Kinetics Screening in Early Stages of Process Development

A. Zogg, U. Fischer, K. Hungerbühler

This project ***focuses*** on the development of a ***small reaction calorimeter with integrated FT-IR probe*** that allows a rather quick gathering of data on kinetic and thermodynamic parameters of a reaction (heats of reaction, rate constants, phase and heat transfer coefficients). Such a device is of particular importance in view of ***process optimization*** at early stages of chemical process design, especially for the synthesis of new fine chemicals and pharmaceutical products, where generally only ***small amounts of test substances*** are available and time-to-market is crucial.

Fig. 1 shows a ***reaction calorimeter*** with a sample volume of ***20-45 ml***. To measure the heat of reaction at ***isothermal conditions***, in this device the ***power compensation*** principle (internal compensation heating) has been combined with the ***heat balance*** approach (external thermoelectric modules). This combination of two calorimetric measuring principles allows the direct measurement of the heat evolved during a reaction without a mathematical baseline correction. This concept and its realization with thermoelectric modules and a teflon-coated metal block as reactor jacket ***has already been patented*** [1]. The calorimeter shown in *Fig. 1* can be operated at temperatures between ***-20 and 200 °C*** and pressures of ***0 up to 10 bar***. It is equipped with a magnetic coupling for the stirrer (*cf. Fig. 1*), with one pressure and several temperature sensors, and two inlets.

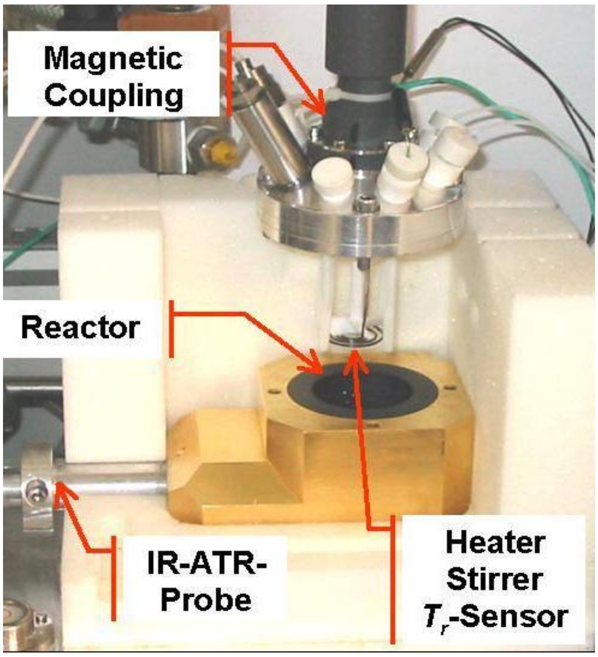

Fig. 1 Small-scale reaction calorimeter (sample volume 20 - 45 ml) with reactor jacket built of teflon-coated copper.

The ***evaluation*** of the measured data is another crucial and time-consuming step and no tools are available yet for a simultaneous evaluation of both the thermal (*Fig. 2*) and the spectroscopic signal (*Fig. 3*). Therefore, we are developing an evaluation algorithm using non-linear optimization in order to estimate the unknown reaction parameters such as heats of reaction, rate constants and phase transfer coefficients.

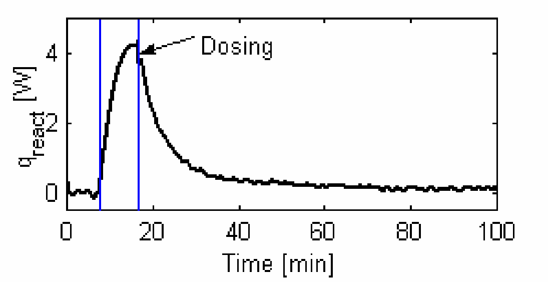

Fig. 2 Heat of reaction versus time course measured with the new calorimeter. The heterogeneous reaction measured can be described as follows:

$$A_{(s)} \rightleftharpoons A_{(l)} \xrightarrow{B} C$$

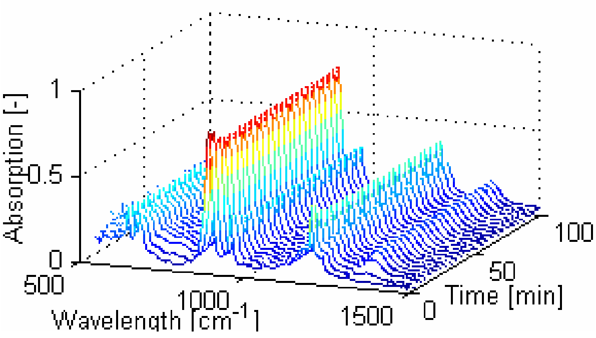

Fig. 3 IR spectra measured online using the new calorimeter. Same reaction as shown in *Fig. 2*.

References

(1) A. Zogg, M. Wohlwend, U. Fischer, K. Hungerbühler, EP 1184649