Isothermal Microcalorimetry - A Universal Tool for Research and Quality Control. Application Examples with Special Reference to the Pharmaceutical and Food Industries

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Isothermal microcalorimetry is a technique by which the heat flow generated by an arbitrary chemical, physical or biological process is continuously monitored while the sample is kept at constant temperature. The heat flow is directly related to the rate of heat produced or consumed by an arbitrary sample placed in the calorimeter. In most cases, the sample is contained in a removable insertion vessel positioned in an ampoule holder inside the microcalorimeter during measurement.

TAM (Thermal Activity Monitor) is a 4-channel calorimetric system originally developed at University of Lund [1] and commercialised by LKB Instruments in Sweden. Since 1983 TAM is manufactured by Thermometric AB who has developed the technology further and recently TAM III – a third generation microcalorimeters was introduced. This new instrument is designed for multi-sample experiments and allows up to 48 measurements to be performed simultaneously. In addition to strictly isothermal measurements it is possible to operate this instrument in step isothermal and slow scanning mode (scanning rate < 2 K h^{-1}).

The calorimetric systems from Thermometric consists of a hierarchically structured range of products or modules. The most basic module is a high precision thermostat, followed by various types of calorimetric units and a range of insertion ampoules specifically designed for a number of applications.

Calorimetric sample vessel can be classified as closed and open ampoules. A closed ampoule is totally sealed from the environment and is generally employed when studying slow processes such as in compatibility or stability experiments. A special case of a closed ampoule is having a solid sample and a saturated salt solution physically separated but connected through the vapour phase inside the ampoule. After a sorption process a physical or chemical reaction of the sample takes place, *e.g.* a slow decomposition reaction, solvate formation or recrystallisation of amorphous phases. Open ampoules are used when a process is to be initiated *in situ* after a base line has been recorded. An example is alteration of the chemical composition of a solution by injecting a catalyst, reactant or a ligand in order to initiate a physical, chemical or biological response, which is then continuously measured. Another example is changing the vapour activity of an inert flowing carrier gas over a solid sample so as to initiate sorption or desorption with a subsequent physical or chemical change of the sample. Examples of such a change include recrystallisation of amorphous phases, solvate formation, acceleration of a humidity dependent decomposition reaction and more.

In the presentation, a few application examples commonly encountered in the pharmaceutical and food sciences will be presented and discussed.

Reference

Jaak Suurkuusk and Ingemar Wadsö. Chemica Scripta. Vol. 20, 155-163 (1982)