

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

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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

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'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

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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

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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<sub>3</sub>COO)<sub>2</sub>, and thiourea, SC(NH<sub>2</sub>)<sub>2</sub> 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<sub>2</sub> and Pb(NO<sub>3</sub>)<sub>2</sub> were employed as the lead ion source instead of Pb(CH<sub>3</sub>COO)<sub>2</sub>.

[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

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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