

movement of a molecule can induce polymorphic transition into a new metastable form.

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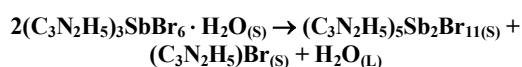
[1] Blagden N., Davey R.J., Lieberman H.F. Williams L., Payne R., Roberts R., Rowe R., Docherty R.J., *J. Chem. Soc. Faraday Trans.*, 1998, 94, 1035. [2] Drebuschak V.A., Drebuschak T.N., Chukanov N.V., Boldyreva E.V. *J. Therm. Anal. Cal.*, 2008, 93, 343. [3] Drebuschak T.N., Chukanov N.V., Boldyreva E.V., *Acta Cryst. C*, 2008, 64, o623. [4] Drebuschak T.N., Chesalov Yu.A., Boldyreva E.V., *Acta Cryst. B*, 2009, 65, 770.

Keywords: phase transitions in solids, drug polymorphism, low-temperature structure

FA4-MS35-P10

Unprecedented solid-state chemical reaction – from $(C_3N_2H_5)_3SbBr_6 \cdot H_2O$ to $(C_3N_2H_5)_5Sb_2Br_{11}$; polymorphism of substrate and products. A. Gagor^[a], A. Piecha^[b], R. Jakubas^[b] and A. Pietraszko^[a]. ^a*Institute of Low Temperature and Structure Research PAS, Wrocław, Poland*, ^b*University of Wrocław Joliot-Curie 14 50-383 Wrocław, Poland*
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A novel organic-inorganic hybrid material, tris(imidazolium) hexabromoantimonate(III) hydrate, $(C_3N_2H_5)_3SbBr_6 \cdot H_2O$ (abbreviated as TIBA) undergoes an unprecedented *in situ* solid-state chemical reaction above 328 K:



that leads to multiphase crystallites dominated by ferroelectric $(C_3N_2H_5)_5Sb_2Br_{11}$.

The room temperature structure of TIBA consists of discrete $SbBr_6^-$ anions, disordered imidazolium cations, and water molecules forming a 3D array of hydrogen bonds. The solid-state chemical reaction leads from $[SbBr_6]^{3-}$ to $[Sb_2Br_{11}]^{5-}$ chemical stoichiometry of anions. The creation of ferroelectric^[1] crystallites – $(C_3N_2H_5)_5Sb_2Br_{11}$ – in an “annealed” sample of $(C_3N_2H_5)_3SbBr_6 \cdot H_2O$ is confirmed by X-ray diffraction phase analysis, dielectric spectroscopy, and pyroelectric measurements. The dielectric response of the electric permittivity and the critical slowing down of the process observed near 140 K in the “annealed” sample of TIBA are treated as a “fingerprint” of a neat $(C_3N_2H_5)_5Sb_2Br_{11}$ ferroelectric. Furthermore, TIBA undergoes isostructural discontinuous phase transition at 212/221 K (cooling-heating) ($P2_1/c \leftrightarrow P2_1/c$). The phase transition mechanism is characterized by two contributions: an order-disorder (cationic substructure) and a displacive one (water molecules).

Crystal structure of two polymorphs of second product of reaction: $(C_3N_2H_5)Br$ that crystallizes from water solution below 273 K are also presented along with the order-disorder phase transition mechanism.

[1] A. Piecha, A. Pietraszko, G. Bator, R. Jakubas *J. Sol. State Chem.* 2008, 181, 1155-1166

Keywords: ferroelectric, halogenoantimonates(III), phase transitions

FA4-MS35-P11

Topotactic phase transformation of $4H-Cu_yTa_{1-x}S_2$ into $4H-Ta_{1-x}S_2$. Bernd Harbrecht^a, Wenjie Yan^a, Otfried Lemp^a, Steffen Zörb^a, Kirsten Bohnen^b

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Tantalum forms a series of polytypic ternary sulfides of general composition $Cu_yTa_{1-x}S_2$.^[1,2] The layered-type phases are good electronic and ionic conductors. The mobile Cu ions (y) and excess Ta (x) are inserted in spatially separated van der Waals gaps of a layered TaS_2 host structure the type of which depends upon x, y and T. Oxidative deintercalation of Cu with iodine affords new polymorphs of $Ta_{1-x}S_2$. The new polymorphs differ from those accessible from the elements at temperatures above 1250 K by higher order staging, i.e. excess Ta (x) is not found in all van der Waals gaps what confers lubricant-like properties to these Ta-rich disulfides. Such polytypes can be classified by a code nN in which n counts the number of TaS_2 slabs in the repeat unit and N refers to the lattice symmetry, e.g., H for hexagonal. Here, we present the structure of a ternary 4H polytype that - upon progressive deintercalation of Cu at ambient temperature - topotactically transforms into a differing 4H polytype. The transition is reflected in divergent X-ray diffractograms showing an offset of the reflections and a drastic change in the intensity modulation of h0l zonal reflections. Structural features of the two polymorphs derived from the distinctive intensity modulations are highlighted and discussed in the light of known polymorphs. Electronic and mechanistic aspects of the transformation are discussed.

[1] Harbrecht B., Kreiner G., *Z. anorg. Allg. Chem.*, 1989, 572, 47-54.

[2] Kirsten Bohnen, Dissertation, Universität Bonn 1995.

Keywords: transition metal dichalcogenides, polytypes, topotactic phase transformation

FA4-MS35-P12

Elucidation of Crystallization Processes – The Polymorphism of Nifedipine. Maria Klimakow^a, Klaus Rademann^b, Franziska Emmerling^a, ^a*BAM Federal Institute for Materials Research and Testing, Berlin, Germany*, ^b*Humboldt-University Berlin, Germany*

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The crystallization process of the polymorphic compound nifedipine (4-(2-nitrophenyl)-2,6-dimethyl-3,5-dicarbomethoxy-1,4-dihydropyridine, $C_{17}H_{18}N_2O_6$) using different solvents was investigated *in situ* by X-ray diffraction and Raman spectroscopy. In medical treatments, nifedipine acts as dihydropyridine calcium antagonist. Despite the pharmaceutical importance, the wide use of the substance, as well as knowledge of its polymorphism, the crystal structures of most of the nifedipine polymorphs still remain unknown. To investigate the formation of intermediate phases, which are kinetically favored and formed preferentially under fast evaporation conditions, an acoustic levitator was employed as sample environment for the combination of time-resolved X-