

SC9

X-Ray Powder Diffraction as Function of Relative Humidity

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Water may have a considerable influence on the chemical and physical stability of drug substances as well as on dosage forms. Water can create enormous difficulties in the use of water sensitive drug substances and auxiliary materials.

The method of X-Ray powder Diffraction (XRD) performed as a function of relative humidity and temperature is rather sophisticated and enables to observe the formation of hydrates and their dehydration on the basis of structural changes in the crystal lattice [1, 2]. Not only physical but also chemical changes, i. e. hydrolysis, can be detected. In combination with other techniques such as thermogravimetry, water vapor sorption, differential scanning calorimetry and classical chemical analysis a global knowledge of the reaction can be established.

The kinetics of these transformations in respect to the changes of relative humidity are an essential parameter. Substances with slow kinetics would have to be equilibrated in chambers of a given humidity and can be measured without special precautions. On the other hand, for substances with quick response to changes in ambient humidity the measurements must be performed in a controlled atmosphere. This will be illustrated by an example. By variation of the humidity in the sample chamber of the X-ray diffractometer the kinetic changes in the crystal structure could be characterized as a function of the humidity change. This gave an explanation of the observed differences in the diffraction patterns of the substance and showed them to be no "artefacts". Some attempts concerning the structural feature will be given.

References

- [1] J. Han and R. Suryanarayanan, *Thermochim. Acta*, 329 (1999) 163-170
- [2] P. Di Martino, F. Piva, P. Conflant and A. M. Guyot-Hermann, *J. Thermal. Anal.*, 57 (1999)95-109