robust mechanized experimental hardware, a flexible instrumentation control system with an intuitive user interface [1] and efficient integration of data collection and data analysis.

A key component of the system is the Stanford Auto- Mounter (SAM), which can mount 198 samples without any manual intervention [2]. The robot, in combination with other automated tasks, allow crystallography experiments to be carried out from the researchers' home institutions and other remote locations while retaining complete control over the experiment. Full remote access was implemented in 2005. Currently close to 80% of the user groups collect data totally remotely [3].

Remote access to the SSRL computers is done via the NX client application provided by NoMachine, which provides a response close to that obtained at the beamline when a broadband connection is used. In addition, a web application, Web-Ice, can be used to analyze test diffraction images, calculate data collection strategy and carry out data processing [4].

The latest efforts have focused on developing specialized workflows to fully automate highly iterative experiments (such as fragment-based drug search or mutant comparisons). To achieve this goal, a declarative programming language, RestFlow, has been developed. RestFlow facilitates the integration and sharing of scripts and programs by different workflows. Currently, a workflow automating all the steps from sample screening and selection to model refinement is under development.

[1] T.M. McPhillips, S.E. McPhillips, H.-J. Chiu, A.E. Cohen, A.M. Deacon, P.J. Ellis, E. Garman, A. González, N.K. Sauter, R.P. Phizackerley, S.M. Soltis, P. Kuhn, *J. Synchrotron Rad.* **2002,** *9,* 401. [2] A.E. Cohen, P.J. Ellis, M.D. Miller, A.M. Deacon, R.P. Phizackerley, *J. Appl. Cryst.* **2002,** *35,* 720. [3] S.M. Soltis, A.E. Cohen, A. Deacon, T. Eriksson, A. González, S. McPhillips, H.J. Chui, P. Dunten, M. Hollenbeck, I. Mathews, M. Miller, P. Moorhead, R.P. Phizackerley, C. Smith, J. Song, H. van dem Bedem, P. Ellis, P. Kuhn, T. McPhillips, N. Sauter, K. Sharp, I. Tsyba, G. Wolf, *Acta Cryst.* **2008,** *D64,* 1210. [4]A. González, P. Moorhead, S.E. McPhillips, J. Song, K. Sharp, J.R. Taylor, P.D. Adams, N.K. Sauter, S.M. Soltis, *J. Appl. Cryst.* **2008,** *41,* 176-184.

Keywords: automation, remote experiment, macromolecular crystallography

MS.13.3

Acta Cryst. (2011) A**67,** C46

Automation and remote access at SPring-8 MX beamlines

Kazuya Hasegawa,[a] Go Ueno,[b] Takaaki Hikima,[b] Hironori Murakami,[b] Kunio Hirata,[b] Yukito Furukawa,[a] Takashi Kumasaka,[a] and Masaki Yamamoto,[b] [a]*Structural Biology Group, SPring8/JASRI (Japan).* [b]*SR Life Science Instrumentation Unit, RIKEN SPring-8 Center (Japan).* E-mail: kazuya@spring8.or.jp

Data collection using synchrotron beamline is indispensable for the structure biology research nowadays. At SPring-8, we provide an opportunity to use high intensity synchrotron radiation facility with robust and automated data collection system, which contributes to both high throughput crystallography and a cutting edge research.

The automation is achieved by an integrated beamline control software *BSS* [1] and a sample changer *SPACE* [2]. For the high throughput crystallography, we use two-mode operation which composed of daytime attended crystal screening and night time fully automated data collection. The scheduling function of *BSS* and a screw type special sample pin handled by *SPACE* enables this operation. Mail-in data collection using this two mode operation is routinely conducted since 2005, where database *D-Cha* manages information such as sample information and data collection conditions [3]. For the cutting edge research, which handles small crystals or low-quality crystals in most cases, *BSS* is equipped with tools assisting data collection, such as helical scan to avoid the radiation damage, and grid scan for the centering of microcrystals and screening a well diffracting part of inhomogeneous crystals.

Based on this automation system, we now introduce a remote access data collection system. We did not use a remote desktop, but developed a robust and secure remote system adopting the server/client architecture. A newly developped remote client GUI program installed on the user's PC communicates with *BSS* for the remote operation. The remote access is only allowed for anauthenticated user under the beamline interlock permission. This architecture not only keeps safety and security but also enables users a stress free operation.

We expect that the beamline automation and the remote access increase the borderless use of the beamlines and contribute to the structural biology.

