

unmounting and centering are all integrated into Blu-Ice through an intuitive custom robot tab; response over internet connections is reasonable from home (or café) wifi networks. Remote connection also allows data processing without the bottleneck of transferring the data home. Successful remote collection has enabled the MBC to institute an (almost) on-demand scheduling paradigm where members request beamtime as needed in blocks of time from 4 to 48 hours while beamline visits and Service Crystallography fill in the gaps. An MBC on-call list is available for beamtime to fill unused shifts and another ad-hoc beamtime request system is available for non-members. Remote collection has the benefit of easing access to the synchrotron beamline, encouraging the Mentor/Student relationship during data collection and provides a teaching platform that may otherwise be unavailable to crystallography labs.

Keywords: remote control, synchrotron radiation crystallography, X-ray data collection

### P01.13.83

*Acta Cryst.* (2008). A64, C196

#### Femtosecond X-ray science at the Swiss Light Source

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Since mid-2006, 120 femtosecond synchrotron x-rays have been generated and used for a variety of experiments at the Swiss Light Source. Specifically, the source has been used to observe with high precision the structural dynamics of highly photoexcited semiconductor and semimetal crystals, allowing a more systematic study of the interaction mechanisms between electronic quasiparticles and phonon modes. We present here an overview of the techniques used to generate the femtosecond x-ray pulses, as well as an overview of the properties of these pulses relevant for experiments. We also demonstrate the successful use of the source to observe and control the femtosecond lattice dynamics of bismuth and tellurium.

Keywords: time-resolved x-ray diffraction, time-resolved effects, laser radiation

### P01.02.84

*Acta Cryst.* (2008). A64, C196

#### WAXS as a novel tool in drug discovery

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A major advance in drug discovery has been the development of techniques to generate large libraries of target-focused probe chemicals. This development, in combination with the ever-increasing numbers of proteins entering screening programs via human genome expression profiling, has intensified the need for

novel rapid screening techniques that can pinpoint those molecules with biologically relevant properties (such as knock-down or knockout activity). Most methods used to date rely on 1 of 2 strategies: either detection of physical binding or impairment of target function. The former class usually requires immobilization or tagging of 1 or more of the binding pairs and will identify ligands that may or may not impair protein activity, whereas the latter require specialized assays for each target function and may be less amenable to a high-throughput approach. Frequently, the functional binding of a small molecule to a protein is accompanied by a change in the structure of the protein. Wide-angle x-ray scattering (WAXS) is a sensitive probe of structural change in proteins and can detect protein changes across all relevant length scales. It addresses the shortcomings of existing screening techniques as it does not require either the protein or the ligand to be immobilized, labeled or modified in any manner and secondly it detects structural changes, not binding per se. We describe here the apparatus used at the Advanced Protein Source to collect WAXS data from small volumes of protein/protein-ligand solutions and proof-of-principle experiments that point towards the potential of WAXS as a novel routine screening tool for the detection of functional interactions between proteins and small molecule ligands for the purposes of drug discovery and development.

Keywords: wide-angle scattering, drug discovery, protein-ligand complexes

### P01.13.85

*Acta Cryst.* (2008). A64, C196–197

#### Time-resolved photo-crystallographic investigation of metastable species

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X-ray crystallography is a 'gold star' analytical tool for obtaining structural information. Recently, crystallography has developed, so that structural information can be obtained as the reaction proceeds. One exciting development in this field is photocystallography [1], which uses crystallography to monitor photochemical processes, in this case the formation of light-induced metastable species [2]. We now report the successful investigation of metastable species in a range of nickel complexes  $[\text{Ni}(\text{NO}_2)_2\text{L}]$  L = (Aminoethyl)-pyrrolidine. In this investigation, the  $\text{NO}_2$  ligand undergoes linkage isomerism [3] when irradiated by LEDs causing a change in coordination mode from the N-bound to the O-bound isomer. The new isomer is metastable and exists for a prolonged period of time. Using photocystallography it is possible to monitor the new metastable conformation and percentage of converted  $\text{NO}_2$  ligands. The use of synchrotron radiation is key to this experiment as the high intensity allows for high quality results, short data collection times and the use of smaller crystals reducing the potential problem of only photoexcited surface ligands. The experimental techniques at Station 9.8 STFC and Station 11.3.1 ALS San Francisco will be discussed. The Pyrrolidine complex produces a 40% conversion at 100K and is metastable for periods over an hour. In temperature experiments, the conversion percentage diminishes below 85K and above 120K. Higher conversions, with more extensive irradiating, were not explored due to crystal strain often resulting in the crystal fracturing.

