PO E 5

Coupled DSC-Microscope System and its Application to Pharmaceuticals

Kurt Pommerenke * and Mitsurzi Ohta **

* Shimadzu Deutschland GmbH, European Operations, Albert-Hahn-Str. 6-10, D-47269 Duisburg, Germany

** Shimadzu Corporation, Kyoto Customer Support Center, Testing & Weighing Equipment Division, l.Nishinokyo-kuwabaracho, Nakagyo-ku, Kyoto 604-8511, Japan

Differential Scanning Calorimetry (DSC) and Thermooptical Analysis (TOA) such as thermomicroscopy or melting-point determination by capillaries are proven thermoanalytical methods. The Shimadzu DSC-60V combines the standard DSC with a common microscope. The DSC provides features such as controlled cooling and heating, high sensitivity or resolution. Sample preparation is almost not needed. The use of a commercial stereo-zoom microscope expands this method with the option to study optical changes of the sample during the run. Especially in the study of polymorphism or of the crystallization process this combination provides the ease of interpretation of data.

Sulfathiazole, 4-amino-N-thiazol-2-ylidene-benzenesulfonamide, $C_9H_9N_30_2S_2$, is a compound with different polymorphic forms. At least five different forms have already been described in literature. The polymorphic forms used were obtained by crystallization from n-propanol and water-ethanol. The first sample showed a white powder which included some almost transparent hexagonal crystals. During the DSC run an endotherm was observed at 174 °C. The video showed the melting of the form III - the transparent crystals. At 201 °C the whole sample was melted. [1]

In a second test the stable form I was grinded and run under the same conditions like the previous sample. A broad endotherm was observed at 157 °C. Though the form II of sulfathiazole is reported to melt at this temperature, the video showed no melting. One melting point could only be observed at 201 °C of this sample. The peak at 157 °C is related to lattice effects formed during the grinding. The grinding process may lower the activation energy of the form I→III transition. The calculated enthalpy values confirm this theory. [2]

[1] M. Lagas & C.F. Lerk, The Polymorphism of Sulphathiazole, Int. J. Pharmaceutics, 8, 11-24, (1981)

[2] K. Sekiguchi, K. Shirotani, H. Yuasa, E. Suzuki & F. Nakagawa, Size Reducibility of Sulfathiazole by Heat Transition and Subsequent Ball-milling, Chem. Pharm. Bull. 28 (11), 3203-3209, (1980)

* *contact address: kup@shimadzu.de*