[1] G. Ueno, H. Kanda, T. Kumasaka, M. Yamamoto, *J. Synchrotron Rad.* **2005**, *12*, 380–384. [2] G. Ueno, R. Hirose, K. Ida, T Kumasaka, M. Yamamoto, *J. Appl. Cryst.* **2004**, *37*, 867–873. [3] N. Okazaki, K. Hasegawa, G. Ueno, H. Murakami, T. Kumasaka, M. Yamamoto, *J. Synchrotron Rad.* **2008**, *15*, 288–291.

Keywords: synchrotron, automation, instrumentation

MS.13.4

Acta Cryst. (2011) A**67,** C46-C47

Automated synchrotron crystallography for drug discovery: The LRL-CAT beamline at the APS

Stephen R. Wasserman,[a] Michael M. Holdmann,[b] John W. Koss,[a] David Lewis,[b] Laura L. Morisco,[a] Sonal T. Sojitra,[a] Kenneth D. Visscher,[b] and Stephen K. Burley,[c] [a]*LRL-CAT, Eli Lilly and Company, Advanced Photon Source, Argonne National Laboratory, 9700 S. Cass Ave, Building 438A, Argonne, IL 60439 (USA).* [b]*Eli Lilly and Company, Lilly Corporate Center, Indianapolis, IN, 46285 (USA).* [c]*Lilly Biotechnology Center, 10300 Campus Point Drive, San Diego, CA 92121 (USA).* E-mail: swasserman@lilly.com

The Lilly Research Laboratories X-ray beamline, LRL-CAT, sited at Sector 31 of the Advanced Photon Source (APS), is a highly-automated facility supporting rapid acquisition of diffraction data from protein crystals. The facility is designed to operate with minimal human intervention and provide medicinal chemistry teams with timely access to protein co-crystal structure information. All LRL-CAT operations can be controlled remotely, including X-ray beam alignment, wavelength calibration, and crystal mounting, screening, and data collection.

In 2010 the LRL-CAT end station underwent a complete upgrade. New cryogenic crystal mounting robotics, including a custom CATS system with a capacity of 540 samples, were installed to permit long-term unattended operation. The upgrade also included installation of new beam-defining components, a high-speed, air-bearing goniostat, and piezoelectric nano-positioners for precise placement of crystals within X-ray beams of 20-150μm diameter.

All beamline operations are controlled through custom software directly linked to a proprietary Laboratory Information Management System (LIMS) built atop an Oracle® database. Crystal quality and diffraction limit are evaluated automatically from recorded screening images. Samples meeting predefined quality criteria are selected for data collection by database-driven software. The software ensures that collection within a group of duplicate crystals is limited to those that will provide the best data. Data collection and initial processing then proceeds without human intervention. Additional software programs evaluate the scaled data to guarantee its quality prior to transmission from the beamline.

The upgraded LRL-CAT facility now supports screening of up to

360 samples per day or collection of up to 130 X-ray diffraction datasets per day. Turnaround of structural data from LRL-CAT averages less than 2.3 days from the time the crystal was created and shipped to the beamline.

Keywords: automation, synchrotron, protein

MS.13.5

Acta Cryst. (2011) A**67,** C47

Optimising X-ray experiment strategy on-the-fly based on feedback from automated structure solution

Richard Cooper, *Department of Chemistry, University of Oxford, Chemistry Research Laboratory, Mansfield Road, Oxford (UK).* E-mail: richard.cooper@chem.ox.ac.uk

The EDNA framework [1] is helping us to develop and run an automated structure solution and refinement pipeline during small molecule X-ray data collections. The partial results can be used to modify the X-ray experiment strategy on-the-fly, and so target the data which provides the best possible answer to a specific problem.

X-ray experiment strategies typically optimise the time taken to collect a unique set of data (within specified limits) followed by efficient collection of data to provide maximum possible redundancy within a given time. However - if the structure can be determined and refined early in the data collection process - information can potentially be fed back to modify the data collection in order to collect the most valuable or useful data in the remaining time. Analysis of the least-squares fit of the model to the data can quickly determine the few observations which may be profitably re-measured to most improve the estimated variance of a given model parameter [2]. Extension of this analysis allows the identification of observations whose re-measurement will most improve the estimated variance of arbitrary functions of parameters (for example: distances; angles; planes; sums and differences of occupancies *etc*.).

If the crystal structure is as expected, the experimentalist can specify particular parameters or functions of interest, in order to generate an updated data collection strategy for the remainder of the experiment.

[1] M.-F. Incardona, G.P. Bourenkov, K. Levik, R.A. Pieritz, A.N. Popov, O. Svensson, *J. Synchrotron Rad.* **2009**, *16,* 872-879. [2] E. Prince, *Mathematical Techniques in Crystallography and Materials Science*, **2004,** 3rd Ed, 122.

