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Polymorphism, the ability of a molecule to crystallize in more than one packing arrangement, is a popular chemical and crystallographic phenomenon [1]. Polymorphs can be generally recognized by virtue of their different unit cell parameters and often from the different crystal symmetries. The occurrence of polymorphs implies that the free energies of the various crystalline forms are comparable. The crystallization process to lead a polymorph is generally sensitive to variation in the conditions such as temperature, type of solvent, pressure and the manner in which the crystals are obtained. Polymorphism results when different (but energetically similar) packing interactions are operative during crystal growth. Here, we present two X-ray crystal structures of  $\text{HgBr}_2 \cdot \text{dppf}$  adduct, briefly, which exhibit conformational polymorphism. The dimorph (1) and (2) are supramolecular adducts bearing ferrocene moiety and two bromide ligand. Crystallization of the complex in THF led to orange crystals whereas recrystallization of the compound in DMSO resulted a mixture of orange (1) and red crystals (2). Polymorphs may be identified from differences in the relative orientation of phenyl rings of diphenylphosphine groups of flexible dppf ligand. This unique phenomenon is not known for the other coordination complexes of mercury(II) halides (chloride and iodide) with dppf as ligand [2-3]. Single crystal X-ray diffraction study of the polymorph (1) shows a monoclinic ( $C2/c$ ) system. Red crystals of the polymorph (2) crystallizes in triclinic ( $P\bar{1}$ ) system.

The independent molecules observed in the dimorphs differ in their angle between the P-C-Fe mean planes of coordinated dppf ligand. The conformational differences between the two polymorphic modifications are depicted in Figure 1.

The conformation of each dppf ligand with respect to the central iron may be defined according to the PC-Fe-PC angle. Conformational changes from one structure to the other lead to formation of two different polymorphs. In order to establish whether conformational changes were reason for the polymorphism.

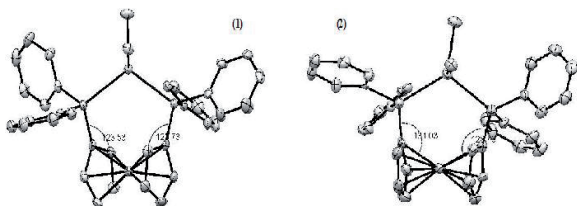


Figure 1. The P-C-Fe bond angle in dimorph (1) (left) and (2) (right).

[1] A. Nangia, *Acc. Chem. Res.* **2008**, *41*, 595-604. [2] Y.Y. Niu, Y.L. Song, S.X. Liu, X.Q. Xin, *Z. Anorg. Allg. Chemie.*, **2002**, *628*, 179-182. [3] J. McGinley, V. McKee, C.J. McKenzie, *Acta Cryst.* **1998**, *C54*, 345-347.

**Keywords:** conformational polymorphism, solvent mediation

### MS35.P32

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**Deposit of ZnO films by SS-CVD to atmospheric pressure,**

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ZnO films were deposited by single source chemical vapour deposition (SS-CVD) technique. The zinc acetate dihydrate was used as a precursor of Zn and oxygen as oxidant agent. ZnO thin films

were grown on glass substrates at different deposition temperatures (250–400 °C in steps of 50 °C) at atmospheric pressure. The structural properties were investigated by X-ray diffraction (XRD) and atomic force microscopy (AFM). Low temperature deposition is provide by SS-CVD system, this characteristic generate high deposition rate which facilitate the ZnO films formation. X-ray diffraction indicates that all deposited films were polycrystalline. The films deposited shown a preferred orientation depending of the substrate temperature. This feature determines the shape of the structure of the ZnO films. The lattice parameter strain decrease in the ZnO films by the increase of the deposit temperatures. The texture coefficient for (002) plane is 2.25. Our samples showed a transmittance in the range of 75 % to 90% in the visible region.

**Keywords:** CVD, zinc oxide, X-ray

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**Structural characterization of a new series of (Gd,R)-cuprate. R=Rare Earth**

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Great advances have been made in achieving superconductivity at high temperatures. Superconductivity up to 120 K has been found in  $\text{Ti}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ , with tetragonal structure with  $a=3.85 \text{ \AA}$ ,  $c=35.9 \text{ \AA}$  and S.G.  $I4/mmm$  [1, 2]. On the other hand, rare earth systems have had a considerable attention because they could give interesting electric and magnetic properties [3].

