MS050.O05

High-flux time-resolved experiments and anomalous scattering at EMBL P12 beamline

Andrey Gruzinov[1], Clement E. Blanchet[1], Martin A. Schroer[1], D.C. Florian Wieland[2], Alessandro Spilotros[1], Daniel Franke[1], Nelly Hajizadeh[1], Cy M. Jeffries[1], Stefan Fiedler[1], Sergey Filippov[3], Manfred Roessle[4], Gergely Katona[5], Dmitri I. Svergun[1]
[1]EMBL Hamburg, Hamburg, Germany, [2]Helmholtz-Zentrum Geesthacht (Außenstelle DESY), Hamburg, Germany, [3]Institute of Macromolecular Chemistry, Prague, Czech Republic, [4]Luebeck University of Applied Science, Luebeck, Germany, [5]University of Goteborg, Goteborg, Sweden
E-mail: agruzinov@embl-hamburg.de

P12 is a low background high brilliance beamline dedicated to the investigation of biological molecules in solution and run by the EMBL at the PETRA III storage ring (DESY, Hamburg). Standard mode of operation includes high throughput robotic sample delivery, online size exclusion chromatography, automatic data collection, reduction and also analysis in near-real time regime (Blanchet et al., 2015). In addition to further improvements of the standard operation on the beamline, recent developments aim at exploiting the high flux and energy tunability for time-resolved and anomalous SAXS experiments.
The kinetics of protein reaction spans over a large range of time from a fractions of seconds for protein folding to minutes or hours for microtubular nucleation or amyloid fibrils formation. While slow kinetics (seconds to hours) can be measured easily on modern beamlines, the study of fast (sub-msec) kinetics is still challenging. In standard data collection mode, (flux of 5*10^12 photons/second, low instrumental background and noise free PILATUS 2M detector) exploitable SAXS curves are collected in 30-50 ms to study sub-second kinetics using e.g. a stopped flow Biologic SFM 400 device. For faster kinetics, a double multilayer monochromator (MLM) was recently taken into operation providing the 4*10^14 photons/second (Blanchet et al., 2017, in preparation). Using the fast EIGER 4M detector with a framerate up to 750 Hz the scattering curves collected in 1.3 ms on different standard protein samples with concentration of a few mg/ml have sufficient quality for data analysis and further structural modeling. A fast rotating beam chopper is currently being built to provide X-ray pulses for stroboscopic or pump-probe experiments with tunable pulse length.
For fast time resolved experiments P12 is equipped with a high energy nanosecond laser of a spectral range from 355 to 2300 nm to trigger reactions through temperature-jump, release of caged compound or by perturbing photo-sensitive protein. An additional option utilizes terahertz (THz) spectroscopy using frequencies between microwave and infrared spectral ranges. The THz setup (source and detector) combined with the MLM and a fast read-out detector gives an opportunity to study sub-millisecond non-thermal shifts of biochemical equilibria and intramolecular vibrations of proteins.
High flux and low background of P12 adds the possibility to detect anomalous scattering signals from weakly scattering macromolecular solutions containing heavier elements. Standard setup with tunable double crystal monochromator ($\Delta\lambda/\lambda$ = 0.01%) with an energy range 6-20 keV allows for energy tuning around the absorption edges of heavy atoms to reveal their distribution in the structure. Pilot ASAXS experiments have recently been conducted using anomalous SAXS to characterize Br-labeled polymeric nanoparticles and the ASAXS option is now accessible to friendly users.
[1] Blanchet, C. E., Spilotros, A., Schwemmer, F., Graewert, M. A., Kikhney, A., Jeffries, C. M., Franke, D., Mark, D., Zengerle, R., Cipriani, F., Fiedler, S., Roessle, M. & Svergun, D. I. (2015). J. Appl. Cryst. 48, 431-443.
Keywords: time-resolved, synchrotron multilayer optics, ASAXS