

## Poster Sessions

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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 xylooxidans* 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**

### MS51.P18

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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 Biology* **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 brightness beam in a low maintenance package. These systems are increasingly used in single crystal 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<sup>3D</sup>) 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<sup>3D</sup> 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**

### MS51.P20

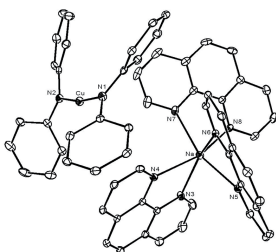
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#### Copper(I) Anilide Complex [Na(phen)<sub>3</sub>][Cu(NPh<sub>2</sub>)<sub>2</sub>]—an Intermediate of Copper-Catalyzed N-Arylation of N-Phenylaniline Reaction

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Complex [Na(phen)<sub>3</sub>][Cu(NPh<sub>2</sub>)<sub>2</sub>] (**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**

can work as an effective catalyst to carry out the C-N coupling under the same reaction conditions indicating that **2** is the intermediate of the catalytic system. Both  $[\text{Cu}(\text{NPh}_2)_2]^-$  and  $[\text{Cu}(\text{NPh}_2)\text{I}]^-$  were observed by *in situ* electrospray ionization mass spectrometry (ESI-MS) analysis under the catalytic reaction condition indicating that they are intermediates in the reaction. A catalytic cycle was proposed based on these observations. Molecular structure of **2** was determined by single-crystal X-ray diffraction analysis.



**Keywords:** C-N coupling, copper, ESI-MS

## MS52.P01

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### Effect of thermal treatment on characteristics nanodiamonds and diamond blend

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Diamond nanoparticles are now the most commonly synthesized nanomaterials. As they have some distinctive properties, such as high adsorptive capacity, high thermal conductivity, high surface energy, hydrophobicity and a large specific surface, they are of great interest as materials with a wide application range. Nanodiamonds (NDs) can be used as adsorbents and catalysts for the production of nanocomposite materials, for growing diamond films, in medicine and biotechnology [1-3]. They can not now be widely used mainly because there are no well-defined standards and the quality of the NDs, offered by different producers, is unstable [4]. Therefore, the study of NDs is important and promising.

The main goal of NDs purification is removal of non-diamond forms of carbon. Practically all known methods are based on the use of the different resistances of diamond and non-diamond forms of carbon to oxidants. To apply them, many technical problems, arising in exothermal reactions at high temperatures and intensive gassing in a limited volume, should be solved [5]. A decisive factor in selecting a purification variant is safety.

The goal of the project was to assess the effect of thermal treatment in vacuum on the structural characteristics and chemical composition of NDs and a diamond blend produced by detonation synthesis. NDs in the form of powders were obtained from two sources: Gansu Liru Lingyun NanoMaterial Co Ltd. (China) and NanoCarbon Research Institute Ltd. (Japan). A diamond blend (brand AIII-A) was offered by Scientific and Technical Enterprise "Sinta" (Belarus).

It is commonly accepted that only a complex of diagnostic techniques gives the possibility to get a true knowledge about the carbon nanostructures properties. Chemical composition was determined by mass-spectrometry (an inductively-coupled plasma mass-spectrometer ICP-MS Xseries2 (ThermoFisher Scientific)) and X-ray spectral analysis (a VEGA II LSH electron microscope (TESCAN OJSC) with an attachment for microprobe analysis); different levels of structural elements aggregation were analysed by SEM (VEGA II LSH), AFM (N'Tegra Aura NT-MDT) and DLS (Malvern Instruments Ltd.) methods; structural parameters of crystallites were analysed by X-ray structural analysis (Thermo Scientific ARL X'TRA Powder X-ray Diffraction System); in order to determine the pair density-density

correlation functions and to estimate parameters of the nanostructure (correlation length, specific surface and fractal dimension) was used SANS.

Our study has shown that thermal treatment under mild conditions is a promising approach to purification of NDs and a diamond blend from the point of view of economy, safety and environmental protection.

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**Keywords:** carbon, nanotechnology, diamond

## MS52.P02

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### Synthesis of nanographene organometallics for deposition on a graphene surface

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Graphene's unique electrical and mechanical properties give it a wide variety of possible applications in the future of electronic devices [1]. Being essentially an extended network of interconnected arene rings, it is also of much interest in chemistry. It can be modified chemically via oxidation, (partial) hydrogenation etc to alter the band gap, or else used as a surface on which to adsorb small molecules which can be visualised using AFM and STM microscopy. Such small molecule adsorption can be used for reactions directed by the graphene surface or for functionalising the graphene [2].

We have successfully synthesised a range of organometallic compounds with coordinated polycyclic aromatic hydrocarbons (or nanographenes) such as pyrene which are either novel or found as a new polymorph. These compounds have been characterised using a variety of spectroscopic methods, as well as X-ray crystallography. These compounds will be deposited on to graphite or graphene and imaged using AFM and STM microscopy. This will allow us to optimise the surface coverage of such molecules and analyse their interactions with the surface and each other. By 'sticking' molecules such as these to graphene we hope to be able to alter the electrical, magnetic and optical properties of graphene.