Keywords: automated, experiment design, least-squares analysis

MS.14.1

Acta Cryst. (2011) A**67,** C47

In search of Nature's secrets – Controls on biomineralisation

S.L.S. Stipp,[a] T. Hassenkam,[a] K. Sand,[a] M. Yang,[a] N. Bovet,[a] L. Schultz,[a] K.E. Henriksen,[b] [a]*Nano-Science Centre, Department of Chemistry, University of Copenhagen, (Denmark).* [b]*Maersk Oil and Gas A/S, Copenhagen, (Denmark).* E-mail: stipp@nano.ku.dk

Calcite, rhombohedral $CaCO_3$, forms easily in nature, as stalagmites and stalactites, as veins in rocks, as sediments in warm seas and in pipes that distribute water to our homes. In veins, it can form large, beautiful crystals, but even in supersaturated laboratory solutions, the most favoured crystal form is the rhomb. Many organisms produce calcite, such as oysters and coral. Earthworms excrete tiny spherules, wood lice store it on their tummies and perhaps the simplest organism, one-celled algae, create exquisite platelets called coccoliths from 20 to 60 individual calcite elements that are less than a micrometer in the longest dimension. As a biomineral, calcite rarely takes the form of a rhomb. Organisms produce complicated organic molecules that adhere to the calcite surface, inhibiting growth at some sites, thus enhancing it at others. Depending on the ecological niche the organism inhabits, it can tailor the calcite to its specific needs.

We learn more about biomineralisation by studying the mineral remains of organisms and by experimenting in ideal systems, where we can control one variable after the other. The calcite atomic structure orders water in contact and even simple compounds such as ethanol, the shortest organic chain molecule that has both a methyl group and a carboxyl group, binds strongly and orders itself. Polysaccharides, produced synthetically, or extracted from cultured coccoliths, have the power to control crystallization in the manner determined by the organism. Through a multidisciplinary approach, combining skills from physics, chemistry, geology, biology and mineralogy, from experimental and theoretical directions, from the field scale to the nanometer scale, and by studying nature and designing model systems, we are getting closer to understanding the secrets of simple organisms.

Keywords: biomineralisation, calcite, polysaccharide

MS.14.2

Acta Cryst. (2011) A**67,** C47

Biological and environmental influence on Mediterranean corals calcium carbonate precipitation

Giuseppe Falini,[a] Stefano Goffredo,[a] Patrizia Vergni,[a] Michela Reggi,[b] Erik Caroselli,[b] Francesca Sparla,[c] Oren Levy,[d] Zvy Dubinsky,[d] [a]*Dipartimento di Chimica 'G. Ciamician', via Selmi 2 40126 Bologna, (Italy).* [b]*Marine Science Group.* [c]*Dipartimento di Biologia Evoluzionistica Sperimentale, via Selmi 3, 40126 Bologna, Alma Mater Studiorum Università di Bologna, (Italy).* [d]*The Mina and Everard Goodman Faculty of Life Sciences, Bar Ilan University, Ramat Gan 52900, (Israel).* E-mail: giuseppe.falini@unibo.it

Scleractinian coral skeletons are made mainly of calcium carbonate in the form of aragonite. The mineral deposition occurs in a biological confined environment, but it is still a theme of

discussion to what extent the calcification occurs under biological or environmental control. Hence, the shape, size and organization of skeletal crystals from the cellular level through the colony architecture, have been attributed to factors as diverse as mineral supersaturation levels and organic mediation of crystal growth. The skeleton contains an intra-crystalline organic matrix (OM) of which only the water soluble component has been chemically and physically characterized. In this work that OM from the skeleton of the *Balanophyllia europaea* and *Leptopsammia pruvoti*, solitary scleractinian corals endemic to the Mediterranean Sea, is studied in vitro with the aim of understanding its role in the mineralization of calcium carbonate. Mineralization of calcium carbonate was carried out in calcium chloride solutions containing different ratios of water soluble and/or insoluble OM and of magnesium ions. The precipitates were characterized by diffractometric, spectroscopic and microscopic techniques. The results showed that both soluble and insoluble OM components influence calcium carbonate precipitation and that the effect is enhanced by their co-presence. The role of magnesium ions is also affected by the presence of the OM components. Thus, in vitro, OM influences calcium carbonate crystal morphology, aggregation and polymorphism as a function of its composition and of the content of magnesium ions in the precipitation media. This research, although does not resolve the controversy between environmental or biological control on the deposition of calcium carbonate in corals, sheds a light on the role of OM, which appears mediated by the presence of magnesium ions.

Keywords: coral, biomineralization, organic matrix