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Effect of high-pressure / low temperature on cysteine, its salts and derivatives

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Structure-property investigation of crystalline amino acids is an important challenge since interactions between individual molecular fragments or even structural domains in the structure can simulate interactions in more complicated biological systems such as proteins and peptides. Besides, crystalline amino acids are applied as drugs, as piezoelectric and nonlinear optical materials. Therefore understanding a crystal structure response to variation in temperature and pressure is significant in such applications.

Cysteine is a remarkable amino acid because its side-chain residue contains a sulfhydryl group involved in formation of additional labile hydrogen bonds (S-H…S or S-H…O). The presence of these very weak bonds in the structure allows cysteine to take a peculiar place between hydrophobic (no contribution of side-chains to H-bonds) and hydrophilic amino acids (with that contribution).

In the present contribution we discuss an evolution of chiral and racemic cysteine crystal structures on cooling and on increasing pressure followed by X-ray crystallography and Raman spectroscopy. We also compare their behavior with that of cysteine crystalline salts and derivatives.

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NH···N hydrogen bonds in high-pressure phase of imidazole

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Imidazole is a prototypic compound were molecules in crystal are linked in infinite NH···N bonded chains. At ambient conditions and low temperatures imidazole forms monoclinic structure (space group P21/c) [1-4]. High-pressure of 0.8 GPa transforms imidazole into a new phase, with a planar arrangement of molecules. This structure has been studied by high-pressure X-ray diffraction and infrared spectroscopy. The results have been interpreted in order to explain the structural differences and the formation of polar order.

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Features and development of BEST

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Program BEST for optimal planning of X-ray diffraction measurements from macromolecular crystals is based on modeling the statistical results of data collection taking radiation damage effects into account [1]. Furthermore, tools are provided for automatically characterization of the radiation sensitivity of macromolecular crystals [2] as well as characterization of crystal diffraction quality for the advanced sample evaluation [3]. In recent years, BEST became integrated in automated data collection and on-line data analysis systems (EDNA/MxCuBE, Web-Ice, GDA, CBASS and others) as a standard strategy module. We will present a review on BEST strategy implementations and current applications. Along with the automation and high throughput applications, BEST strategies appear particularly useful for difficult, e.g. small, weakly or very anisotropically diffracting and radiation sensitive crystals. In many experiments, though still with some involvement of human intelligence, the software was helpful in designing an optimal strategy for collecting a data set using small beams and multiple crystal centerings, or multiple crystals. Recent developments in BEST provide the tools for generalization and automation of such experiments, as well as for experiments utilizing ultrafast area detectors and planing of data collection at room temperature.

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Remote access and automation at SSRL

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Complete automation of the macromolecular crystallography experiment has been achieved at SSRL through the combination of

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.

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Keywords: automation, remote experiment, macromolecular crystallography

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Automation and remote access at SPring-8 MX beamlines

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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.

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

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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