A New Small-Scale Reaction Calorimeter with Combined Temperature Control for the Determination of Kinetics Parameters

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During the early stages of process engineering chemical industry needs flexible and versatile tools to determine detailed information about chemical reaction systems. An important part of the optimisation of a process considering economic factors, risk analysis and environmental impacts is the determination of a reaction mechanism and its associated parameters (i.e. activation energies, reaction rates and heats of reaction).

We present here a new fully automated small-scale reaction calorimeter combining a power-compensation heating element and a thermoelectrically regulated metal surrounding. This dual temperature control makes the reactor particularly suitable for fast and exothermic reactions and eliminates the need for time-consuming calibration of heat transfer coefficients. With a working volume from 25 to 50ml, the device is particularly suited to the fine and pharmaceutical chemical industries, where only small amounts of test substances are available and time to market is crucial.

Thanks to an integrated ATR IR probe coupled to an FT-IR spectrometer complex reaction mechanisms can be studied. Moreover, the design allows the simultaneous use of additional online devices such pH or UV-Vis probes. The performance of the new reaction calorimeter has been successfully demonstrated based on several reaction examples: the hydrolysis of acetic anhydride; a highly exothermic acetylation of a substituted benzopyranol; and palladium-catalysed hydrogenation of nitro-compounds under high pressure.

This work is closely related to the development of an evaluation algorithm to extract kinetic information from different measurement signals to obtain a single set of reaction parameters.