short x-ray free electron laser pulses. A special molecular dynamics model has been developed to describe the Coulomb explosion of the clusters [1]. We use numerical modeling based on the non-relativistic classical equation of motion. Quantum processes are taken into account by the respective cross sections. The explosion dynamics is examined for various conditions: pulse length, constituent atomic number, number of photons in a pulse. We use our model to get an estimate of the time available for imaging before the cluster deteriorates significantly. Based on these results we calculate the continuous elastic scattering pattern of the sample and try to reconstruct the original atomic order from this pattern. We use a density modification type algorithm analogous to the Fienup hybrid input output reconstruction method [2,3]. Since this method needs a 3D dataset in the reciporocal space, scattering patterns have to be taken at various sample orientations. That requirement leads to a multi shot experiment. Therefore the full dataset have to be built from scattering patterns of several independent exploding clusters. We included this complication in the calculations. We found that the shorter the pulse the higher the ratio of the photons useable for imaging. The conclusion of the calculations is that the pulse length of the presently planed x-ray free electron lasers is too long.

[1] Jurek Z., Faigel G., Tegze M., *European Physical Journal D*, 2004, **29**, 217. [2] Fienup J.R., *Optics Letters*, 1978, **3**, 27. [3] Jurek Z., Oszlanyi G., Faigel G., *Europhysics Letters*, 2004, **65**, 491.

Keywords: X-ray free-electron lasers, clusters, molecular dynamics simulations

MS22.25.5

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Three-dimensional Data merging of Randomly Oriented Continuous Diffraction Patterns

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We have developed methods for the assembly of three-dimensional diffraction data from noisy and randomly oriented continuous diffraction images. Before a structural reconstruction is possible, the patterns must be oriented with respect to each other and the signal to noise ratio must be increased by averaging of redundant data. While certain aspects of this problem are similar to problems in single-particle electron tomography, there are also significant differences. In single-particle electron tomography, similar images are located based on their correlation and the mutual orientation of the averaged images is determined from the common lines of intersection of their Fourier transforms. We present an extension of this scheme to the case of diffraction images, which intersect in spherical sections in Fourier space rather than in planar sections and which have statistical properties different from those of tomograms. We study how our scheme works on both real and simulated sets of three-dimensional data.

Keywords: three dimensional image, imaging, fourier transform

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MS23 PUTTING THE PEDAL TO THE METAL: SPEEDING UP BIOLOGICAL STRUCTURE DETERMINATION

Chairpersons: E. Yvonne Jones, Peer R. Mittl

MS23.25.1

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Development of a High-throughput Structure Determination Pipeline at BM14

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The basic operations to be performed for the collection of MAD and SAD data from protein crystals are well established and have been implemented successfully at beamlines across the world. There is now, however, intense activity aimed at revolutionizing the possibilities via a series of improvements, centered around (i) the mechanical aspects of automation, improved precision and improved visualization and (ii) the software aspects of automation and integration.

BM14 is a tunable bending magnet beamline at the ESRF operated by the UK research councils in collaboration with the EMBL Grenoble outstation. A high-throughput pipeline for structure determination by SAD and MAD phasing is being developed through our participation in the BBSRC e-science project e-HTPX (www.e-htpx.ac.uk).

An overview of the hardware and software implemented at BM14 for automation of macromolecular data collection will be presented. In particular, software developments which allow the user to keep track of the sample from their home lab to and from the beamline, as well as management of experimental data acquired, through the development of an easy to use beamline Laboratory Information Management System (LIMS) will be described. Our experiences in the use of SAD phasing with naturally occurring light atoms, such as sulphur and manganese, and their application for use in a high-throughput structure determination pipeline are summarized.

Keywords: MAD phasing, automation, LIMS

MS23.25.2

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Automating Crystallographic Structure Determination Calculations

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Structural genomics efforts require a high throughput at all stages of the structure determination process. Simultaneously, it is important to reduce the cost per structure, which means reducing the time spent on each structure. We have focused on the structure determination calculations going from processed, merged data through to initial model. The Automated Crystallography System (ACrS) utilizes existing software and algorithms but a distributive program interface administers the programs for determining protein structures. A relational data base stores initial data for starting the process as well as harvesting and warehousing data generated during the structure determination process.

The ACrS default mode of operation is to try several defined pathways in parallel. Analysis of the results in the database provides information for improving the pathways and for selecting software with complementary strengths.

An example is a recently determined structure of a member of the ROK family of transcription regulators that used a “native” data set and SAD phasing from one bound zinc to automatically built 384 residues of 405 without any intervention or optimization of parameters.

Keywords: structural genomics, automatic structure solution, macromolecular structure

MS23.25.3

Acta Cryst. (2005). A**61**, C34-C35

HT Structure Determination at SER-CAT: Five Structures in 23 Hours

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Researchers at the University of Georgia (UGA) have developed an optimizing, high throughput structure determination pipeline (SCA2Structure) capable of providing fitted or partially refined structures in a matter of hours from anomalous scattering (MAD or SAD) data [1]. This powerful structure determination engine coupled with the excellent data collection facilities provided by the SER-CAT, beamlines at the Advanced Photon Source (www.ser-cat.org) provides the basis for high-throughput structure determination.

