

Use of high throughput microcalorimeter for faster determination of amorphous content

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Isothermal microcalorimetry is a very sensitive technique with a great number of applications. The technique is widely used for the determination of amorphous content of food and pharmaceuticals. The analyte subjected to a solvent vapour atmosphere in the instrument crystallises and the heat flow generated continuously is measured while the analyte is kept at constant temperature. Our laboratory described several applications in pharmaceutical GMP environment using the classic isothermal heat conduction microcalorimeter of Thermometric, model 2277 TAM [1-2].

The bottleneck for application of isothermal microcalorimetry as a routine method is the time needed for each experiment, including equilibration time before analysis. The new generation multichannel microcalorimeter TAM III in 48 channel version increases considerably the sample throughput and consequently reduces the time needed for the development of appropriate parameters such as the selection of solvent, the amount of analyte, the range of amorphous content and for the validation of the analysis method. The instrument can then be used to get fast results, needed in process development and for routine analysis.

The advantage of the scanning mode of the TAM III is used for the efficient calibration of the instrument by the specific heat of water or the melting point of appropriate standards , as e.g. NIST 2222 (biphenyl).

Examples will show the advantage of this highthroughput instrument compared to the model 2277 TAM for method development.

Selected examples will give a comparison of results delivered by TAM 2277 for validated methods and by TAM III.

[1] S. Garnier, STK Lausanne, 2001

[2] D. Giron, M. Mutz and S. Garnier, J. Therm. Anal. Cal.,77 (2004) 709.