resolved X-ray diffraction

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Shutter-less continuous rotation data collection from protein crystals with the X-ray CMOS detector

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The fine phi-sliced oscillation method is expected to be useful for high S/N data collection in protein crystallography. However, a small oscillation step increases total number of diffraction images and the experiment time gets longer due to the readout time of detectors. In order to enable efficient data collection with the fine phi-sliced method, we proposed shutter-less continuous data collection using an X-ray CMOS detector. In this method, diffraction images are captured by the X-ray CMOS detector with a constant frame rate, as rotating crystal in a constant speed. The shutter is kept open during data collection. The characteristic feature of the X-ray CMOS detector is rapid readout speed, and so dead time due to readout is negligible. We have been developing the X-ray CMOS detector suitable for protein crystallography in collaboration with Hamamatsu Photonics K.K. (Japan). The performance of our data collection method was examined at SPring-8 protein crystallography beamlines. Comparison with the conventional coarse oscillation method with a CCD detector demonstrated that the data processing statistics was significantly improved by this method. We also successfully determined protein structures with MAD and SAD phasing using diffraction data recorded with this method. Our results demonstrated that the shutter-less continuous rotation method with the X-ray CMOS detector has a promising potential in protein crystallography.

Keywords: protein cryocrystallography, detector development, data collection methods

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Novel pixel detector for in-house XRD applications

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A novel pixel detector, namely PILATUS 100K, has been developed at the Paul Scherrer Institut (PSI). It is constructed using the state-of-art semiconductor technology and demonstrates superb performance. Its single photon counting, extremely low background, very short read-out time and ultra-high count rate features are not like the other conventional area detectors. The PILATUS detector was initially designed for macromolecule study at synchrotron facilities. However, the PILATUS 100K can be used with in-house XRD system. Some of the in-house XRD application results will be shown.

Keywords: hybrid pixcel detector, XRD, SAXS

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Protein diffraction experiments with Atlas CCD detector

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A combination of an enhanced Cu x-ray source and of a recently acquired Atlas single chip CCD detector brings new possibilities for in-house x-ray diffraction experiments on protein samples. Introductory measurements and comparative studies regarding the performance of the CCD detector with high sensitivity, dynamic range and fast readout were performed with protein samples such as xylanase from Trichoderma reesei. Diffraction data sets of high quality were collected, including those of human CD69 receptor and other study targets. Diffraction data were processed by alternative methods. The results suggest that the Gemini Enhanced Ultra diffractometer with the Atlas CCD detector offers a viable option for in-house diffraction experiments and characterisation of protein samples before synchrotron experiments. Moreover, macromolecular diffraction data with the total Rint < 0.03 up to diffraction limit 2.0 Å can be achieved. Data processing was performed with use of the instrument software Crysalis as well as with the standard protein crystallographic software Mosflm and Scala.
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Keywords: X-ray data collection, CCD detectors, protein crystallography applications

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The XPAD3 hybrid pixel detector applications

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The 3rd generation synchrotron sources have been a major progress in materials sciences. The hybrid pixel detectors have been developed to take a full profit of the intense monochromatic X-ray beam provided by these sources. Following the previous XPAD detectors [1] design the XPAD3 hybrid pixel detectors has been designed with a pixel size of 0.130 mm x 0.130 mm[2]. First detectors using wide Si sensors of 15 mm x 76 mm, 0.500 mm thick, are under assembly, they consist in 8 modules of 7 chips. Some others detectors have been realized using CdTe sensors and a dedicated test board allowing to connect 2 chips to the monolithic CdTe sensors [3]. This poster will report on tests carried out on single chip and single module Si detectors and CdTe detector. Using Si sensors, the XPAD3 chip can be used from low energy (4keV) up to 25 keV where the detector efficiency becomes too weak. The CdTe detector was designed to improve the efficiency at high energy and to allow 60 keV X ray to be used, but it can be still used at low energy and data have been

collected with it at 8 keV. Within the argument favoring the hybrid pixel detectors, one is related to its point spread function which is nearly limited by the pixel size. Data have been collected on the Small Angle X ray Scattering camera of BM2/ESRF using the same setting with XPAD3 and the beamline CCD camera show that not only the hybrid pixel detector allow higher count rate but it allows to measure weaker signals.

