

Poster Sessions

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CrysAlis^{Pro}, the software for controlling data collection and data reduction Agilent's single crystal X-ray diffractometers, contains easy to use features for variable temperature and variable wavelength studies.

The results presented here show how the many multi-temperature experiments identify a clear phase transition of a spin crossover compound during the ramping. The Cryojet (manufactured by Oxford Instruments) was used to study the compound at 5° intervals and controlled to within ±0.1° from room temperature to 100K.

An example of the variable temperature software is shown in the figure below.

Temperature	Slit	Sample	Heater	On/Off	Auto Vent	Motor
295.0	5.0	7.0	ON	YES	YES	YES
292.0	5.0	7.0	ON	YES	YES	YES
289.0	5.0	7.0	ON	YES	YES	YES
286.0	7.0	9.0	ON	YES	YES	YES
283.0	5.0	7.0	ON	YES	YES	YES

During the poster session, further information and a demo of the software will be available.

Keywords: Variable Temperature, Small Molecule, CrysAlisPro

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I04-1, a fixed wavelength protein crystallography station in its first year of operations

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Diamond Light Source is the third generation synchrotron light source in the UK, producing an electron beam energy of 3 GeV aiming at a beam current of 300 mA. Four macromolecular crystallography (MX) beamlines I02, I03, I04 and I24, tunable in the energy range of 5 to 25 keV, are already operational and I23, the long wavelength MX beamline is in design stage.

I04-1, the last phase II macromolecular crystallography beamline, entered its user operation program at the beginning of April 2010 and is fully operational since April 2011. The beamline was designed to be highly reliable and the very stable beam is producing high quality data with structures starting to be deposited in the protein databank (PDB). The experimental end-station is equipped with the MD2 diffractometer and a single-photon counting Pilatus 2M detector, running at 30Hz, which allows high throughput data collection and features such as fast grid and helical X-ray scanning of the samples. In combination with the CATS sample changer robot, an average of ten datasets per hour can be routinely achieved. Further developments of the robot involve speeding

up the exchange process and *in-situ* plate screening capability. A set of software pipelines ranging from data collection strategy, diffraction image visualization, streamlined data processing, structure solution and difference map calculation is in place to meet the high throughput needs of academic and industrial researchers studying ligand binding. Finally, with the wavelength fixed to 0.92 Å (bromine K-edge), selenium (Se-) and bromine (Br-) single anomalous diffraction (SAD) experiments were also successfully performed.

Keywords: high_throughput_crystallography, Pilatus_2M, SAD_experiment

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In-situ Crystallisation Screening and Data Collection on Microfocus Beamline I24

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Despite significant progress in high-throughput methods within macromolecular crystallography, the production of diffraction quality crystals remains a major bottleneck. Effort in a crystallization trial can be guided with feedback on the nature and quality of early crystallisation 'hits'. Using a dedicated goniometer installed on the Diamond beamline I24, crystals can be studied directly in their crystallization plates. Valuable time is saved and manual intervention minimised with the removal of the requirement to loop and freeze samples individually. The intense and flexible microfocus beam of I24 allows weakly diffracting samples, such as membrane proteins and complexes, to be assessed with maximal signal-to-noise against the background scatter from the plate medium. In combination with novel methods in data merging, we demonstrate the ability to solve structures entirely *in-situ*, illustrating the potential of the technique to facilitate the sequence to structure pipeline.

Keywords: crystallization, screening, microbeam

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Rapid shutterless data acquisition with a frame transfer CCD

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For CCD detectors, the speed of synchrotron single crystal data acquisition is usually limited by the readout time of the detector and the mechanical synchronization of shutter with rotation motor. To avoid these complexities and obtain faster data collection rates we have tried using a shutterless acquisition with continuous rotation of the crystal. This method is already used with Pilatus detectors and we have developed a version for a frame transfer CCD camera [1] at beamline ID11 at the ESRF. In this mode the camera reads out one image of x-ray data while the next is exposed. The frame transfer is a rapid image shift (4.2 ms) which switches buffers to allow the process to continue. During the continuous rotation fast electronic hardware is used to record the angular encoder positions and times when frame transfers begin and end. Our Frelon2K [1] camera can provide up to 26 images per second at full resolution with 1024x2048 pixels in this mode, and

even greater frame rates if hardware binning is used.

With this new mode of operation we are able to obtain ultra-fine sliced data (0.002°) on reasonable time scale ($3^\circ/\text{min}$). This means that rocking curve evolution can be mapped with high resolution while cooling through a phase transition. Fine slicing also offers a novel method for wavelength calibration by measuring many Friedel pairs from a standard granular powder material, such as NIST LaB₆.

