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Carbon nanotubes are nanometer sized channels that have a very high potential for applications such as selective chemical filtration or water desalination. For such purposes, large membranes of parallel nanotubes, where all metal-based catalyst particles potentially obstructing nanotubes have been removed, are needed.

An original experimental setup based on X-ray scattering and fluorescence has been designed to characterize membranes of arbitrarily large surface (cm<sup>2</sup> to m<sup>2</sup>) [1]. We are able to determine quantitatively the nanotube alignment in the membrane, the density of nanotubes and to check for the removal of metal-based nanoparticles. We will present the set-up, the models we developed to analyze experimental data and some results obtained on the set-up.

The set-up/modeling can also be adapted to others membranes than those of nanotubes, using small or wide angle scattering depending on the membrane composition.

It should be underlined that although the analysis requires complete orientational modeling within the framework of X-ray scattering theory, the set-up is designed for a non-specialist and could be used for instance on an industrial production line, automatic fitting of the data providing one with two-dimensional mappings of the density, chemical composition and nanotube orientation in direct space (figure).

[1] M. Huard, J. Cambedouzou, D. Petermann, P. Joly, G. Guillier and P. Launois *CNRS patent*, December 2010.

**Keywords:** X-ray, Diffraction, membrane

## MS13.P04

*Acta Cryst.* (2011) A67, C277

### JBluIce-EPICS beamline control system for macromolecular crystallography

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The report overviews the open-source JBluIce-EPICS control system [1] developed for the trio of highly successful GM/CA-CAT macromolecular crystallography beamlines in Sector 23 at the Advanced Photon Source. To make the control system fast, robust, intuitive for users and easily adaptable to new beamline developments, several technical solutions were implemented. First, the system consists of only two software layers, the EPICS backend providing robust control of distributed beamline hardware, and multiple graphical user interface (GUI) clients powered by Java Eclipse RCP. This provides the GUI with direct access to any advanced hardware features, such as on-the-fly scanning capabilities of motion controllers, and it is easy to add new features. Second, the GUI was chosen to conform to the task-oriented philosophy and to mimic the look and feel of the successful SSRL BluIce. Most users become proficient with the software within minutes. Third, JBluIce clients are designed to operate in parallel with other beamline controls, thus streamlining such staff tasks as beamline preparation and maintenance, operations auditing and user assistance. Further, JBluIce-EPICS deploys multiple plugins that can be written in any programming language, thus involving more staff

in the development.

From the users' prospective, JBluIce provides: one-click change between 5, 10 and 20 µm beam sizes; one-click beamline energy change that may involve switching undulator harmonics, mirror lanes and beam realignment; automated diffraction rastering over multiple user-defined polygon shapes for finding small crystals and 'sweet' spots on poorly diffracting crystals [2], data collection along a vector [2]; automated on-the-fly fluorescent rastering with a shuttle scanning option, a faster and lower-irradiation compliment to the diffraction rastering [3]; fully automated fluorescence measurements for MAD that include signal optimization, fast on-the-fly energy scanning and automated adaption of scan range to chemical shifts [3]; fly-scan minibeam realignment; automated loop and crystal centering; controls for sample automounter; automated crystal screening; data collection auditing; remote access and more.

[1] S. Stepanov, O. Makarov, M. Hilgart, S.B. Pothineni, A. Urakhchin, S. Devarapalli, D.Yoder, M.Becker, C.Ogata, R. Sanishvili, N. Venugopalan, J.L.Smith, R.F.Fischetti *Acta Cryst.* **2011**, D67, 176-188. [2] M. Hilgart, R. Sanishvili, C. Ogata, M. Becker, N. Venugopalan, S. Stepanov, O.Makarov, J.L.Smith, R.F. Fischetti *J.Synch.Rad.* **2011**, submitted. [3] S. Stepanov, M.Hilgart, D.Yoder, O.Makarov, M.Becker, R.Sanishvili, C.Ogata, N.Venugopalan, D.Aragão, M.Caffrey *Acta Cryst.* **2011**, D67, submitted.

**Keywords:** X-ray, biocrystallography, automation

## MS13.P05

*Acta Cryst.* (2011) A67, C277

### Using GDA for novel data collection methods

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Generic Data Acquisition (GDA, [www.opengda.org](http://www.opengda.org)) is the Java-based data acquisition software developed and used at the Diamond Light Source, the third generation synchrotron light source in the UK. GDA is designed to have a generic and flexible client-server architecture that has proven itself by running on almost all beamlines at Diamond and is now in use at several other synchrotrons.

Graphical user interfaces in the GDA client are controlled with intuitive motions to set up experiments. Standard oscillation experiments with crystals at a single position are now routine. In addition to the standard experiment, we have implemented automated techniques that are useful for evaluating crystals or larger sample holder (grid scan) or for collecting data sets along a three-dimensional crystal (line scan). Aided by the development of rapid detectors and suitable sample stages that allow continuous data collection during scans, these techniques have become even more powerful tools for examining samples in ways that would have been too tedious before their automation. The GDA client also aids in evaluation of the results, with feedback from external software presented graphically to the user. This immediate feedback and automation of the experiment are two ways that the beamline control software can contribute to improving the data collected at beamlines.

**Keywords:** GDA, software, data\_acquisition

## MS13.P06

*Acta Cryst.* (2011) A67, C277-C278

### CrysAlis<sup>Pro</sup>: facilitating variable temperature experiments

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## Poster Sessions

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CrysAlisPro, 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 Size (mm)	Sample Size (mm)	Heater	On/Off	Auto Vent	Total Time	Make room
295.0	5.0	7.0	ON	YES	YES	15.0	YES
292.0	5.0	7.0	ON	YES	YES	15.0	YES
289.0	5.0	7.0	ON	YES	YES	15.0	YES
286.0	7.0	9.0	ON	YES	YES	15.0	YES
283.0	5.0	7.0	ON	YES	YES	15.0	YES

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

**Keywords:** Variable Temperature, Small Molecule, CrysAlisPro

### MS13.P07

*Acta Cryst.* (2011) A67, C278

#### 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

### MS13.P08

*Acta Cryst.* (2011) A67, C278

#### *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

### MS13.P09

*Acta Cryst.* (2011) A67, C278-C279

#### 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