starting dehydration temperatures of the CNSH and RNSH crystals are 111°C and 126°C respectively. Real structure of CNSH and RNSH crystals was studied by projection X-ray topography.

Keywords: crystal growth, optical properties, structural analysis

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Protein Crystal Growth in Planar and Integrated Gel Interface Diffusion Device

Akira Sanjoh, Ritsuko Tanimura, Masakazu Moro'oka, *R&D Center, Protein Wave Corporation, Kyoto, Japan.* E-mail: asanjoh@pro-wave.co.jp

A liquid-gel-liquid counter diffusion capillary system has successively used for macromolecular crystallization both on ground and in microgravity environment [1], [2]. This system however shows slow kinetics of crystal growth and non-uniform deposition of crystals in a capillary, which mainly arise from one directional (vertical) mass transport of precipitant reagent into protein solution. This is also not provided for the purpose of high-throughput crystallization/X-ray diffraction data collection experiments, due to the difficulty of handling of each glass capillary.

A novel type of gel counter diffusion device is developed for both purposes of conventional and high-throughput protein crystallization. Precipitant and protein cells are separated by thin gel layer(1-2mm in thickness), and are horizontally arranged in a planar well pate. Protein crystals grown in the protein cell in which dimensions of width/length are about 1mm/100mm, are easily observed by CCD camera and also accessed by loop device so as to mount them. To improve the non-uniform deposition of protein crystals during the counter diffusion process, inert liquid layer [3] is embedded beneath the protein solution cell. Without circulation of the inert liquid layer, protein crystals are distributed uniformly over the whole range of protein solution cell.

[1] Garcia-Ruiz J.M., et al., *J. Crystal Growth*, 2001, **232**, 165. [2] Maes D., et al., *Acta Cryst.*, 2004, **D60**, 463. [3] Adachi H., et al., *Jpn. J. Appl. Phys*., 2002, **41**, L1025.

Keywords: crystallization of proteins, crystallization methods, structural genomics

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How to Optimize Gel-tube Method

Mari Yamanaka[a], Hiroaki Tanaka[a], Koji Inaka[b], Masaru Sato[c], Sachiko Takahashi[a], Shigeru Sugiyama[b], Satoshi Sano[c], Moritoshi Motohara[c], Tomoyuki Kobayashi[c], Susumu Yoshitomi[c], [a]*Japan Space Forum.* [b]*Maruwa Food Industries, Inc.* [c]*Japan Aerospace Exploration Agency.* E-mail: m-yamanaka@jsforum.or.jp

'Gel-Tube' is a method for a protein crystallization using a simplified counter-diffusion technique as we previously reported [1]. A gel in a silicon tube, through which protein and precipitant solution diffuse each other from different direction, is attached to the end of a capillary. Crystallization experiments with a wide range of conditions can be carried out by only one capillary unless crystallization occurs, which suggests that there is a higher possibility for obtaining crystals by a single experiment than conventional method such as vapour-diffusion. Moreover, if combined with 1-dimensional (1-D) simulation program to know concentration change of protein and precipitant solution in a capillary, it is possible to estimate the concentration of protein and precipitant in a certain position of a capillary when crystallization occurred, so that optimization of crystallization condition for further crystallization experiment can be performed. According to the results of the simulation, polyethylene glycol (PEG) might work well in Gel-Tube method as a precipitant because it diffuses so slowly that crystal grows gradually. The effects of the gel-tube length, sample length in a capillary and type of precipitant will be shown.

[1] Tanaka H., et al., *J. Synchrotron Rad*, 2004, **11**, 45-48.

Keywords: crystal growth apparatus design, counter-diffusion, gel-tube

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Optimized Crystallization Solution Analyzed from JAXA Cryoprotectant Database

Sachiko Takahashi[a], Hiroaki Tanaka[a], Masaru Sato[b], Moritoshi Motohara[b], Satoshi Sano[b], Tomoyuki Kobayashi[b], Susumu Yoshitomi[b], [a]*Japan Space Forum.* [b]*Japan Aerospace Exploration Agency.* E-mail: s-takahashi@jsforum.or.jp

Techniques for cryofreezing protein crystals are essential for X-ray diffraction experiment to reduce radiation-induced damages caused by X-ray beam in synchrotron facility. However, even though high optical quality crystals are obtained, inappropriate way of adding cryoprotectant to mother liquor often causes deterioration of crystal quality.

If the mother liquor is also suitable for cryoprotectant, the damage caused by soaking in artificial mother liquor before diffraction analysis could be avoided. Therefore, we picked up data from International Space Environment Utilization Research Data Base (ISRDB) (http://idb.exst.jaxa.jp/) constructed by Japan Aerospace Exploration Agency (JAXA) and analyzed crystallization solution data which are effective both in crystallization and cryoprotection. We will show the results which will be useful both for crystallizing and for cryofreezing protein crystals without any damages.

