MS01-P03

Serial synchrotron crystallography at EMBL PETRA III beamline P14

Johanna Hakanpää[1], Gleb Bourenkov[1], Ivars Karpics[1], Guillaume Pompidor[1], Isabel Bento[1], Thomas Schneider[1]

1. European Molecular Biology Laboratory, Hamburg, Germany

email: johanna@embl-hamburg.de

Serial synchrotron crystallography (SSX) combines X-ray images taken from randomly oriented crystals, passed through the beam using diverse delivery methods, into a single dataset. The method requires a high-brilliance synchrotron source with a beam size similar to the sample size, an appropriate sample delivery method, a detector with sufficient frame rate and a data processing pipeline. SSX can act as a pre-screening method for XFEL experiments or a standalone experiment when crystal growth to larger size cannot be achieved, e.g. in vivo grown crystals. Proof of principle experiments (1) have shown the feasibility of the method.

Our SSX setup at the EMBL beamline P14 (PETRA III, DESY Hamburg) utilizes the tools of conventional crystallography, minimizing setup time and sample consumption. In situ experiments can be done in CrystalDirectTM plates, also in meso. Cryo-samples are mounted in loops or harvested from plates with the CrystalDirectTM Harvester. Generally, the sample size is a few microns. Data collection runs as series of helical line scans, typically a dataset is collected in a few minutes, depending on the size of the region of interest. Progression of the data collection is monitored throughout the experiment as on-the-fly calculated heat map, displaying the diffraction scores as estimated by program DOZOR (2).

The acquired diffraction images are sorted and bunched into sub datasets according to the DOZOR score. The sub datasets are processed in parallel as small rotation datasets with XDS and scaled using XSCALE. We have demonstrated the feasibility of the pipeline using 5-micron lysozyme crystals as test objects. Using an Eiger4M detector, a data set of 65120 images was collected at P14 in 3 minutes as an in situ experiment in a CrystalDirectTM plate. About 2000 sub datasets, each containing 5-10 diffraction images, were integrated and scaled to yield complete data to 1.7 Å resolution. The structure could be solved by molecular replacement and electron density maps were of good quality.

We have established protocols for sample delivery, data collection and processing, making the pipeline feasible to use without previous experience in serial crystallography. Alternating between serial and conventional data collections is user controlled. Beamtime for experiments at P14 can be applied for through the EMBL user program at https://smis.embl-hamburg.de.

References

[1] Gati G., Bourenkov G., Klinge M., Rehders D., Stellato F., Oberthür D., Yefanov O., Sommer B.P., Mogk S., Duszenko M., Betzel C., Schneider T.R. , Chapman H.N., Redeckec L. (2014) IUCr J. 1 87-94

[2] Popov, A. N. & Bourenkov, G. P. (2015). DOZOR. European Synchrotron Radiation Facility, Grenoble, France

Keywords: serial synchrotron crystallography

MS01-P04

Design of BL06-XAIRA, the new microfocus beamline for MX at ALBA

Damià Garriga[1], Nahikari González[1], Jordi Marcos[1], Josep Campmany[1], Carles Colldelram[1], Josep Nicolás[1], Judith Juanhuix[1],

1. ALBA synchrotron, CELLS, Cerdanyola del Vallès, Spain

email: dgarriga@cells.es

ALBA is a third generation 3-GeV synchrotron radiation facility built near Barcelona servicing worldwide academic and industrial users since 2012. It currently hosts eight operating beamlines, including one dedicated to Macromolecular Crystallography (MX), BL13-XALOC. Current expansion plans of the facility include further 4 beamlines, one of them being a specific microfocus MX beamline, BL06-XAIRA, now in design stage.

The scientific case for XAIRA includes two aims: 1) to provide a full beam with a size of 3×1 µm2 FWHM (h×v) and a flux of >3×1012 ph/s (250 mA in storage ring) at 1 Å wavelength (12.4 keV); and 2) to reach a wide range of energies, 4-14 keV, to support MX experiments at long wavelengths exploiting the anomalous signal of the metals naturally occurring in proteins (native phasing), which is enhanced in the case of small crystals.

To match these requirements, the design of the beamline foresees a powerful photon source, a 2.3-m long in-vacuum undulator device, and the optics system prioritizes a high beam spatial stability. A channel-cut monochromator (CCM) will be installed, followed by a vertically focusing mirror long enough to accept the variations of the beam height introduced by the monochromator. This way, thanks to the large demagnification factor, the parasitic excursion of the beam at the sample position will be kept within only 60 µm for the whole energy range, which greatly simplifies the alignment of downstream elements. A high flux operation mode will also be possible, at the expense of energy resolution, by using a multilayer monochromator. To minimise the loss of flux at the low energy limit, the air path will be reduced between the sample and the detector by means of a He cone, and the whole beamline will only include two vacuum windows, upstream and downstream the sample.

The beam size will be adjustable to the users needs, producing larger sizes by defocusing the beam, or closing the horizontal secondary source slits to obtain a smaller spot size at sample, down to 1×1µm2. A high precision air bearing goniometer with vertical rotation axis will provide a stable alignment of micron-sized samples to the small beam.

With a state-of-the-art pixel array detector and automated sample mounting and data analysis, XAIRA will allow rapid sample screening and data collection of crystals, and it will support a broad range of advanced diffraction experiments using micron-sized beam, from raster scanning to serial crystallography.

Keywords: microfocus beamline, XAIRA, ALBA