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Complementing structural data obtained by X-ray diffraction with optical spectroscopic techniques has become a growing interest in structural biology. In-situ spectroscopy can reveal the nature of chemical species that remain ambiguous in the electron density maps.

Here we present some of the results obtained by the use of the on-axis micro-spectrophotometer developed at beamline X10SA of the Swiss Light Source [1].

The on-axis geometry of the micro-spectrophotometer is perfect for studying radiation damage and/or the X-ray induced phenomena. Photo-reduction of the copper centers in Copper nitrite reductase from *Achromobacter cycloclastes* has been monitored using UV-Vis absorption spectroscopy. A 'low-dose' data set with the Cu centers still oxidized has been collected and the structure has been validated by spectroscopy.

Raman spectroscopy under resonant conditions (in the Soret absorption band), has been carried out on two different hemoproteins: myoglobin from horse heart and cytochrome c' from *Alcaligenes xylosoxidans* by the use of laser probes at either 405 or 413 nm. In both cases vibrational spectroscopy results complement the active site picture provided by X-ray diffraction.

Non-resonant Raman experiments, with an excitation wavelength in the near infra red domain (785 nm), have been successfully performed on horse heart insulin and hen egg-white lysozyme. For these two proteins, the disulfide bond breakage due to X-ray exposure has been followed by the decreasing intensity of the S-S stretch band.

[1] R.L Owen, A.R. Pearson, A. Meents, P. Boehler, V. Thominet, C. Schulze-Briese, *Journal of Synchrotron Radiation* **2009**, *16*, 173-182.

Keywords: spectroscopy, Raman, UV-Vis absorption

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The HC1 at Diamond, setup, use and first successful results

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A sample humidity control device (HC1) [1-3] has been integrated on the beamlines of the Macromolecular Crystallography Village at Diamond Light Source. The device is primarily used to improve the diffraction quality of crystals via controlled dehydration but is also used for room temperature data collection, as mounting samples is very simple and easy. The current implementation allows easy transfer between the different beamlines and even the beamline laboratories to suit user demand permitting a very efficient use of beamtime. Users can characterise their crystals and optimise their dehydration protocols in a few hours of beamtime. Later, with the device off-line, they can apply these protocols, conditioning and cryo-cooling as many samples as needed in order to obtain the desired dataset. We'll give examples of use that will include some of the most recent results our users have achieved.

[1] J. Sanchez-Weatherby *et al*. *Acta Crystallographica D Biological Crystallography* **2009**, *65*, 1237-46. [2] S. Russi, *et al*. *Journal of Structural Biology* **2011**, doi:10.1016/j.jsb.2011.03.002 [3] J. Kadlec, *et al*. *Nature Structural & Molecular Bilogy* **2011**, *18*, 142-149.

Keywords: diffraction, relative humidity, dehydration

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More flux, less background: Improvements in low power X-ray beam delivery systems

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Microfocus sealed tube sources coupled to advanced x-ray optics provide a high brighness beam in a low maintenance package. These systems are increasingly used in single cristal diffraction applications due to increased performance compared to traditional rotating anode generators in particular for small crystal analysis. Small Angle X-ray Scattering applications require sample illumination with a high brilliance x-ray beam having a well controlled spatial and angular distribution. Indeed high intensity at the sample is required with small beam expansion towards the detector to achieve low values of wave vector.

We will present new developments in the field of beam delivery and beam conditioning systems enabling the optimum use of low power high brightness microfocus sources. These developments include both aspheric multilayer optics with increased capture angle as well as new collimation devices for reduced background signal.

Application data illustrating the capabilities of this new generation X-ray beam delivery system (the GeniX^{3D}) will be presented. The results include diffraction measurements on tiny and poorly diffracting crystals and aperiodic crystal analysis. First results on the combination of the GeniX^{3D} with new hybrid pixel detectors will also be presented highlighting the capabilities of these systems for fast fine slicing measurements.

Small angle X-ray scattering requires well controlled beam propagation with low parasitic scattering while maintaining a high intensity at the sample. We will present how the unique combination of new scatterless collimation with aspheric multilayer optics impacts the useful flux for SAXS applications in particular on diluted solutions.

Keywords: source, optics, saxs

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Copper(I) Anilide Complex $[Na(phen)_3][Cu(NPh_2)_2]$—an Intermediate of Copper-Catalyzed *N*-Arylation of *N*-Phenylaniline Reaction

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Complex $[Na(phen)_3][Cu(NPh_2)_2]$ (**2**), containing a linear bis(*N*-phenylanilide)copper(I) anion and a distorted octahedral tris(1,10-phenanthroline)sodium counter cation, has been isolated from the catalytic C-N cross coupling reaction based on the CuI-phen-*t*BuONa catalytic system. Complex **2** can react with 4-iodotoluene to produce 4-methyl-*N,N*-diphenylaniline (**3a**) with 70.6 % yield. In addition, **2**