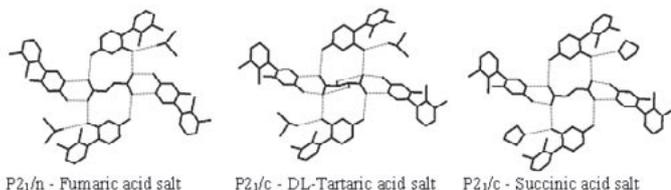


and  $P2_1/n$  space groups, being isomorphous dicarboxylic acid salts of lamotrigine. The corresponding powder samples have been characterized by thermal methods (DSC-TGA), PXRD, FT-IR spectroscopy and their water solubility have been determined. In all cases water solubility of the presented lamotrigine salts are higher respect to lamotrigine free base. The water solubility value appears directly related with the solubility of the acid involved in the salt, thus the higher is the water solubility of the free acid, the higher is the solubility of the salt formed. The isomorphous nature of the salts obtained allows relating their solid state properties with those of the counterion involved in each salt. The importance of the counterion solubility on the final solubility of the salts is rationalized considering their crystal structures.



Keywords: isomorphism, pharmaceutical, structure-property relationships

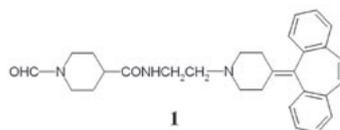
### P08.10.107

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#### Polymorphs and humidity-induced transition of a serotonin receptor antagonist, $C_{29}H_{33}N_3O_2 \cdot HCl \cdot xH_2O$

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The serotonin ( $5-HT_{2A}$ ) receptor antagonist, *N*-[2-[4-(5*H*-dibenzo[*a,d*]cyclohepten-5-ylidene)-piperidino]ethyl]-1-formyl-4-piperidinecarboxamide ( $C_{29}H_{33}N_3O_2$ ; **1**) monohydrochloride crystallizes as a monohydrate (Form  $\alpha 1$ ) from an acetone solution and as a trihydrate (Form  $\beta 3$ ) from an aqueous solution. Humidity-induced phase transitions proceed in both hydrates. Form  $\alpha 1$  changes to an anhydride (Form  $\alpha 0$ ) below 5 % relative humidity. While  $\beta 3$  transforms to an anhydride (Form  $\beta 0$ ) through a dihydrate (Form  $\beta 2$ ) and a monohydrate (Form  $\beta 1$ ) in 0-30 % relative humidity range. Both transitions are reversible, and hysteresis is observed in only the case of Form  $\beta$ . The crystal structures of Forms  $\alpha 1$ ,  $\alpha 0$ ,  $\beta 3$ , and  $\beta 2$  were determined. **1** consists of a non-polar dibenzocyclohepten ring and a polar tail. Hydrophilic and hydrophobic layers are constructed in both crystal forms, and crystal water molecules and  $Cl^-$  ions are located in hydrophilic region surrounded by polar groups. Characteristics of crystal structures, transformation of hydrogen-bonding schemes, and conformational changes of **1** coupled with the phase transitions are discussed.



Keywords: polymorphs, phase transitions, hydrates

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#### Order-disorder transitions in sodium vanadylphosphate $Na_4VO(PO_4)_2$

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The new vanadyl phosphate  $Na_4VO(PO_4)_2$  was synthesized and investigated by X-ray powder and single-crystal diffraction, high-temperature X-ray diffraction, electron diffraction and high-resolution electron microscopy. Its crystal structure contains isolated infinite chains of the corner-sharing  $VO_6$  octahedra. The octahedra within the chains are additionally linked to each other by the tetrahedral  $PO_4$  groups. Sodium atoms are situated in the positions between the chains. Depending on the conditions of synthesis, the number of sodium atoms in the unit cell of the  $Na_{4+x}VO(PO_4)_2$  compounds may vary resulting in a change of the oxidation state of vanadium atoms and a change of their coordination environment. Electron diffraction study revealed an existence of various structural transformations occurring in situ in the transmission electron microscope. The charge redistribution was supposed in the gamma-modification of  $Na_{4+x}VO(PO_4)_2$  where the  $V^{4+d}$  and  $V^{4-d}$  cations orderly occupy octahedral positions in different chains. The origin of this phenomena is discussed.

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

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#### Polymorphs of picryl bromide

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Polymorphism is a phenomenon that has been observed often during the rich history of energetic materials development. Different polymorphs of the same compound can have different properties, which, in turn, can lead to substantial alterations in their stability and performance. Two polymorphs of the common energetic precursor, 2,4,6-trinitrobenzene (picryl bromide), were discovered in 1933, however no x-ray crystal structures were reported then, or have since been reported. Structural details of the two known polymorphs, the alpha and beta forms are detailed here. In addition, three new polymorphs are also presented, the gamma, delta, and epsilon forms. Two different triad motifs, consisting of C-H...O hydrogen bonds and N-O...Br dipole induced interactions, universal to all five forms of picryl bromide will be discussed, as well as apparent nitro-pi associations between adjacent layers of molecules.

Keywords: polymorph, energetic, intermolecular interaction