characterized. Single crystal structure determination revealed only slight conformational differences of the hexamidine molecule, which causes a doubling of the primitive unit cell volume of the low temperature form. However, the overall packing of both forms is maintained. Based on the thermo-chemical data a semi-schematic energy-temperature diagram [2] was constructed, which permits the visualization of the relationships and relative thermodynamic stability of the polymorphic forms in this complex system. This knowledge allows us to derive valuable information that is relevant for the design and control of the production and formulation strategy of a particular polymorph.

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Keywords: polymorphs, pharmaceuticals, thermal analysis

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Order and disorder in the $Sr_2VO(XO_4)_2$ (X=V,P) phases

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The $M_2VO(XO_4)_2$ compounds (M = Sr, Ba, Pb; X = V^{+5}, P) ("239 phases") have been actively studied during the last decade as low-dimensional magnetic systems. Among these oxides the Sr239 phases are especially interesting since they have similar crystal structures for X = V and P. The crystal structures of these phases contain infinite chains of the corner shared $V^{+4}O_6$ octahedra linked into layers by XO_4 tetrahedra. The vanadium cation is slightly shifted by about 0.2 Å towards one of the O-ion. This results in a formation of one short V-O vanadyl bond, elongated opposite V-O bond. In the Sr239 phases the vanadyl bond, pointing toward the oxygen atom connecting the octahedra along the chain, but it is not clear in which sense it is directed since the reported refinement of the X-ray diffraction data led to a splitting of the V-position towards both directions with statistical occupancy of 50%. Order-disorder in the crystal structures of the S239 phases were investigated by means of single crystal and powder X-ray diffraction, electron diffraction (ED) and high-resolution electron microscopy (HREM). We succeeded in obtaining of $Sr_2VO(VO_4)_2$ and $Sr_2VO(PO_4)_2$ single crystals having ordered structures where the short vanadyl bonds have same direction inside the layers and are directed oppositely in the adjacent layers. Such ordering results in a decrease of the symmetry from the body-centered unit cell to primitive one. Electron microscopy study revealed a presence of both primitive and body-centered crystallites in the samples as well as a new type of superstructure. Additionally, the different types of defects were observed on the HREM images. Magnetic properties of the compounds are briefly described.

Keywords: order-disorder transitions, low-dimensional materials, vanadium compounds

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Powder X-ray diffraction study of polymorphic drugs: fluconazole and mebendazole

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Polymorphism and pseudopolymorphism in pharmaceutical compounds can affect bioavailability and therapeutic efficiency of drugs. Therefore, this characteristic that might affect the stability or availability of the drug substance in a solid dosage form must be monitored and controlled [1]. The aim of this work is to characterize the solid state crystalline forms presents in raw material and drug product tablets of the pharmaceutical active compounds fluconazole (FCZ) and mebendazole (MBZ). The samples were studied by powder X-ray diffraction (XRD) using synchrotron radiation and thermal analysis (TGA and DSC), respectively. The raw material of FCZ employed to manufacture drug product usually is identified as a mixture of a monohydrate and anhydrate form I. In the case of MBZ, for which up to now is known three polymorphic forms (A, B e C), is well established that the form C is the only one therapeutically efficient. The XRD, TGA, and DSC results obtained for FCZ showed significant polymorphic differences comparing the five samples manufactured by different makers. Of the eleven raw materials of MBZ studied here, four were identified as form A, which is considered therapeutically inactive. The more alarming result is that the drug tablets analyzed here do not show the presence of the active form. It is important to emphasize that the XRD data collected using synchrotron facilities was mandatory to characterize the pharmaceutics solids studied here. The high resolution was important to identify the mixture of polymorphic forms, especially to FCZ and MBZ for which as of yet the crystal structures of some polymorphic forms was not determined.

Aknowlegments: FAPEMIG and FUNED.

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Keywords: pharmaceutical crystallography, polymorphism, powder crystallography

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Low temperature and ambient phases of decane-1,10-diammonium dichloride monohydrate

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Investigations into structure-property relationships and applications of *n*-alkyl-ammonium and *n*-alkyl-diammonium salts are of continued interest and form the basis of our continuing investigations of these materials. In particular, we have focused on the structural characteristics of the *n*-alkyl-diammonium salts as they are precursors to layered inorganic-organic perovskite-type hybrids(1); they are bidentate ligands in transition metal complexes that have applications in propellants, explosives and pyrotechnic compositions(2); they have structure directing properties in the synthesis of a number of

zeolites(3); and many have biological applications(4). In this work we report an interesting and unusual non-ambient solid state phase transition of the title compound and postulate a mechanism for how the phase change occurs. We also describe and compare the packing arrangements of both phases as well as the complex intermolecular interactions that occur within the compound itself. The room temperature phase, Form I, is disordered and occupies the space group $P2/c$. The low temperature phase, Form II, is perfectly ordered and occupies the space group $P2_1/c$. Differential scanning calorimetry confirms the evidence of both phases of the title compound.

