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X-ray diffraction and microscopy study of supramolecular networks of amido functionalized compounds

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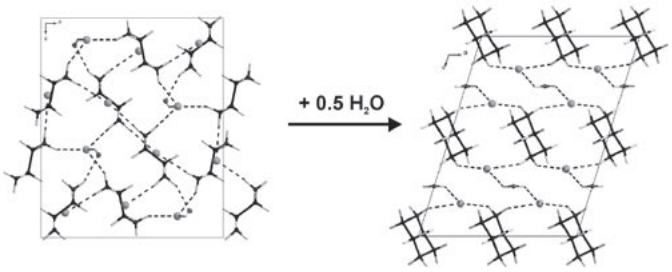
Crystalline amido functionalized organic compounds have been investigated towards the forming of solvates, halogenides and their reactivity towards various metal salts at different temperatures and under variable recrystallization conditions. Crystal structure determination, temperature-dependant X-ray powder diffraction as well as hot-stage microscopy experiments have been carried out successfully and revealed new aspects for a crystal engineering of isotypical but not isomorphous co-crystals or supramolecular compounds. Even though the crystal structure of co-crystals of melamine and EDTA (ethylenediaminetetracetic acid) contains water molecules, the two components form a solvent-less stable supramolecular network. In contrast to this, the inclusion of different solvents into the structure of 1,4-diaminocyclohexane chloride and 1,4-piperazine chloride (see Fig.) causes significant structural changes as demonstrated by hot-stage microscopy and X-ray diffraction experiments at various temperatures. We investigate the reasons for these structural changes due to solvent inclusion.



Keywords: crystal engineering, hydrogen bonding, supramolecular amides

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Systematic mutation study toward the engineering of protein crystals

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It is well-known that the protein crystallizability is influenced by a site-directed mutagenesis on the molecular surface of proteins, indicating that the intermolecular interactions at crystal packing regions might have a crucial role on the structural regularity at atomic resolution of protein crystals. Here we systematically examined the improvement of the resolution of protein crystals by introducing a single mutation to the crystal packing residue so as to provide more favourable packing interactions, using a model system of diphthine synthase from *Pyrococcus horikoshii* OT3. All the designed 21 mutants in total at 13 different crystal packing residues yielded nearly isomorphous crystals from the same crystallization condition to that for the wild-type crystals which diffracts X-rays at 2.1 Å resolution. Of the 21 mutants, eight have provided crystals with improved resolutions of 1.8 Å or better. Thus it has been clarified that the crystal quality can be improved by introducing a proper single mutation to the crystal packing residue. In the improved crystals, more intimate crystal packing interactions than those in the wild-type crystal are seen. Notably, the mutants K49R and T146R yielded crystals with outstandingly improved resolutions of 1.5 and 1.6 Å, respectively, in which a large-scale rearrangement of packing interactions is unexpectedly observed in spite of retaining the same isomorphous crystal form. On the other hand, the mutants that provided results in good agreement with the designed putative structures tend to achieve only moderate improvements in resolution up to 1.75 Å. These results suggest a difficulty in the rational prediction of highly effective mutations in the crystal engineering.

Keywords: protein crystallography, crystal engineering, crystal packing

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Crystal engineering of materials with potential NLO properties using barbituric acid as component

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The barbituric acid and urea addition compound have been considered in crystal engineering of materials with potential NLO properties. Using different crystallization conditions three polymorphs have already been obtained and their crystal structures were determined. Two polymorphs belong to monoclinic space groups $P2_1/c$ and Cc, whereas the third one is triclinic P-1. Due to complementarity of the donor and acceptor functional groups of the barbituric acid and urea molecules, the moderate hydrogen bonds were formed. The interactions dominate in the structures. The recognized tautomers of barbituric acid [1] seems to be responsible for different hydrogen-bond systems observed in the polymorphs and for different packing topology. The polymorphic forms have been easily differenciated already at the first-level graph-set analysis [2] of their hydrogen bond systems, newertheless the higher-level approach enabled to reveal significant features of the spacial mutual arragement of the structural components. Only one polymorph of polar space group *Cc* could be considered as a potential non-linear material and the appropriate measurements are in progress.

[1] V.B. Delchev, V.B. (2004). *J. Struct. Chem.* 45(4) 570-578.
[2] Bernstein J., Davis R.E., Shimoni L., Chang Ning-Leh (1995). *Angew. Chem. -Int. Edition in English* 34, 1555-1573.

Keywords: polymorphism, barbituric acid and urea addition compound, graph-set analysis

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Energy versus 3D geometry - A study of intermolecular interactions using theory and experiment

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Crystal structures are in general held together by “a combination of intermolecular pair interactions that, taken separately, are less