

P.01.15.2*Acta Cryst.* (2005). A61, C149**Symmetrisation of Powder Diffraction Peak Profiles by a Fourier Method**Takashi Ida, *Ceramics Research Laboratory, Nagoya Institute of Technology, Tajimi, Japan*. E-mail: ida.takashi@nitech.ac.jp

Powder diffraction intensity data with asymmetric peak profiles measured with a conventional Bragg-Brentano diffractometer and a high-resolution synchrotron diffractometer are treated with a fast Fourier transformation method [1, 2] to obtain intensity data with symmetric peak profiles. The method is based on deconvolution of analytical expressions of the optical aberrations of the diffractometers [3-6]. The symmetrised peak profiles enable simplified analytical procedures for individual peak profile fitting, whole pattern decomposition and Rietveld refinement.

The symmetrised diffraction data of fine SiC powder (JFCC, RP-2) measured with a conventional powder diffractometer has revealed a "super-Lorentzian" character of intrinsic diffraction peak profiles, which is reasonably explained by a theory for diffraction from small spherical crystallites with broad log-normal size distribution [7]. The results of a least-squares refinement applied to integrated intensity values of 42 reflections extracted from symmetrised high-resolution diffraction data of standard ZnO powder (NIST, SRM674) measured at Photon Factory in Tsukuba has been coincidence factor of $R = 0.45\%$ with reasonable structure parameters.

[1] Ida T., Toraya H., *J. Appl. Cryst.*, 2002, **35**, 58. [2] Ida T., Toraya H., *J. Appl. Cryst.*, 2003, **36**, 890. [3] Ida T., *Rev. Sci. Instrum.*, 1998, **69**, 3837. [4] Ida T., Kimura K., *J. Appl. Cryst.*, 1999, **32**, 634. [5] Ida T., Kimura K., *J. Appl. Cryst.*, 1999, **32**, 982. [6] Ida T., Hibino H., Toraya H., *J. Appl. Cryst.*, 2001, **34**, 144. [7] Ida T., Shimazaki, S., Hibino H., Toraya H., *J. Appl. Cryst.*, 2003, **36**, 1107.

Keywords: powder diffraction, profile analysis, Fourier methods**P.01.15.3***Acta Cryst.* (2005). A61, C149**The Role of e-Science in Service Crystallography: The UK National Crystallography Service on the Grid**Simon Coles^a, Jeremy Frey^a, Michael Hursthouse^a, Mark Light^a, David DeRoure^b, Hugo Mills^b, Ken Meacham^{b,c}, Mike Surrudge^{b,c}, ^a*School of Chemistry*, ^b*School of Electronics and Computer Science*, ^c*IT Innovation Centre, University of Southampton, Southampton, UK*. E-mail: s.j.coles@soton.ac.uk

The The EPSRC funded UK National Crystallography Service (NCS) facility has been exploring the use of Web/Grid services in e-Science applications. The NCS approach [1], [2] combines aspects of software and instrument automation to produce a service that increases user interaction and provides sample submission and data acquisition, processing and analysis services on the Grid.

A prospective user of the NCS applies for an allocation by filling in an electronic form and uploading a case for support, initiating the metadata capture process. Following successful peer review, the user and is provided with digital keys that enable secure access to the NCS Grid Facility. The user may now submit samples to the NCS through an electronic interface, which gathers all the chemical metadata concerning the sample, e.g. synthetic pathway, proposed formula 2D structure, sensitivity, COSHH safety information, etc.

A sample status database is used to monitor the progress of sample(s) in the queuing system. When a sample is scheduled for examination the user may initiate a secure, Web services based, interactive experiment from the sample status database. The crystal is mounted on the diffractometer by the sample changing robot or by a service operator. The user is then involved in a series of decision making stages, either automatically or with the service operator, which control the outcome of the unit cell determination and data collection procedures. At the conclusion of the experiment the data is automatically processed and made available to the user for download.

[1] S.J. Coles, J.G. Frey, M.B. Hursthouse, M.E. Light, K.E. Meacham, D.J. Marvin & M. Surrudge. *J. Appl. Cryst.*, Submitted [2] Coles S.J., Frey J.G., Hursthouse M.B., Light M.E., Surrudge M., Meacham K.E., Marvin D.J., De

Roure D.C., Mills H.R., (2002), In Hopgood, F.R.A., Matthews, B. and Wilson, M.D. (eds.), British Computer Society. (<http://eprints.soton.ac.uk/346/>)

Keywords: e-science, service crystallography, grid computing**P.01.15.4***Acta Cryst.* (2005). A61, C149**Automated Data Collection at the IMCA-CAT Advanced Photon Source User Facility**Anne Mulichak, Kevin Battaile, Irina Koshelev, J. Lewis Muir, Kathleen Favale, Ann Bertling, Lisa Keefe, *Sector 17, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA*. E-mail: mulichak@anl.gov.

The Industrial Macromolecular Crystallography Association Collaborative Access Team operates a data collection facility for protein crystallography at the Advanced Photon Source. The IMCA-CAT insertion device beam line is the only facility for protein crystallography at APS currently offering fully functional robotics for routine use. A Rigaku/MSC ACTOR robot provides automated mounting, centering and retrieval of protein samples. Integrated with user-friendly software, robotics enables high-throughput sample screening and unattended data acquisition according to user-programmed schedules, thus significantly reducing both the time necessary for crystal screening and the need for direct operator interaction.

While targeting the needs of drug discovery research for IMCA member pharmaceutical companies, the automation capabilities at IMCA-CAT are also ideally suited for structural genomics and other research efforts requiring high-throughput experiments. IMCA-CAT facilities are available to interested researchers through the APS General User Program.