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3. P. R. Raithby; *Chem. Commun.*, 2006, page 2448-2450

Keywords: time resolved analysis, metastable structure

determination, photochemistry coordination compounds

### P01.13.86

*Acta Cryst.* (2008). A64, C197

#### Picosecond crystallography of homogeneous [2+2] photodimerisation reactions

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In the following we will present ultrafast crystallographic studies on the photo-induced single-crystal-to-single-crystal dimerisation of  $\alpha$ -styrylpyrylium(TFMS) ( $C_{23}H_{29}O_3F_3S$ ).  $\alpha$ -styrylpyrylium(TFMS) is well known as an optical switcher with the ability to be used as a storage device. The mechanism underlying the storage process is a ultrafast [2+2] photodimerisation reaction, which is reversible (higher temperature) or irreversible (lower temperature) implying temporal stability. In this contribution we show the possibility to switch the material between the ground state (monomer phase) and the photoactivated state (dimer phase) in an ultrafast manner with time-scales below 50 ps. The static x-ray diffraction studies have been performed down to electron density resolutions. For the time-resolved studies of the structural dynamics in the solid, picosecond time-resolved crystallography has been employed. Additionally, we discuss the influence of the exciting photon energy on the structural photoswitching of  $\alpha$ -styrylpyrylium(TFMS) and its influence on the dimerisation mechanism.

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[4] submitted

Keywords: photodimerization, time-resolved crystallography, organic crystals

### P01.14.87

*Acta Cryst.* (2008). A64, C197

#### Modeling of single molecule imaging by X-ray free electron laser

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We study the possibility of imaging a small cluster of atoms by short x-ray free electron laser pulses. Since this type of sources are under construction no experimental data is available, which could reliably tell the conditions of successful imaging. Therefore modeling of the experiment and evaluation process is crucial. On the experimental part one of the most important questions is how fast the sample deteriorates in the beam. We have developed a special molecular dynamics model for the description of the Coulomb explosion of the clusters. Using this model we can follow the time evolution of all particles (including atoms, ions, electrons) of the sample. Beside the modeling of samples with different compositions, we also studied the effect of a thin sacrificial temper layer about the sample. We found that with a proper choice of the temper layer the deterioration of the sample can be significantly slowed down. This may help

the realization of the planned experiment. Based on the results of molecular dynamics modeling we can calculate the 2D continuous elastic scattering pattern of the sample for a given shot. However, the most often used density modification type algorithms needs 3D data in the reciprocal space. Therefore the full dataset have to be built from scattering patterns of several independent exploding clusters taken at various unknown sample orientations. The first and most critical step in the evaluation process is the classification of the many million 2D patterns into a few thousands of bins. In every bin the sample orientation is the same, so that one can add the intensities of these patterns to improve the statistics to a level where reconstruction is possible. We show the results of our numerical study of the classification process.

Keywords: free electron laser, single molecule imaging, X-ray diffraction

### P01.11.88

*Acta Cryst.* (2008). A64, C197

#### Geometry and resolution of area detectors for X-ray powder diffraction

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Application of area detectors for powder x-ray diffraction has dramatically increased in recent years due to many advantages, including high speed and better sampling statistics with small samples or textured samples. In high-throughput x-ray diffraction screening, such as pharmaceutical research and discovery, two-dimensional x-ray diffraction is the preferred choice for many users. The Bragg-Brentano (BB) geometry has long been the classic configuration for collecting phase identification data from bulk powder or polycrystalline samples. With the increased usage of area detector, many users have noticed some discrepancies between the diffraction patterns collected with a BB system and an area detector system. In the BB geometry, the  $2\theta$  resolution is controlled by the divergence slit, receiving slit and scanning steps of the point detector. With an area detector, the  $2\theta$  resolution is mainly determined by the spatial resolution of the detector and the sample-to-detector distance. In order to have the optimum combination of  $2\theta$  range and resolution with an area detector, several frames covering different  $2\theta$  ranges may have to be collected and merged. The  $2\theta$  resolution may vary with  $2\theta$  range, sample shape and detector geometry. The relative peak intensity from a textured sample may be different from the results measured with BB geometry. It is imperative to study the nature of these discrepancies so that the diffraction patterns collected with area detectors can be used for phase ID with proper understanding and correction. This presentation compares the conventional Bragg-Brentano diffractometer with diffractometers using area detectors in terms of geometry convention, detector resolution, data collection and processing strategies.

Keywords: area detectors, X-ray powder diffraction, instrumentation

### P01.15.89

*Acta Cryst.* (2008). A64, C197-198

#### Effect of pressure and temperature on the crystallization behavior of As Te glasses with selenium

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