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Crystallization of Sulfonamides: a phase diagram, calorimetric and NMR spectroscopy study

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Achieving tight control over the selective crystallization of specific crystal forms is still one of the holy grails of crystallization science, which has a strong impact in industries such as pharmaceuticals, that rely on the manufacture of products with highly reproducible properties and performance. The main difficulty lies in the complexity of the self-assembly process behind crystal formation, which is often strongly influenced by factors that are hard to control in practice (e.g. container walls, temperature fluctuations, presence of dust). The problem needs, therefore, to be studied from many angles. Thus, in this work several aspects of the crystallization of the antibiotic sulfanilamide (Figure 1) will be presented namely: the determination of temperature vs. concentration phase diagrams in different solvents, that can guide the selection of appropriate conditions for the formation of a given crystal phase; details of the crystallization mechanism that can be inferred from calorimetric measurements carried out with a MicroCal DSC apparatus and from NMR spectroscopy experiments.

Figure 1. Molecular structure of sulfanilamide.

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References:

 A.Y. Lee, D. Erdemir, A.S. Myerson, Annu. Rev. Chem. Biomol. Eng., 2, 2011, 259-280.
 C.E.S. Bernardes, M.L.S.M. Lopes, J.R. Ascenso, M.E. Minas da Piedade, Cryst. Growth Des. 14, 2014, 6663-6681.

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