P.16.01.1
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Use of reductive methylation of proteins to increase crystallization efficiency at the Midwest Center for Structural Genomics
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The highest attrition rate in high-throughput structural genomics (SG) projects utilizing x-ray crystallography occurs at the step of obtaining diffraction quality crystals. The Midwest Center for Structural Genomics (MCSG) has developed and continues to optimize the high-throughput crystallization pipeline. The pipeline was set up with nano-liter crystallization robots such as Honeybee and Mosquito, and a Matrix Maker equipped with Crystal Monitor software for screening initial crystallization conditions and crystal optimization. As a part of effort to increase the success rate of obtaining diffraction quality crystals, chemical modification of proteins has been tested in the MCSG crystallization pipeline. Particularly, reductive methylation of lysine residues to alter the crystallization properties has been evaluated with more than 100 proteins, most of which have not been crystallized previously. Following the method described by Ivan Rayment (in Volume 276 of *Methods of Enzymology*) the proteins were methylated and screened using standard MCSG crystallization pipeline. Several structures have been obtained using this approach and detailed analysis and progress will be presented.
This work was supported by the by grants from the National Institutes of Health (GM62414) and the U.S. Department of Energy, Office of Biological and Environmental Research, under Contract W-31-109-ENG-38.
Keywords: reductive methylation, structural genomics, attrition rate

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Implementation of a Novel Protein Crystal Image Analysis Package
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Crystal Resolve is automated image analysis software for protein crystallography. It is offered as an add on package for the Crystal Farm product line.

Crystal Resolve uses proprietary high precision subpixel image processing and object recognition algorithms to decompose an image into thousands of significant micro features. It applies specific knowledge of the drop type and plate geometry along with neural networks and advanced heuristic methods to classify these features while developing a macroscopic comprehension of image contents. From this, it computes two key metrics. F_C describes how crystal-like (0.0-1.0) the image is. F_P describes how precipitate-like the image is.

For all its complexity, Crystal Resolve is very easy to use. Users are given the flexibility to choose which images should be reviewed in terms of their F_C and F_P values. Crystal Resolve easily identifies images with no interesting content, greatly saving researchers' time.

Crystal Resolve benefits projects with hard to find crystals by allowing users to select and view images with poorer F_C figures. Crystal Resolve frequently finds objects that some crystallographers miss while not getting confused by dust, bubbles, and plate features.

Some examples of analyzed images from different crystallization plates will be presented.
Keywords: protein crystallization, imaging, protein crystal growth

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AFM Observation and Morphing-reconstruction of Dynamics of Crystal Growth
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With *in situ* investigations by an atomic force method (AFM) and using statistical approach we studied evolution of the crystal surface of sodium chloride in the solution. The determined mean values of tangential rate and distances between the steps for every interval showing a normal rate of the growth of the scanning area associating with thermodynamic conditions of growth [1]. Tangential rate of movement of each step is calculated by comparison of two successive snapshots. But not always the following AFM snapshot can be wrong for such comparison with the previous. Or statistics requires recovering a picture of the surface at the "offscreen" interval between two snapshots that is 40 sec. In crystallogenetic investigations there is often necessity for dynamic reconstruction of the occurring processes. Currently, there is a number of graphic programs as morphing programs enabling to estimate up to 100 intermediate images and thus, to obtain an image of a surface each 0.4 sec and to use the estimated images for statistical calculation. Moreover, it is important that all images can be combined as motions. We have made several demo videotapes with duration of 1 to 6 minutes used at the lectures for university students as visual aids showing peculiarities of development of the surface structure, formation of solution inclusions, surface growth in the directed flow of the solution.
This work was supported by RFBI (project №05-05-65112a).

