SMALL SCALE REACTION CALORIMETERY

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To meet the need for a systematic and quick gathering **of kinetic and thermodynamic reaction parameters** in early phases of process design **a small reaction calorimeter (25 to 45 ml) with an integrated IR-ATR probe** was developed during this work.[1,2,6] The new prototype reaction calorimeter (see Fig.1) uses a copper block as an intermediate thermostat instead of a double wall vessel (typically glass) with a circulation fluid.

The reaction temperature is controlled at **isothermal conditions using the Power-Compensation principle**. To allow an **online measured baseline** (compensate changes of the heat transfer through the reactor wall during the reaction) **an additional heat-flow balance using Peltier elements is implemented and was patented.**[1] Due to this combination **no calibration steps are required**, and several devices could be connected to the same cryostat (parallelisation). The new calorimeter has a very **small time constant of about 4 s** (time constant of the jacket about 15 s) and is therefore ideally suited to measure **fast and highly exothermal** reactions at isothermal conditions.[2,5,6]

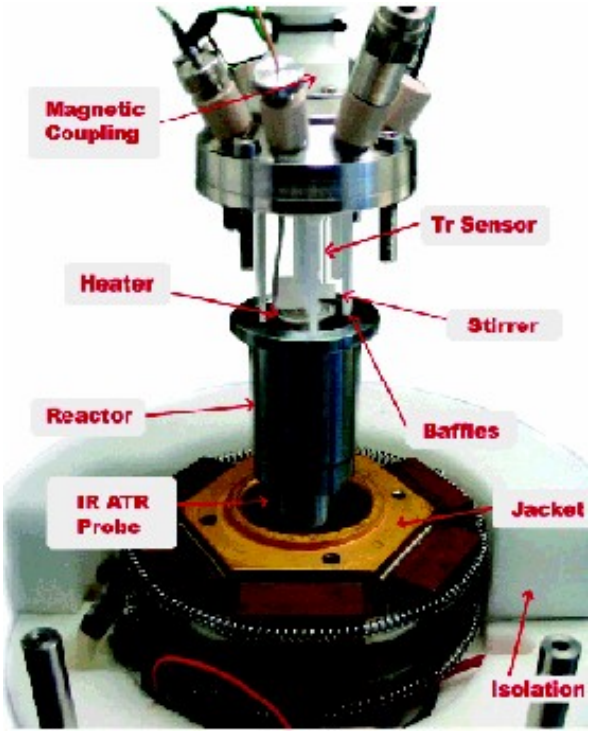

Fig. 1 Latest version of the small-scale reaction calorimeter (sample volume 20 - 45 ml) with reactor jacked made of copper and removable reactor (Hasteloy C22). The reactor is pressure resistant up to 20 bar, Temperature rage -20 … 200 °C.[6]

The performance and the accuracy of the new device will be demonstrated based on several reaction examples: The **neutralization of NaOH with H_2SO_4,** the **hydrolysis of acetic anhydride,** as well as two **highly exothermal industrial reactions** (maximal reaction power of ≈ 2.5 kW/l).

Furthermore a **new evaluation principle** for the measured reaction was developed [2,3,4,5] that allows the identification of the unknown reaction parameters such as rate constants, activation energies, reaction orders and reaction enthalpies. The evaluation is based on an empirical reaction model. **In contrast to conventional evaluation procedures the infrared and calorimetric data are simultaneously evaluated.** Only a short introduction into this evaluation algorithm will be given.

References

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