In this work, we present preliminary results in making ionic substitutions on  $\text{R}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}:\text{Li}$  (R = Gd, Dy, Ho) mixing the appropriate stoichiometric amounts of reactants for perform a conventional solid state reaction. The synthesis was nearly followed by X-ray diffraction, in order to reach the best conditions for synthesis. To observe the thermal stability, a combination of Thermo-Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) were carried out. The characterization was completed by the study of morphology by Scanning Electron Microscopy (SEM) and chemical analysis by Electron Dispersive X-Ray Spectroscopy (EDX).

[1] C.C. Torardi, M.A. Subramanian, J.C. Calabrese, J. Gopalakrishnan et al, *Science*. **1988**, *240*, 631-634. [2] C.C. Torardi, M.A. Subramanian, J.C. Calabrese, J. Gopalakrishnan et al, *Phys.RevB:Cond.Matt.* **1988**, *38*, 6624-6630. [3] C bryn-Jacobsen, D.F Mc Morrow, R.C.C Ward, M.R Wells *J. Phys: Condens. Matter* **1997**, *9*, 8727-8735.

**Keywords:** x-rd, synthesis, rare-earth

### MS35.P34

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**Synthesis and structural characterization on  $\text{RESrMnO}_3$  (RE=Rare Earth) system**

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Manganites with perovskite structure are the class of compounds referred as the rare earth manganites that is characterized by extremely interesting structural and physical properties and phenomena, including Colossal Magnetoresistance (CMR) [1].

The structure of the  $RE_{1-x}M_xMnO$  (RE= rare earth) oxides is close to that of the cubic type perovskite ( $CaTiO_3$ ), but the structure of manganites generally corresponds to a lower rhombohedral symmetry ( $LiNbO_3$ ) or orthorhombic ( $GdFeO_3$ ) structure [2] such as  $LaSrMnO_3$ , S.G. Pnma.

We present preliminary studies and results of the  $RESrMnO_3$  system where substitutions  $RE=Dy, Yb$  for  $La$  in perovskite type structure were carried out. Samples were prepared by the solid-state reaction method in air at ambient pressure. Process of synthesis was followed by thermal analysis (TGA and DTA) and X-ray powder diffraction (XRD). Morphology of resultant samples have been observed by Scanning Electron Microscopy (SEM) and the stoichiometry has been analyzed by Electron Dispersive X-Ray Spectroscopy (EDX).

[1] T.V. Ramakrishnan, H.R. Krishnamurthy, S.R. Hassan, G. Venkateswara. *Proc. Indian Acad. Sci. (Chem. Sci.)*, **2003**, *115*, 767–774. [2] A.M. Haghiri-Gosnet, J.P. Renard, *J. Phys. D: Appl. Phys.* **2003**, *36*, R127.

**Keywords:** perovskite, manganite, rare-earth.

### MS35.P35

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#### Crystallization in lipidic cubic phase. The impact of additives on phase behaviour

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To solve the structure of a membrane protein to atomic resolution is still a challenging task. Apart from conventional crystallization techniques the use of lipidic cubic phase (LCP) is of growing interest and was successfully applied to crystallize a number of G protein coupled receptors [1-4]. The LCP three-dimensional networks consist of a continuous bilayer defined by two distinct water channels, referred to as bicontinuous networks. Most commonly monoolein (MO) is used, forming cubic phases at temperatures suitable for crystallization.

A versatile tool to study this phase behaviour is x-ray diffraction analyzing the data in the small angle regime. Fitting scattering models to the experimental data allows to identify structural changes in the aqueous and the lipid bilayer subcompartments.

To make use of the cubic phase and to facilitate the crystallization of membrane proteins, detergents, precipitants and other additives need to be added. These chemicals tend to destabilize the network structure. Therefore it is crucial to understand their impact on the phase behaviour.