Using prescreened crystals and data collected at SER-CAT, UGA researchers were able to determine five SAS structures on-site during a recent 24-hour run. Data were processed at SER-CAT and input to

the SCA2Structure pipeline running on a Linux cluster at UGA via the web. The average total time for data collection and structure determination was 191 minutes. The structures solved represented an average mix of structural genomics targets with molecular weights ranging from 12 - 25 kDa. Details of the experiments will be presented. Work supported in part with funds from the NIH (GM62407), The Georgia Research Alliance and The University of Georgia Research Foundation.

[1] Liu, et al., *Acta Cryst*., 2005, D**61**, *in press*.

Keywords: HT structure determination, SCA2 Structure, SER-CAT

MS23.25.4

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The Integration of Data Reduction and Structure Solution - from Diffraction Images to an Initial Model in Minutes

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A new approach that integrates data collection, data reduction, phasing and model building significantly accelerates the process of structure determination and, on average, minimizes the number of data sets and synchrotron time required for a structure solution. The initial testing of the system with 50+ of novel structure determinations proved its high value for MAD/SAD experiments. The heuristics of choosing the best computational strategy for different data resolution limits of phasing signal and crystal diffraction are being optimized. Typical end result is interpretable electron density map with partially built structure and in some cases even almost complete, refined model. The current development is oriented towards a very fast structure solution, in order to provide feedback during the diffraction experiment. Work is also proceeding towards improving the quality of phasing calculation and model building.

Keywords: high throughput structure determination, phasing, model building

MS23.25.5

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Medium throughput Protein Crystallography: Limiting Steps in the Pipeline

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The **S**tructural **P**roteomics **IN** **E**urope (SPINE) project was the first EC funded structural genomic project. Its aim was to foster the high throughput determination of proteins relevant to human health. The major bottlenecks was recognised to be the expression of soluble and stable proteins in sufficient amounts for crystallization, and this has proved to be true. The pipeline will be briefly summarized and the success rate for a set of proteins described. The presentation will concentrate on a set of targets from *Bacillus anthracis* from the SPINE partner groups in York and Oxford. A number of targets were selected using bioinformatics tools and put through the expression pipeline.

While only a small part of SPINE funds was allocated to crystallographic software, a number of scientists have recently been contributing to automation developments. Recent experience on applying these to SPINE targets will be described and bottlenecks indicated.

Keywords: macromolecules, automation, software

MS24 MOLECULAR CRYSTALS UNDER NON AMBIENT CONDITIONS

Chairpersons: Judith A.K. Howard, Jacqueline Cole

MS24.25.1

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Blowing Hot and Cold and its effect on some crystals

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The benefits of collecting single crystal diffraction data at low temperatures are well known and the use of low temperature devices is now very well established and widespread for small molecule crystallography; in many cases to usefully collect data at a single temperature. The combination of easily controllable devices with a wide temperature range and the use of area detectors allowing rapid data collection makes variable temperature studies and thus exploration of the structural changes that occur with changes in temperature much more accessible.

At the higher end of the temperature scale, and as part of a wider project, we are investigating the effect of temperature on selected porous coordination networks and hydrogen-bonded arrays. These networks, which can be considered metal-organic zeolite analogues, form channels, pores and cavities which may include guest organic molecules. By heating the crystal and collecting data *in situ* we can monitor the structural changes that occur with increased temperature, in particular due to desorption of these guest molecules.

At the lower temperature range we have been investigating structural changes at the metal centre of some transition metal complexes.

Examples from studies carried out in the temperature range 35-500K using an open flow HeliX helium cryostat and a Cryostream plus will be presented.

Keywords: low and high temperature devices, metal-organic compounds, porous materials

MS24.25.2

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Photo-induced Molecular Switching : Neutron Diffraction Studies

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The design of molecules that could be utilized for information storage is one of the main challenge in molecular material science and optical switching is one of the most intense areas of interest in memory molecules. Polarized neutron diffraction (PND) was used for the first time to investigate the photo-magnetic properties of photo-switchable inorganic molecular solids. Spin crossover compounds containing an octahedrally coordinated Fe^{2+} ion present a low spin diamagnetic (S = 0) ground state which can be switched, under light illumination with a suitable light wavelength, to a high spin paramagnetic (S = 2) metastable state having an extremely long lifetime at low temperatures.

A new experimental setup, allowing for both in-situ light illumination and PND measurements, has been developed on the 5C1 diffractometer at the LLB and tested on the $[Fe(ptz)_6](BF_4)_2]$ (ptz = 1-propyltetrazole) spin crossover compound [1]. The photo-excitation kinetics was followed by PND, which evidenced a complete photo-excitation process. The first magnetization density map in a photo-induced magnetic state has been obtained at 2K using a laser beam with 473 nm.

[1] Goujon A., Gillon B., Gukasov A., Jeftic J., Nau Q., Codjovi E., Varret F., *Phys. Rev. B*, 2003, **67**, 220401(R).

Keywords: polarized neutron scattering, molecular magnetism, molecular switches

MS24.25.3

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Photo Excited State Crystallography of Iodo-bridged Dicopper (I) Complex

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Luminescent dicopper(I) complex $[Cu_2I_2(PPh_3)_2(4,4'\text{-bpy})]_\infty$ (bpy=$C_{10}H_8N_2$) consists of $\{Cu_2I_2\}$ planar units, which are bridged by