Small-scale reactor for data oriented process development

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During the early stages of research and development chemical industry needs flexible and versatile tools to investigate 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, rates and heat of reactions).

We present here a new fully automated small-scale reaction calorimeter combining a power-compensation heater and a thermoelectrically regulated metal surrounding. This dual temperature control makes the reactor highly 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 45ml the device is particularly suited for the fine and pharmaceutical chemical industries where only small amounts of test substances are available.

An integrated ATR-IR probe coupled to an FT-IR spectrometer allows the investigation of complex reaction mechanisms. Moreover, the new reactor design allows the simultaneous use of various additional in-situ analytics such as UV-Vis, gas intake/uptake and particle size analysis. The performance of the new reaction calorimeter has already been successfully demonstrated based on several reactions [1,2,3].

Different analytical techniques may point to different optimum reaction parameters. Algorithms and mathematical tools, such as multi-objective multivariate kinetic modelling, become an important research area in data oriented process development. Only a short introduction into these methods will be given here.

[1] Zogg, A., Fischer, U., & Hungerbühler, K. (2003). A new small-scale reaction calorimeter that combines the principles of power compensation and heat balance. *Industrial & Engineering Chemistry Research*, *42*, 767-776.

[2] Visentin, F., Gianoli, S. I., Zogg, A., Kut, O. M., & Hungerbühler, K. (2004). Pressure-resistant small-scale reaction calorimeter that combines the principles of power compensation and heat balance (CRC.v4). *Organic Process Research & Development*, *8*(5), 725-737.

[3] Visentin, F., Puxty, G., Kut, O. M., & Hungerbühler, K. (2006). Study of the hydrogenation of selected nitro compounds by simultaneous measurements of calorimetric, FT-IR, and gas-uptake signals. *Industrial & Engineering Chemistry Research*, *45*(13), 4544-4553.