

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, D61, *in press*.

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

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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

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Medium throughput Protein Crystallography: Limiting Steps in the Pipeline

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The Structural Proteomics IN Europe (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

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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

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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 Fe²⁺ 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](BF₄)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., Jęftić 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 [Cu₂I₂(PPh₃)₂(4,4'-bpy)]_∞ (bpy=C₁₀H₈N₂) consists of {Cu₂I₂} planar units, which are bridged by

diimine ligands constructing infinite chain structure. The assignments of intense emission bands of these oligo-copper compounds are complicate because weak interacted multi-copper centered (CC) charge transfer is often concurrent with common Cu(I) to ligand charge transfer. We have tried to observe direct geometrical distortion of the complex by photo excited state crystallographic technique in order to figure out this emission nature.

Single crystal X-ray diffraction experiments were performed by using the low-temperature vacuum X-ray camera at SPring-8 BL02B1 beamline. Full intensity data of both under light irradiated and non-irradiated conditions were collected by multiple-exposure method by using IP detector. Photo-difference Fourier syntheses show that a small portion of two I atoms in {Cu₂I₂} unit move toward close to each other, while two Cu atoms tend to move apart from each other toward N atom of the bipyridine ligand.

[1] Henary M., Wootton J.L., Khan S.I., Zink J.I., *Inorg. Chem.*, 1997, **36**, 801.
[2] Ozawa Y., Terashima M., Mitsumi M., Toriumi K., Yasuda N., Uekusa H., Ohashi Y., *Chem. Lett.*, 2003, **32**, 62.

Keywords: photochemistry coordination compound, accurate measurement, crystallography instrumentation synchrotron radiation

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Multitemperature X-ray Diffraction Analysis in the Study of Phase Transitions, Molecular Dynamics and Crystal Disorder

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Routine ability to analyze crystal structures of different compounds at many temperatures (including "low" temperatures - up to 100K, and "very low" ones - up to 20-30K or even less) is important in many cases for different applications in the study of phase transitions, the nature of crystal disorder, and the study of dynamics of molecular (atomic) motions in the solid state. In addition, low-temperature data collection allows one to increase dramatically real accuracy of diffraction data and to increase the field of its analytical applications (the study of low-melting and/or unstable compounds). In particular, several methods of low-temperature crystallization techniques were elaborated during last time to analyze crystal structures of compounds which are liquids or even gases at normal conditions. Some new examples of the low/multitemperature X-ray diffraction analysis of molecular crystals will be demonstrated. These examples include the study of strong H-bond dynamics in organic compounds, polymorphism, phase transitions in liquid-crystalline precursors, structural studies of high-energetic and non-linear optical materials.

Keywords: multitemperature X-ray diffraction analysis, phase transitions, crystal disorder

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Melting-point Variation in Isomeric Dibromobenzenes

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Single crystals of 1,2- and 1,3-dibromobenzenes have been *in situ* pressure crystallized in diamond anvil cells and studied by X-ray diffraction. Both structures are orthorhombic: 1,2-dibromobenzene (0.3 GPa), space group *Pbca*, $Z = 8$, $a = 7.831(1) \text{ \AA}$, $b = 15.256(1) \text{ \AA}$, $c = 11.701(2) \text{ \AA}$; 1,3-dibromobenzene (0.2 GPa), space group *P2₁2₁2₁*, $Z = 8$, $a = 4.105(1) \text{ \AA}$, $b = 12.899(5) \text{ \AA}$, $c = 25.987(28) \text{ \AA}$. The relationship between the molecular symmetry and the melting point of the dibromobenzene isomers has been discussed. According to the empirical Carnelley's rule high molecular symmetry is related with high melting point [1], [2]. This effect however can be justified in various ways. The melting points are: 278-280 K for 1,2-, 266 K for 1,3-, and 356-360 K for 1,4-dibromobenzene. The Br...Br intermolecular interactions are distinct in both these structures, and they can be considered to be the main factor responsible for the

molecular rearrangements in these crystals.

[1] Carnelley T., *Philos Mag. 5th series*, 1985, **13**, 112-130. [2] Brown R. J. C., Brown R. F. C., *J. Chem. Educ.*, 2000, **77**, 724-731.

Keywords: high-pressure structure determination, halogens, structure-property relationships in solids

MS25 STRUCTURE DETERMINATION FROM POWDER DIFFRACTION DATA (ORGANICS)

Chairpersons: Andrew Fitch, John Faber

MS25.25.1

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Solving Organic Structures from Powder Diffraction: News from the FOX

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FOX, "Free Objects for Xtallography" [1][2] is a program for *ab initio* crystal structure solution using direct-space methods. While it was first developed and used to solve inorganic structures (oxides, intermetallic, hydrides), the algorithms used were not dedicated to any type of compound and organic structures could also be solved.

Over the last two years many modifications have been brought to the algorithm (mostly through a restraint-based description rather than a z-matrix one), to allow a better, more flexible description of molecular compounds.

We will present how the evolution of the algorithm has improved the ability to solve organic structures, along with new Fox features (multiple solutions, maximum likelihood,...).

[1] Favre-Nicolin V., Černý, R., *J. Appl. Cryst.*, 2002, **35**, 734. [2] Favre-Nicolin V., Černý R., *Z. Kristallogr.*, 2004, **219**, 847.

Keywords: powder structures, *ab initio* structure determination, algorithms

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Molecular Crystal Structures from Low-Resolution Powder Diffraction Data: Reliability and Validation of the Results Obtained

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In recent years, an obvious progress has occurred in the determination of molecular crystal structures *ab initio* from powder diffraction data. There are no doubts now, the molecular crystal structures with the known connectivity set in the molecule(s) can be routinely solved even from the data measured at the laboratory powder diffractometer. However, there are the questions still waiting for an exhaustive answering. One of them is a question on how to estimate the reliability of the solution obtained. Another one concerns the accuracy of structural parameters extracted. These questions are exceptionally important for the results obtained at laboratory commonly used devices. There are problems, which can not be clarified with the use of laboratory data only. So the problem of positioning of the selected hydrogens is beyond the facilities of many laboratory X-ray powder diffractometers, which are virtually "insensitive" to the position of H atoms. In this particular case, the neutron diffraction often helps to find a correct position, especially when some, or even all, of the hydrogens are replaced by the deuteriums.

For the new crystal structure solved from laboratory powder data we have a good chance to validate its correctness by the comparison of the results with those, obtained either from high-resolution synchrotron data or, in some cases, from neutron powder diffraction data. The aforementioned comparison allows us to estimate the real accuracy of the results derived from the laboratory powder pattern.