

MS32-P8 X-ray structural analysis of sodium salt of acetylsalicylic acidMiroslava Dědová¹, Jan Čejka¹¹. Department of Solid State Chemistry, University of Chemistry and Technology Prague, Technická 5, Prague 6, Czech Republic

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Pharmacological effects of acetylsalicylic acid have been known for a long time. Recently, new therapeutic uses of aspirin were discovered, and investigated, e.g. prevention of colon cancer or treatment of dementia. This promotes a detailed screening for new physical (polymorphs) and chemical (salts, co-crystals) forms of aspirin. An improved dissolution rate of the forms is a desirable property in order to decrease stomach wall damage.

Many patents are focused on the preparation of the sodium aspirin. Surprisingly, no structure of sodium aspirin has been solved yet. The sodium salt can be prepared by the reaction of acetylsalicylic acid with sodium bicarbonate. Both known and patented forms arise under very similar conditions - sodium acetylsalicylate dihydrate and anhydrate. Their preparation is very difficult and the reproducibility is fairly low due to ester hydrolysis. Therefore a new screening of salt preparation and crystallization was performed to achieve better reproducibility.

The preparation of sodium acetylsalicylate dihydrate was successful, the structure was determined by single-crystal X-ray diffraction. The dihydrate form was later dehydrated to sodium acetylsalicylate anhydrate. (not verified yet). The dehydration was studied by thermoanalytical methods (TGA/DSC). However, the structure of anhydrate has not been solved yet.

Further, a new form of sodium aspirin was discovered - sodium acetylsalicylate monohydrate. Its structure was determined by single-crystal X-ray diffraction. Unlucky, no attempts to reproduce the monohydrate form were successful.

This work was supported by the Grant Agency of Czech Republic, Grant no. 106/14/03636S.

Keywords: x-ray crystallography, salt formation, pharmaceuticals

MS32-P9 Temperature-induced single-crystal-to-single-crystal polymorph transformation in cadmium(II) trimer with pyridine-4-propanamideBoris-Marko Kukovec¹, Ivan Kodrin², Marijana Đaković¹¹. Division of General and Inorganic Chemistry, Department of Chemistry, Faculty of Science, University of Zagreb, Horvatovac 102a, HR-10000 Zagreb, Croatia². Division of Organic Chemistry, Department of Chemistry, Faculty of Science, University of Zagreb, Horvatovac 102a, HR-10000 Zagreb, Croatia

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Single-crystal-to-single-crystal (SCSC) transformations have been established as an interesting solid state phenomenon and have gained some interest, as the transformed structure can be also determined, providing insight into the possible mechanism of transformation [1]. SCSC transformations may be induced by external stimuli like heat, light or sorption/desorption of guest/solvent molecules and usually occur in metal-organic frameworks (MOFs). The studies of SCSC polymorph transformations of metal-organic compounds are scarce, but they start to gain attention [2]. The molecular structure of cadmium(II) coordination trimer with pyridine-4-propanamide (4-Propy), $[\text{Cd}_3(\text{4-Propy})_6](\text{CH}_3\text{CH}_2\text{COO})_3$ (**1**), consists of three cadmium(II) ions bridged with chloride ions; each is coordinated by two *N*-monodentate 4-propy ligands. Two water molecules are bound to the terminal cadmium(II) ions of the trimer, preventing further polymerization of the trimer units. High-temperature polymorph (**A**) of **1** crystallized in triclinic system (*P*-1 space group) at room temperature. SCSC transformation to low-temperature polymorph (**B**) of **1** occurred by cooling a single crystal to 150 K on a diffractometer (Fig. 1). Polymorph **B** crystallized in monoclinic system (*P*2₁/*n* space group). In **A**, trimer units are linked via propanoate ions by N-H...O interactions, forming a 1D chain along the [0 1 1] direction. However, in **B**, similar 1D chains are assembled into a 2D network in the plane (0 1 0) due to the additional N-H...Cl interactions, present only in **B**. Therefore, lowering temperature enables the assembly of the 1D chains of **A** into 2D network of **B** by introducing different type of interaction (N-H...Cl). It seems that the transformation of **A** into **B** is possible due to slight change of the molecules' orientation in the structure of **A**, enabling the formation of N-H...Cl interactions in the same crystal, without a collapse of the crystal structure. This SCSC polymorph transformation was also studied by PXRD and DSC. The Hirshfeld surface analysis was performed to identify the interactions present in **A** and **B**, revealing differences between the two polymorphs. **References:** [1] J. P. Zhang, P. Q. Liao, H. L. Zhou, R. B. Lin, X. M. Chen, Chem. Soc. Rev. 43 (2014) 5789.; [2] D. Aulakh, J. R. Varghese, M. Wriedt, Inorg. Chem. 54 (2015) 8679. **Acknowledgment:** This work has been fully supported by Croatian Science Foundation under the project UIP-11-2013-1809.

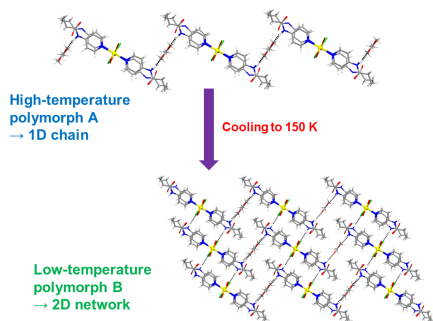


Figure 1. SCSC transformation of high-temperature polymorph A of $[\text{Cd}_3\text{Cl}(\text{4-Propyl})_3](\text{CH}_2\text{CH}_2\text{COO})_2$ (1) into low-temperature polymorph B by cooling to 150 K

Keywords: single-crystal-to-single-crystal transformation, polymorph, cadmium(II) trimer

MS32-P10 Polymorphism, what it is and how to identify it.

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Polymorphism is a very important phenomenon not only in basic research, but certainly in pharmaceutical industry and materials science. Polymorphs possess different properties, for instance the solubility or the mechanical resistance can differ dramatically from one polymorph to the other – properties which can be crucial for their application. Hence, it is important to be able to control the formation of polymorphs and to understand their formation. We here gave some insights into the basic knowledge of polymorph formation and their identification and characterization in order to give an overview on the current state of the art. In order to give interested peoples a tool in hand to test their compounds for polymorphism, we established a series of flow sheets to follow, depending on the class of compounds, hoping that they are useful for many scientists who are not so well acquainted with polymorphism. The presented schemes resume thus the identification steps for polymorphs. It should also help to use the term polymorph correctly in order to reduce the number of publications in which this term is not used in a correct way.

References: J-P. Brog, C-L. Chanez, A. Crochet, K. M. Fromm, *RSC Adv.*, **2013**, 3, 16905-16931.

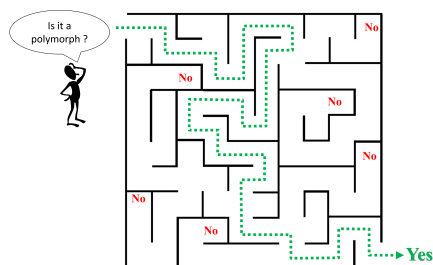


Figure 1.

Keywords: Polymorphism, organic, organometallic, inorganic.