheduled to be commissioned in 2006 and will come on-line for users in 2007.

Keywords: macromolecular synchrotron X-ray crystallography, crystallography instrumentation synchrotron radiation, automation