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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<sub>2</sub>PO<sub>4</sub> (KDP), NaCl, Sr(NO<sub>3</sub>)<sub>2</sub>, KNO<sub>2</sub>, Ca(OH)<sub>2</sub>) 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 70°C. 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

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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 presented 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 Ltyrosine 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:  $96^{\circ}$ C and  $97^{\circ}$ 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<sub>1</sub>/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,  $\beta$ =106.97(01)°, V=718.4 Å<sup>3</sup>, Z=2, D<sub>c</sub>=2.887g·cm<sup>-3</sup>.

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  $\alpha$ -NSH, ANSH and KNSH. They have similar transmission bands in visible and UV – ranges of spectrum. Thermo-gravimetric analysis showed that the