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Keywords: low-temperature crystallography, differential scanning calorimetry, n-alkyl-diammonium salts

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Synthon polymorphism in dihydroxybenzoic acids

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Polymorphism, the existence of more than one crystalline modifications of the same compound, has great importance in pharmaceuticals, agrochemicals, pigments, dyes and explosives. Polymorphs have different physical and chemical properties, such as melting point, density, compressibility, solubility, hardness, dipole moment and bioavailability. Multifunctional molecules are capable of making different supramolecular synthons in different crystal structures, or synthon polymorphs. Sublimation and melt crystallization were shown to give guest free forms through a green methodology.[1] These techniques are now employed to generate new polymorphs of 2,3-, 2,4-, 2,5-, 2,6-, 3,4-, and 3,5-positional isomers of dihydroxybenzoic acids. These compounds are prone to give solvate/hydrate forms upon crystallization. Two polymorphs of 3,5-dihydroxybenzoic acid, a new polymorph of 2,3-dihydroxybenzoic acid and guest free form of 3,4-dihydroxybenzoic acid were crystallized by melting and sublimation. A new hydrate polymorph of 3,4-dihydroxybenzoic acid was isolated. These polymorphs differ in the nature of hydrogen bond synthons in their crystal structure. Structural and thermal characterization of polymorphic phases having multiple Z' will be presented.

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Keywords: polymorphism, inclusion compounds polymorphism, organic crystal structures

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Conformational polymorphs of temozolomide and furosemide

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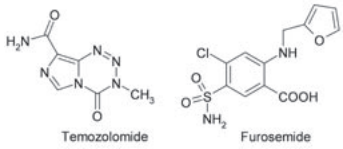
The pharmaceutical industry is frequently confronted with the phenomenon of multiple crystal forms of the same chemical entity. Two Active Pharmaceutical Ingredients (API), Temozolomide (TMZ) and Furosemide (FUR) are chosen to study the influence of conformer changes on hydrogen bonding and crystal packing. Apart from a known crystal structure of TMZ form 1, two new crystalline modifications, form 2 and 3, were obtained during attempted co-crystallization with carbamazepine and 3-hydroxypyridine-N-oxide. Conformers A and B of TMZ are stabilized by intramolecular hydrogen bonds with imidazole and tetrazine N atoms. The stable conformer A is present in TMZ polymorphs 1 and 2 whereas both conformers crystallized in TMZ form 3. Similarly three polymorphs of FUR are characterized. High energy conformers (4.49 kcal/mol) are present in stable FUR form 1 while a stable conformer is present in the metastable FUR 2 and 3 crystal structures. Polymorphism in both these systems is based on differences in conformations and hydrogen bond synthons.



Keywords: drug polymorphism, conformational polymorphism, X-ray characterization of single crystals

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Crystal structures and pseudo polymorphism of anionic surfactants

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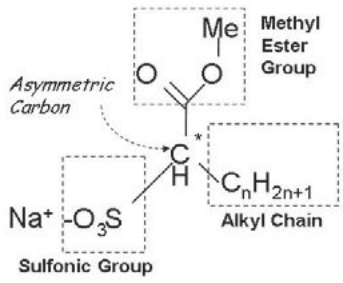
Crystal structures of anionic surfactants, methyl 2-sulfoalkanoate sodium salts (MSA) in Fig. have been analyzed by X-ray. These types of surfactants are called as methyl ester sulfonate (MES) notably used for commodity detergent as the view point of eco-friendry materials derived from vegetable oil and less emission of carbon dioxide. The determined crystal structures of surfactants are very rare because of the difficulty of the formation of single crystals. The crystals of MSAs show pseudo polymorphism with several hydrated numbers. A hydrated type of crystals transformed to other type of those by the humidity and the hydrated number affects the physicochemical properties of MSAs. Crystals of MSA showed 4 types of hydrate number of anhydrate, two, six, and ten in previous report, 1). The crystal structure of dihydrate (*Pbcm*) has been determined by X-ray in 2). New crystal structures with several hydrate salts of MSAs of racemic crystals have been determined. The single crystals were obtained from aqueous ethanol.



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Keywords: surfactants, detergents, hydrates