ray diffraction and Raman spectroscopy (simultaneous data acquisition within a 20 seconds period) [1]. This setup provides a contactless sample positioning and eliminates the influence of solid container walls. In addition, it enables *in situ* monitoring of solutions during evaporation of the solvent (methanol, ethanol, acetone, dichloromethane, and acetonitrile) over a concentration range of three orders of magnitude. These real time measurements allow the determination of the phase content from the onset of the first crystalline molecular assemblies to the stable system. The results indicate the existence of solvent dependent transient phases during the crystallization process. The quality of the data allowed the assignment of the lattice constants of the hitherto unknown crystal structure of the β-polymorph. In the final stage the fast evaporating solvents always led to the formation of the α-polymorph. Using the slow evaporating solvent dimethyl sulfoxide (DMSO), a novel pseudo-polymorph was observed. Its crystallization process was followed by means of light microscopy and ESEM coupled with EDS analysis. The single-crystal X-ray structure of the solvated species nifedipine · DMSO (1:1) is reported for the first time [2]. With the results of the presented investigation, a tool for studies on polymorphic transition mechanisms and the influence of solvents on these processes is provided.

[1] Klimakow, M., Leiterer, J., Kneipp, J., Rössler, E., Panne, U., Rademann, K., Emmerling, F., *Langmuir* 2010, la100540q. [2] Klimakow, M., Rademann, K., Emmerling, F., *Crystal Growth & Design* 2010, submitted

Keywords: polymorphism, nifedipine, synchrotron radiation

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**Structural crystal-to-crystal phase transitions in 1,4-diaminium bis(dichloroacetate)** <u>Maciej Kubicki</u>[a], Agnieszka Paul[a, b], Marek Szafrański[a]
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Crystals of putrescinium bis(dichloroacetate), $(C_4H_{14}N_2)^{2+} \cdot 2C_2HCl_2O_2^-$ undergo two reversible phase transitions between 100 and 295K. The first transition occurs about 140K, and the second one around 249K. The low temperature structure (α-phase) is best described in $P2_1/c$ space group. The transition to $I2/a$ space group (β-phase) involves the doubling of one of the unit-cell parameters. The second phase transition recovers the initial $P2_1/c$ symmetry (α'-phase). Only in the low-temperature α-phase the putrescinium dication is disordered across the inversion center (in other phases it is ordered), while the disorder of the anion occurs always for the temperatures close to the phase transitions. It looks like the disordering of the anion starts the sequence of changes which eventually cause the change of the phase. The hydrogen bond networks are similar, but some essential changes occur. Using graph-set method, the hydrogen bond patterns are built from the same motifs for α and α' phases, whereas for the β-phase these "building blocks" are different, for instance the typical for diaminium salts $R^2_4(8)$ ring occurs.

100K

140K
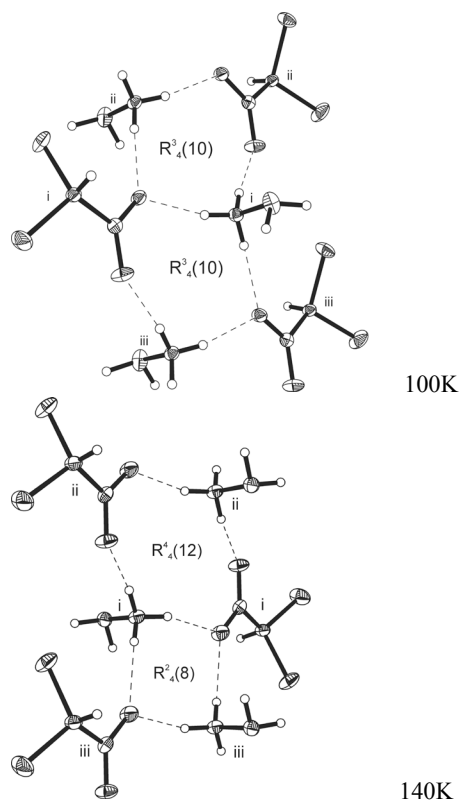
Despite these differences, the layer structure is present in all three phases. The additional calorimetric and dielectric data were collected in order to help in the description of the process of phase change. Also the Hirshfeld surfaces were used to better visualize the changes.

Keywords: phase transitions; hydrogen bonds; putrescinium

FA4-MS35-P14

Solid state transition in $[Cd(\mu\text{-Br})_2(py)_2]_\infty$. <u>Kevin Lamberts</u>[a], Ulli Englert[a], [a]*Institute of Inorganic Chemistry, RWTH Aachen University, Germany*
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Earlier research on halide-bridged Cd(II) coordination polymers with axial pyridine ligands indicated a phase transition around 180 K for $[Cd(\mu\text{-Br})_2(py)_2]_\infty$ [1]. It is remarkable that this crystal-to-crystal transition seems to be fully reversible. The room temperature structure has already been determined [2] but the low temperature phase and the transition itself still have to be analysed more precisely.

Task of the work is the concrete determination of the twinned low temperature phase of $[Cd(\mu\text{-Br})_2(py)_2]_\infty$ and the specification of the phase transition. For the crystal structure determination the group-subgroup relationship is of particular interest. In connection with the phase transition the exact transition point is going to be specified and its thermodynamic conditions will be assayed. For these purposes single crystal X-ray diffraction, powder diffraction and differential scanning calorimetry will be used.

Another point of interest is the structure similarity of the low temperature phase with chemically related complexes and the effect of mixed crystals on the phase transition.