[1] Piskunova N.N., Rakin V.I., *Proceedings of ICCG-14*, Grenoble, 2004, **347**.
Keywords: AFM, crystal growth from solution, surface morphology

P.16.03.2
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Catalysed Growth of Doped TGS Single Crystals with Pt(IV) Ions
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The exploitation of the ferroelectric triglycine sulfate, $(NH_2CH_2COOH)_3.H_2SO_4$ (TGS) in some technical applications [1] has increased the need for high-quality single crystals with stabilized domain structure [2].

A novel type of full facetted single crystals of triglycine sulfate with various contents of Pt(IV) complex-forming ions and L-alanine, D-phenylalanine or L-arginine were grown from aqueous solutions. Morphology, domain structure and *P-E* hysteresis loops have been investigated.

On the wafers prepared from <110> pyramids we measured the main physical properties, in particular: spontaneous polarization P_s, coercive field E_c, internal electrical field E_b, dielectric permittivity ε_r, and dielectric losses $\tan\delta$.

The effect of the dopant on the growth velocity is explained on the basis of catalytic action of supposed platinum complexes [3]. The value of E_b, important for technical applications of grown crystals, can be adjusted by Pt(IV)-ions concentration in the growth solution.
The work was supported by the Academy of Sciences of the Czech Republic under projects No. S2067204 and COST Action P11 as well as by the COST D14.40.

[1] Whatmore R.W., *Rep. Prog. Phys.*, 1986, **49**, 1335. [2] Neumann N., *Ferroelectrics*, 1993, **142**, 83. [3] Novotný J., Březina B., Zelinka J., *Cryst. Res. Technol.*, 2004, **39**, 1089.
Keywords: crystal growth, ferroelectric crystals, domains

P.16.03.3
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Crystallization of Water-soluble Inorganic Salts in Microwave Field
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Influence of microwave field on the origin and crystal growth from water solutions was studied. Crystallization by methods of temperature gradient and evaporation of solvent of some inorganic salts (KH_2PO_4 (KDP), NaCl, $Sr(NO_3)_2$, KNO_2, $Ca(OH)_2$) is investigated. It is established, that growth rates of single crystals in microwave field are much more higher in comparison with growth with the use of other known technologies under the same temperatures and supersaturations. For example growth rate of a prism {100} of KDP crystals reaches 11 mm/day with supersaturations ~ 1.2 %, and temperature 70ºC. Fine dispersion crystallites of investigated salts were obtained by evaporation of solvent.

Use of microwave field for heating of crystallization water solutions leads to significant increase of crystal faces growth rate.

Microwave field more actively destroys adsorption and diffusion layers on crystal faces in comparison with other methods (mechanic, ultrasonic, etc.) in water solutions, providing more intensive moving of substance in superficial area.

At mass crystallization by evaporation of solvent microwave radiation promotes significant decrease of crystallite size. More distinctly it is shown for hard soluble compounds.

Microwave technique provides uniform heating of the whole crystallization volume with active hashing of the solution and simultaneous origin of a significant amount of crystallites over the whole volume of a crystallizer.

Keywords: crystal growth, microwaves, solutions

P.16.03.4

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Monitoring Polymorphic Transformations in Solution

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Applying diffraction techniques to the study of crystallisation from solution is a way to study the process of crystallisation under different solvent conditions, supersaturation and cooling regimes. For polymorphs systems this approach provides a means to map the stability of one polymorph in relation to another during the crystallisation process in real time. An issue is the trade off between solid diffraction and solution scatter on the overall pattern obtained, and specifically the overall signal to noise.

Even using the light intensity from a synchtron the diffraction from the solid phases present, remains buried in thesignal due to the solution scattering the x-rays. A novel clarifying crystalliser has been developed which by virtue of the design forms a plume of solid for the beam to probe. Thereby increasing the weight fraction of solid pressnted to x-ray beam thus the overall signal to noise obtained from the solid present. To date the crystallisation of urea, citric acid, glutamic acid and piracetam has been studied using the cell on station 16.4 at the SRS Daresbury, and the outcome for these systems will be presented. These examples systems highlight how it has been possible to monitor the evolution of morphology, induction times and the rates of inter conversion from one polymorphs to another.