Keywords: synchrotron beamline, automation, robotics**P.01.15.5***Acta Cryst.* (2005). A61, C149**An Ultra-fast Mechanical Shutter for Sub-microsecond Time-resolved Experiments**Milan Gembicky, Philip Coppens, *Department of Chemistry, State University of New York at Buffalo, NY 14260-3000, USA*. E-mail: gembicky@buffalo.edu

A new high speed, high rep-rate X-ray beam shutter for time-resolved photo crystallography [1] at synchrotron sources has been developed and tested. The new design is based on a commercially existing DC servomotor and a frequency-lock control capable Linear Amplifier. Accurate speed control combined with an air bearing results in extremely low jitter in the motor rotation. Measured jitter at rotation speeds of 200 Hz to 500 Hz is less than 2 ns at a 6 σ level confidence. The chopper disk is interchangeable, allowing maximum flexibility. The current chopper disc with 45 radial slots allows synchronizing with the APS storage ring from the 12th to the 24th subfrequencies, corresponding to X-ray pulse frequencies of 11.3 to 22.6 kHz. At 30000 RPM the opening time for 350 μ m slots is 1.65 μ s, i.e. less than half the orbit time at the APS source. Results demonstrate that low cost components can be used to build an exceptionally precise instrument. The shutter provides high accuracy and efficient use of X-rays at a modest cost.

[1] Coppens P., Vorontsov I., Graber T., Gembicky M., Kovalevsky A. Y., *Acta Cryst.*, 2005, A61, 162-172.

Keywords: photo-crystallography, synchrotron, ultra-fast shutter**P.01.15.6***Acta Cryst.* (2005). A61, C149-C150**High-throughput Protein Crystallization**Masahiko Hiraki^a, Ryuichi Kato^a, Yusuke Yamada^a, Naohiro Matsugaki^a, Noriyuki Igarashi^a, Soichi Wakatsuki^a, ^a*Structural Biology Research Center, Photon Factory, Institute of Materials Structure Science, KEK, Japan*. E-mail: masahiko.hiraki@kek.jp

For high-throughput protein crystallography, we are developing a fully automated X-ray structural analysis system that consists of several subsystems for protein crystallization, harvesting and freezing

crystals, mounting inside a hutch and data acquisition.

Obtaining X-ray quality crystals might require setting up thousands of crystallization trials on different conditions for protein solutions and precipitants. To this end, we have developed a large-scale protein crystallization system with the capability of setting up 240,000 crystallization trials per day. This system allows efficient crystallization trials of large protein complexes and structure-based drug design. It includes a crystallization tray supplier, a dispensing system using disposable tips which are specially designed for high dispensing accuracy, a sealing system, an observation system for taking images of drops in wells according to a pre-programmed schedule and storing them to the file server, incubators and a carrying robot which moves the trays between these components. Users can access the file server from anywhere in the world through a Web browser for checking the status of their crystal growth.

We will also describe a crystal harvesting system and automated sample exchange robots based on the SAM (SSRL Automated Mounting) system as part of our efforts in automating X-ray structure determination.

Keywords: crystallization robots, automated crystallization, automated data collection

P.01.15.7

Acta Cryst. (2005). A61, C150

Miniaturization in Structural Biology Pipelines

Enrique E. Abola^a, Peter Kuhn^b, Raymond Stevens^a, ^a*Department of Molecular Biology, The Scripps Research Institute, La Jolla, CA, USA.* ^b*Scripps-PARC Institute, La Jolla CA, USA.* E-mail: abola@scripps.edu

High-throughput (HT) structural biology is making significant contributions to our attempts at developing a complete understanding of biological systems. Process pipelines designed using HT approaches have now been implemented to explore protein fold/function space, to accelerate the Structure Based Drug Discovery, to study complete proteomes, and to develop and disseminate methods and technologies for working with integral membrane proteins.

The central theme of the technology development in our laboratory and that of our collaborators has been automation, integration, and miniaturization of processes in High-throughput structural analysis pipeline. These developments have dramatically increased success rates reducing the cost per structure by decreasing time from gene to structure, material usage, and number of personnel needed to accomplish large number of tasks. These factors also shorten feedback loops between processes leading to an almost two-fold decrease in time to arrive at a structure.

New tools for the HT pipeline include development of robust micro-expression protocols, use of NMR micro-probe for rapidly identifying protein targets amenable to further structural studies, use of nanocalorimetry or enthalpy array for biophysical characterization of interactions, and miniaturization, automation of the crystallization experiment, and use of workflow and GRID technologies for use in computational tasks in HT structure determination efforts.

Keywords: structural genomics, high-throughput structural biology, biophysical characterization

P.01.15.8

Acta Cryst. (2005). A61, C150

High-resolution Data using short Wavelength Confocal Optics and a Cylindrical IP

Lee Daniels, Joseph Ferrara, Ron Benson, *Rigaku/MSB, Inc., 9009 New Trails Dr., The Woodlands, TX, 77381 USA.* E-mail: ldaniels@RigakuMSB.com

Combination of modern techniques for allows for new laboratory experiments that were previously inaccessible without specialized equipment. A new confocal optic for silver (Ag) radiation coupled with a rotating anode allows for greatly increased intensities at the sample position from this very short wavelength radiation.

By coupling this source with a cylindrical image-plate detector, very high resolution (high-Q) diffraction data can be obtained from a variety of samples, including single-crystal, powder, thin films, etc.

Several experiments are described and the results from high-resolution experiments are discussed.

Keywords: X-ray optics, high-Q, Ag radiation