Keywords: cryoprotectant database, X-ray diffraction, statistical analysis experimental data

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Optical and Morphological Properties of Lead Sulphide (PbS) thin Films

Samira Kaci, Mohammed Boudjelab, *Unité de Développement de la Technologie du Silicium 2, Bd Frantz Fanon PO Box 399 Alger Algérie.* E-mail: samira_dz20022002@yahoo.fr

In this work we studied the optical and morphological properties of thin films of lead sulphide (PbS). Lead sulfide (PbS) films have been deposited by chemical deposition method on glass substrates from a solution of lead acetate, Pb $(CH_3COO)_2$, and thiourea, $SC(NH_2)_2$ diluted in water. The deposition is performed in alkaline medium, using sodium hydroxide (NaOH), the starting solution pH being 11. The advantage of this method is simple, relatively inexpensive and easily controlled method that is producing large area films. Some data about the optical properties, structure, composition of the films and thermal stability of the powder samples have been reported.

The structure and crystallite sizes were determined by X-ray diffraction studies. The optical properties were obtained using Fourier transforms infrared (FTIR) spectroscopy. The films are very adherent to the substrates and are polycrystalline. The surface morphology of the as deposited films was studied with a scanning electron microscope. From two to ten, multiple layers have been deposited. The terminal thickness has been determined. Experiments [1] showed that the shape of the product depended on the initial reactants. Under the same experiment condition, $PbCl_2$ and Pb $(NO_3)_2$ were employed as the lead ion source instead of Pb $(CH_3COO)_2$.

[1] Yonghong Ni, Hongjiang Liu, Fei Wang, Yongye Liang, Jianming Hong, Xiang Ma, Zheng Xu, *Cryst. Res. Technol*., 2004, **39**, 3, 200.

Keywords: lead sulfide, thin films, chemical bath deposition

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The Effect of Polyelectrolytes on Nano Hydroxyapatite Crystal Growth

Özlem Doğan, Mualla Öner, *Yildiz Technical University, Chemical Engineering Department, Davutpasa Campus, Esenler 34210 İstanbul, Turkey.* E-mail: dogano@yildiz.edu.tr

The precipitation and dissolution of calcium phosphate salts is of particular interest because of its importance in industrial water systems, in waste water treatment processes, in agriculture as fertilizers and in biological calcification processes [1]. Under

physiological conditions the most stable calcium phosphate is hydroxyapatite ($Ca_5(PO_4)_3OH$, HAP). The growth mechanism of HAP has received considerable attention in view of its importance in understanding the mechanism of hard tissue calcification such as bone and teeth and in many undesirable cases of pathological mineralization of articular cartilage, dental caries and kidney stones [2].

In this work that we investigate the individual effect of polymeric additives for the hydroxyapatite (HAP) crystallization as a model for biomineralization. The higher affinity of PAA for HAP corresponds to the more significant effect of this polymer on the rate of HAP crystal growth.

The results indicate that polyelectrolyte concentration and the larger number of negatively charged functional groups markedly affect the growth rate. The fit of the Langmuir adsorption model to the experimental data supports a mechanism of inhibition through molecular adsorption of polymers on the surface of growing crystals.

[1] Amjad Z., *J. Colloid and Interface Science*, 1987, **117**, 98. [2] Koutsopoulos S., Dalas E., *J.Crystal Growth*, 2000, **217**, 410.

Keywords: hydroxyapatite, biomineralization, crystallization

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Morphological Control of Calcium Oxalate by Hydrophilic Block Copolymers

Emel Akyol, Mualla Öner, *Department of Chemical Engineering, Yildiz Technical University, Davutpasa, Istanbul 34210, Turkey*. E-mail: eakyol@yildiz.edu.tr

Biomineralization processes have attracted considerable attention due to their importance in life sciences, especially with respect to pathological effects[1]. On the aspect of biomineralization, it is of interest to study the crystallization of calcium oxalate monohydrate (COM), because COM crystals have been known as a possible source of urinary and kidney stones[2]. Previous works have shown that the significant influence of urinary macromolecules on calcium oxalate crystallization[3]. Both inhibition and promotion of crystal growth and crystal aggregation by these biopolymers has been reported [4]. An understanding of biological solid-state interactions would be of immense value in many areas.

In this study, we prepared a range of acrylic polymers with different architectures to explore their relative effectiveness in inhibiting crystal growth of calcium oxalate. We investigated the effect of polymers on the particle size, morphology and precipitation of crystals. The presence of copolymers inhibited the crystal growth of calcium oxalate possibly through adsorption onto the active growth sites for crystal growth due to the charge and hydrophilic effects.