At the price of smearing some reflections during the frame transfer, single crystal data are also available much more quickly than before. Intensity errors due to frame transfer depend on the rocking width of the crystal compared to the angular widths of the integration and transfer steps. Very sharp reflections that arrive during transfer may be completely corrupted so that these should be identified and removed during integration and scaling. For broader rocking curves, particularly where reflections are present on several frames, the main concern is that weak peaks could be contaminated by smearing from strong peaks. In practice these problems are not serious, as demonstrated by the commissioning studies that will be presented.

[1] J. C. Labiche et al, *Rev. Sci. Instrum.* **2007**, *78*, 091301.

Keywords: Experimental, Fast, Detector.

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Automation and remote control at GM/CA CAT at the APS

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GM/CA CAT, sector 23 at the APS, encompasses two insertion device beamlines and one bend magnet beamline. The X-ray sources for the insertion device beamlines are a pair of canted undulators. All of the beamlines are controlled via a Graphical User Interface (GUI) based on the tab-style organization and presentation of SSRL's BluIce. The GM/CA-CAT implementation (JBluIce-EPICS), which communicates with the hardware through lower level EPICS control software, has evolved to include a variety of features that exploit the small beam size and divergence properties of this 3rd generation source.

In terms of automation, experimenters operate the beamlines with the same GUI whether they are using local control (sitting at the beamline) or remote control (anywhere with a valid IP). Remote access is directed through NX or Teamviewer servers to the beamline computers. This has allowed the users to gain experience in person then smoothly transition to remote control. The system has grown to include the necessary indicators for the synchrotron ring status and controls for the shutters, automounter and mini-beam. The automounters are modified versions of the Berkeley/ALS robots. These are pneumatically controlled systems coupled to motor-controlled Dewars that efficiently deliver samples from the Dewar to the goniostat. Hardware improvements to the pin base sensors and collets improved robot reliability and led to increasing user demand. All of the necessary coordination of commands to control the selection, mounting, loop and crystal centering, washing and annealing are housed in the Screening Tab or a Tools sub-Tab of the Sample Tab of JBluIce-EPICS. Future developments in the automation include the commissioning of a new Cartesian Robot in collaboration with T. Earnest and C. Cork, Lawrence Berkeley National Lab.

Two features in the automation of the data collection, the Raster Tab

and vector collect feature, are continually improving in the JBluIce-EPICS system and take advantage of the micron-sized mini-beams (5, 10, and 20 μm). At present the mini-beams are produced by accurate placement of the appropriate sized pinhole to restrict a larger focused beam. The system has evolved from the use of a single collimator to a single-button selection from a uni-body quad collimator. The tool has spawned the development of automated 2D rastering techniques (Raster Tab) to locate small or hidden crystals or better diffracting regions in larger crystals. For data collection, a feature to move the sample along a 3D vector has been incorporated into the Collect Tab. Improvements for a seamless transition from regions of interest identified in the Raster Tab and the expansion of options in the vector collect mode are currently underway. Here we will present a status report on automation and remote control applications available at the GM/CA CAT beamlines.

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Keywords: automation, macromolecular, synchrotron

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Automated *in-situ* diffraction screening at beamline X06DA at the swiss light source

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X06DA is the third macromolecular crystallography beamline at the Swiss Light Source. It has been designed to fulfill the requirements of both academic and industrial users. To achieve maximum efficiency, high degree of automation was implemented from the optics to the experimental environment. A Bartels dual channel cut monochromator (DCCM) ensures rapid energy changes (6 – 18 keV) with a true fixed exit. The obtained X-ray beam has a focal spot size of 80 x 45 microns at the sample position and a total photon flux of 51011 photons/sec at 12.4 keV. The mini-hutch end-station allows both rapid manual mounting and robotic sample exchange.

In addition, a crystallization facility, directly adjacent to the X06DA mini-hutch, has been implemented. Crystallization experiments are performed using nano-dispensing robots and drops inspection is done via an automated imaging system. The unique feature of this facility is the possibility to test the crystals for diffraction directly in the crystallization plates (in situ screening) by transferring them from the crystal hotel to the mini-hutch in an automated manner. Without any manipulation to the crystals, this gives users a rapid feedback on important parameters such as diffraction limit, anisotropy, cell parameters or mosaicity, and aids to prioritize subsequent optimization steps. Moreover, users are welcome to bring any kind of SBS standard crystallization containers, including microfluidic chips and the CrystalHarpTM which yield a particularly low background in the diffraction image.

First results obtained at the crystallization facility and future improvements will be presented. Other methodological developments such as a new type of multi-axis goniometer and phasing with weak anomalous scatterers will be described as well.

Keyword: Beamline automation, *in-situ* Diffraction Screening, crystallization