1 Basolo S. et al., J. Synchrotron. Rad, 2007
2 Pangaud et al. NIM A, 2008, in press
3 Basolo S. et al. , NIM A 2008, in press

Keywords: detector development, CCD detectors, SAXS

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More signal, less noise : Making good use of bright sources & fast detectors

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With the availability of in-house high-brilliance X-ray sources, highly reflective X-ray optics and low-noise, fast read-out detectors we have the tools available to screen protein crystals and often to collect complete data sets in the home laboratory. For CCD detectors the read out time is in the order of seconds. New types of high-speed detectors with zero dead-time are now on the horizon. For in-house use we have recently released the ÅXIOM 200, a gas-filled Microgap detector. This is a high speed photon-counting detector with no intrinsic noise. The zero read-time allows shutter-free data collection and infinitely fine slicing. The availability of these new properties opens avenues for new protocols for crystal screening and data collection, which exploit the benefits of this new detector. By using these new protocols it is possible to get much more accurate data then would be obtained with traditional detectors and data collection methods. The new data collection method can lead to a dramatic rise in signal to noise ratio, with the same amount of X-ray photons falling on the crystal. This increases the chances of a structure solution using SAD phasing, as the solution of the sub-structure will be more straightforward and solvent flattening will more rapidly converge.

Keywords: high speed detectors, accurate intensity data collection, SAD phasing

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DATAVIEW: A new post processing analysis tool

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CCD detectors have become the standard in modern X-ray laboratories. The use of 'black box' methodologies (integration) to convert diffraction images into discrete hkl's and intensities, has led to the possibility of systematic errors creeping into any given dataset. Multi-scan absorption techniques such as SADABS[1], given sufficient redundancy, smooth out random errors and can potentially highlight the more significant systematic errors, such as an incorrect masking of the primary beamstop. Potentially far more damaging are the possibilities of inaccurate detector corrections being applied to the data during the integration phase of data processing. These corrections, which are necessarily machine dependant, may require modification after initial instrument installation. Analysis of the effects of these corrections on real data can in principle be conducted relatively easily, although can be time consuming. Data will be presented demonstrating a new program (DATAVIEW[2]) that reads raw integrated diffraction data and presents graphical representations of the internal data integrity. The output will also provide a suggested improvement to the data treatment during integration, highlighting potential corrections that may not have been applied correctly. DATAVIEW currently operates on Bruker .raw data but could easily be converted to read multiple data formats.

1. SADABS, Multi-scan absorption correction, Sheldrick, G.M. (2003)
2. DATAVIEW, Post Processing Data Analysis Tool, Probert, M.R. (2008)

Keywords: accurate measurement, data processing optimization, X-ray diffraction data

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A hyperquenching tool

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It has been clearly elucidated by Warkentin, M. et al that the removal of the cold gas layer above liquid nitrogen in a dewar dramatically increases the cooling rate of a protein crystal (Warkentin, M., Berejnov, V., Husseini, N. and Thorne, R. "Hyperquenching for protein cryocrystallography" J. Appl. Cryst. (2006). 39, 805-811) . By removing the cold gas layer, one can not only raise the success rate but also reduce concentrations of cryoprotectant therefore minimize damages to crystals caused by changes in osmotic pressure. Being stimulated with this eye-opening study, we created a dewar having a simple mechanism to remove the cold gas layer. It consists of a dewar and a fan and the cold gas layer is suck in to the sideway. We have done a simple measurement and observed rising temperature from -175 to 10 degs. ca. 5mm above the surface of liguid nitrogen. We will perform additional experiments to confirm the effect of the device by the conference.

Keywords: hyperquenching, cryo-crystallography, protein crystal

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In situ crystallizations of Cl and Br substituted anilines and its intermolecular interactions

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In situ cryocrystallization of a liquid, the subsequent structure determination and its study of intermolecular interactions has emerged as an area of contemporary interest. *In Situ* crystallization of fluoro substituted anilines and evaluation of the variability in halogenated trifluoroacetophenones [1] clearly bring out the importance of interactions generated by fluorine in crystalline