These effects are studied by addition of detergents and precipitants, known to facilitate the crystallization of membrane proteins, according to a ranking from the Membrane Protein Data Bank. In preceding experiments, depending on the type of detergent or additive and the concentration clear differences in the phase boundaries could be detected.

[1] S.G.F. Rasmussen, H.-J. Choi, J.J. Fung, E. Pardon, P. Casarosa, P.S. Chae, B.T. DeVree, D.M. Rosenbaum, F.S. Thian, T.S. Kobilka, A. Schnapp, I. Konetzki, R.K. Sunahara, S.H. Gellman, A. Pautsch, J. Steyaert, W.I. Weis, B. K. Kobilka *Nature* **2011**, *469*, 175-180. [2] D.M. Rosenbaum, C. Zhang, J.A. Lyons, R. Holl, D. Aragao, D.H. Arlow, S.G.F. Rasmussen, H.-J. Choi, B.T. DeVree, R.K. Sunahara, P.S. Chae, S.H. Gellman, R.O. Dror, D.E. Shaw, W.I. Weis, M. Caffrey, P. Gmeiner, B.K. Kobilka *Nature* **2011**, *469*, 236-240. [3] B. Wu, E.Y.T. Chien, C.D. Mol, G. Fenalti, W. Liu, V. Katritch, R. Abagyan, A.

Brooun, P. Wells, F.C. Bi, D.J. Hamel, P. Kuhn, T.M. Handel, V. Cherezov, R.C. Stevens *Science* **2010**, *330*, 1066-1071. [4] E.Y.T. Chien, W. Liu, Q. Zhao, V. Katritch, G.W. Han, M.A. Hanson, L. Shi, A. Hauck Newman, J.A. Javitch, V. Cherezov, R.C. Stevens *Science* **2010**, *330*, 1091-1095.

**Keywords:** X-ray\_diffraction, membrane\_protein, biocrystallography

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#### Crystal growth and characterization of 4-(dimethylamino) benzaldehyde doped TGS Crystals

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Single crystals of 4-(dimethylamino) benzaldehyde doped ferroelectric Tri Glycine Sulphate (DBTGS) were grown by slow evaporation from its aqueous solution at ambient temperature, using solution growth method. Solution grown DBTGS crystals were characterized by dielectric and pyroelectric measurements. The capacitance of DBTGS was measured using HP 4194A impedance/gain phase analyzer at 10 kHz at a cooling rate of 1°C/min using Mettler hot stage FP90 and the pyroelectric current was measured using Keithley 610C electrometer over the temperature range of 30 to 60°C in the ferroelectric direction. The direct method of Byer and Roundy was used for the measurement of pyroelectric current. The sample preparation and the measurement technique used in the measurement of capacitance are discussed in detail. Pure TGS crystals exhibits ferroelectric phase transition at 48.5°C with  $\epsilon'_{peak}$  8800. However for doped DBTGS crystals phase transitions is observed at 51°C with decreased  $\epsilon'_{peak}$  (1930). Higher Pyroelectric coefficient was observed for the doped DBTGS crystal. The doped crystal was irradiated with graded dosages from 5 kGy to 80 kGy electron beam from 8MeV Microtron (Energy-8MeV, pulse current-50mA (max), pulse duration-2.5µsec, pulse repetition rate-250 Hz (max)) at room temperature and radiation effects on the dielectric and pyroelectric properties of the crystals were investigated. The dielectric study shows that there is a gradual reduction in dielectric constant at  $T_c$  and shifting of Curie temperature towards lower temperature region with increase in electron radiation dose. The material figure of merit (Fv) was found to be higher for the irradiated crystals.

**Keywords:** crystal growth, TGS crystals, ferroelectric

### MS35.P37

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#### Generation of high quality crystal surfaces of small soft organic crystals

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In a time resolved micro-crystallography experiment the X-ray beam is focused to a size of a few microns. This allows probing a small crystal volume very close beneath the surface, where high populations of the excited state can be generated (visible pump light has penetration depths of a few microns only at the absorption maximum) [1]. Such experiments require well defined and low-roughness crystal surfaces. Traditional crystal polishing methods can not be applied for many