

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 $\text{Sr}_2\text{VO}(\text{XO}_4)_2$ ( $\text{X}=\text{V},\text{P}$ ) phases

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The  $\text{M}_2\text{VO}(\text{XO}_4)_2$  compounds ( $\text{M} = \text{Sr}, \text{Ba}, \text{Pb}$ ;  $\text{X} = \text{V}^{5+}, \text{P}$ ) ("239 phases") have been actively studied during the last decade as low-dimensional magnetic systems. Among these oxides the  $\text{Sr}_2\text{39}$  phases are especially interesting since they have similar crystal structures for  $\text{X} = \text{V}$  and  $\text{P}$ . The crystal structures of these phases contain infinite chains of the corner shared  $\text{V}^{4+}\text{O}_6$  octahedra linked into layers by  $\text{XO}_4$  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  $\text{Sr}_2\text{39}$  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  $\text{S}239$  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  $\text{Sr}_2\text{VO}(\text{VO}_4)_2$  and  $\text{Sr}_2\text{VO}(\text{PO}_4)_2$  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 pharmaceuticals 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.

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References

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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