Keywords: polymorphism, insitu diffraction, crystal growth

P.16.03.5

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Growth of the KDP filamentary crystals from solution with impurities

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The filamentary crystal formation is one of the qualitative indicators of an impurity preferential adsorption.

The effect of different trivalent metal ions impurity on growth of the KDP filamentary crystals at high concentrations (0.4-1.5 g/l) and supersaturations (50-90%) of a solution has been studied. The first stages of whiskers growth formation have been studied by Atomic Force Microscopy. In aggregate with optical measurements it can help to find out both mechanism of the whiskers formation, and the influence of an impurity. The dependences of growth rate of KDP filamentary crystals on relative supersaturation of a solution and on the impurity concentration for different trivalent metal ions were compared.

Short time submergence of the {101} KDP single crystal substrates in the solution with impurity added and following scanning in air already allowed to trace the dynamics of the growing surface. Being adsorbed on a surface, the impurity interferes with the step motion, that results in non-uniform face growth. The separated bulges at later stage are evidence of this process. The similar relief was constructed by a method of statistical trials for a model Kossel crystal face. Pyramidal asymmetrical growth hills and separated bulges are probably bases of the incipient filamentary crystals.

A model of formation and growth of the KDP filamentary crystals and of the mechanism of the effect of the impurity on the growth process are proposed.

Keywords: crystal growth, impurity additives, AFM

P.16.03.6

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Crystal Growth and Characterization of Non-linear Optical L-tyrosine Chloride

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L-tyrosine hydrochloride (L-THC) a semiorganic non-linear optical material has been synthesized at ambient temperature and characterized by chemical analysis, melting point measurement and FTIR studies. The solubility of L-THC was determined in different solvents at different temperatures. Bulk single crystals of L-THC were grown by slow evaporation method. Powder X-ray diffraction pattern of the grown L-THC has been recorded . Thermal properties of L-THC were studied by recording TGA/DTA and DSC curves. The Kurtz powder second harmonic generation test shows that the crystal is a potential candidate for frequency conversion in the optical region of electromagnetic spectrum. The L-THC crystal has a wide transparency window in the UV - vis-IR region.

Keywords: crystal growth, L-tyrosine chloride, second harmonic generation

P.16.03.7

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Growth of $Cs_2Ni(SO_4)_2{\cdot}6H_2O$ and $Rb_2Ni(SO_4)_2{\cdot}6H_2O$ and their Characteristics

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At the present time ammonium nickel sulfate hexahydrate (ANSH) and potassium nickel sulfate hexahydrate (KNSH) crystals are successfully used as ultraviolet light filters. However their starting dehydration temperatures are relatively low: 96°C and 97°C for ANSH and KNSH. The purpose of our work is to grow $Cs_2Ni(SO_4)_2{\cdot}6H_2O$ (CNSH) and $Rb_2Ni(SO_4)_2{\cdot}6H_2O$ (RNSH) crystals for further investigations of their atomic structure, optical transmission spectra and thermal stability. These crystals belong to the Tutton's salts as well as crystals mentioned above.

CNSH and RNSH crystals belong to the monoclinic space group $P2_1/C$. Transparent green CNSH and RNSH single crystals with dimensions of 50×50×25 mm of good optical quality have been grown from water solutions. First the crystal structure of CNSH was determined by X-ray diffraction method; the lattice parameters are: a=6.3576(8) Å, b=12.7660(17) Å, c=9.2550(10) Å, β=106.97(01)°, V=718.4 $Å^3$, Z=2, D_c=2.887g·cm^{-3}.

We carried out the comparative analysis of the optical transmission spectra of the CNSH and RNSH crystals. On the whole, their optical characteristics are similar to those of α-NSH, ANSH and KNSH. They have similar transmission bands in visible and UV – ranges of spectrum. Thermo-gravimetric analysis showed that the