[1] Mann S., *Biomineralization: Principles and Concepts in Bioinorganic Materials Chemistry*, Oxford University Press, 2001. [2] Jung T., Kim W.S., Choi C.K., *Materials Science and Engineering C*, 2004, **24**, 31-33. [3] Bramley A.S., Hounslow M.J., Ryall R.L., *Chemical Engineering Science*, 1997,**52**, 747-757. [5] Konya E., Umekawa T., Iguchi M., Kurita T., *European Urology*, 2003, **43**, 564-571.

Keywords: calcium oxalate, crystallization, morphology

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Searching the Crystallisation Parameter Space using Evolutionary Algorithms

Naomi E. Chayen[a], Emmanuel Saridakis[a,b], [a]*Biomedical Sciences Division, Imperial College Faculty of Medicine, London SW7 2AZ, U.K.*. [b]*School of Health Sciences, T.E.I. – Athens, GREECE.* E-mail: e.saridakis@imperial.ac.uk

When trying to crystallize a new protein, the researcher usually explores a multi-dimensional parameter space using a sparse-matrix or other type of screen. Frequently, the results of such a search consist in a small number of 'promising' conditions. The researcher then conducts a finer mesh search, centered at each of the 'promising' points of the parameter space. If this fails to produce diffracting crystals, other screening conditions must be thought up.

We propose the further probing of such 'promising' conditions, using small-scale Evolutionary (Darwinian) Optimisation Algorithms. Each promising condition is pictured as a 'chromosome', the values of the various parameters (type of precipitant, buffer, pH, temperature, ...) being the alleles on that chromosome. The original 'promising' conditions of the screen constitute a 'first generation' of experiments. A second generation is constituted by random 'recombination' of these 'alleles', i.e. by combining successful values of parameters from different conditions. The most successful of the second generation of experiments will in turn be the 'parent conditions' of a third generation. 'Mutations', i.e. as yet untried values of parameters, can be sparsely introduced in each generation.

This method will not be as robust as for the purely computational optimisation problems for which it is normally used, due to the limitations on the number of 'generations'. It can however lead to optimal combinations of parameters, provided judicious choice of the conditions that will be the parents to each successive generation.

Keywords: biocrystallization, crystallogenesis, crystallization strategies

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Crystallization Platform Integrating Screening & a Novel Optimization Strategy

Dietrich Suck, *EMBL Heidelberg, Meyerhofstrasse 1, D-69117 Heidelberg, Germany*. E-mail: suck@embl.de

Obtaining diffraction quality crystals is a common bottleneck in macromolecular crystallography. With the number of projects increasing exponentially, searching for the right crystallization conditions is a time consuming effort. We have now setup a medium-throughput crystallization platform at the main laboratory in Heidelberg in order to centralize resources, cut costs, and provide efficient and rapid service to EMBL-Heidelberg research groups using X-rays.

The platform is based on use of a nanoliter dispensing robot, standardized crystallization screens with a total of 1800 different conditions, and a database linked to an imaging system for data archiving. The advantages of the service are multiple. It significantly reduces crystallization setup costs by using fewer crystallization solutions and fewer samples. It also saves valuable time. What started as a medium throughput crystallization platform has rapidly grown and 300,000 crystallization drops have already been set up.

As a standard and simple approach for crystal optimization, we are now using the prefilled Nextal Opti-Salts crystallization microplates. This tool allows us to rapidly generate our own new subset of conditions expanded around the hits obtained at initial screening. It works using a combinatorial optimization approach. We will present three projects where the Opti-Salts generate a significant improvement with minimum efforts and investment.

Keywords: crystallization robots, optimization, biomacromolecular crystallization

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$Li_{3.17}(P_{0.69}Ge_{0.24}Mo_{0.07})O_4$: Growth under Electrical Field and the Structure

Dmitry A. Ksenofontov[a], Ludmila N. Dem'yanets[a], Natalia V. Zubkova[b], Alexei K. Ivanov-Schitz[a], Dmitry Yu. Pushcharovsky[b], [a]*Institute of Crystallography RAS, Moscow, Russia.* [b]*Geology Department, Moscow State University, Moscow, Russia.* E-mail: ksenofant@rambler.ru

The influence of electrical field on crystal growth from flux has been studied in the system Li_3PO_4-Li_4GeO_4-Li_2MoO_4-LiF. Growth occurs on the Pt-rod (anode) immersed into the flux due to temperature decrease with the simultaneous application of direct electrical current. The starting molar ratio between the starting components of the system (Li_3PO_4 : Li_4GeO_4 =1: 1) corresponded to the ratios which provided the stable crystallization of solid solution $Li_{3+x}P_{1-x}Ge_xO_4$ with x=0.31 at the absence of electrical field. When electrical current (V=0.08V) was applied to the growth system, the