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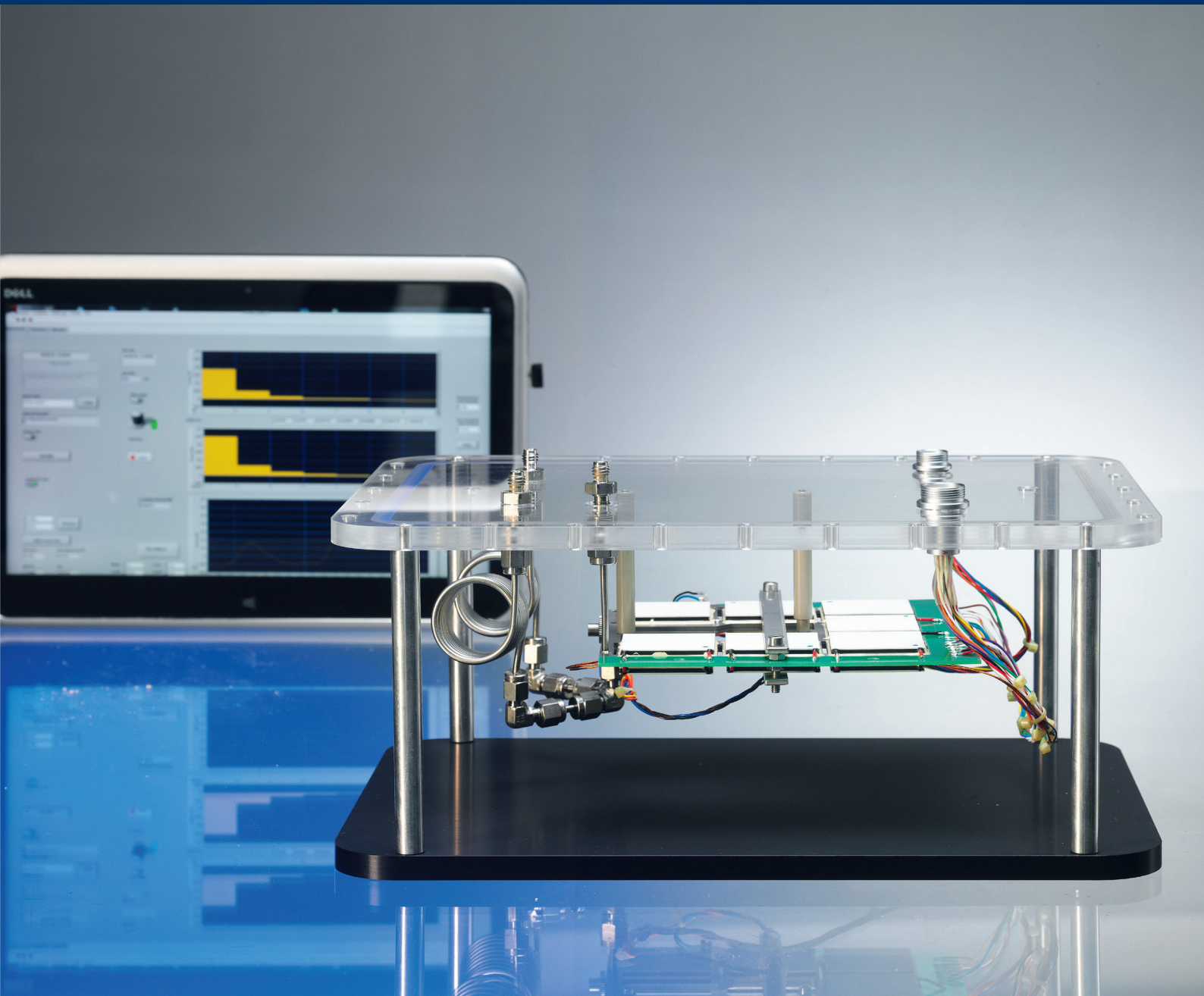
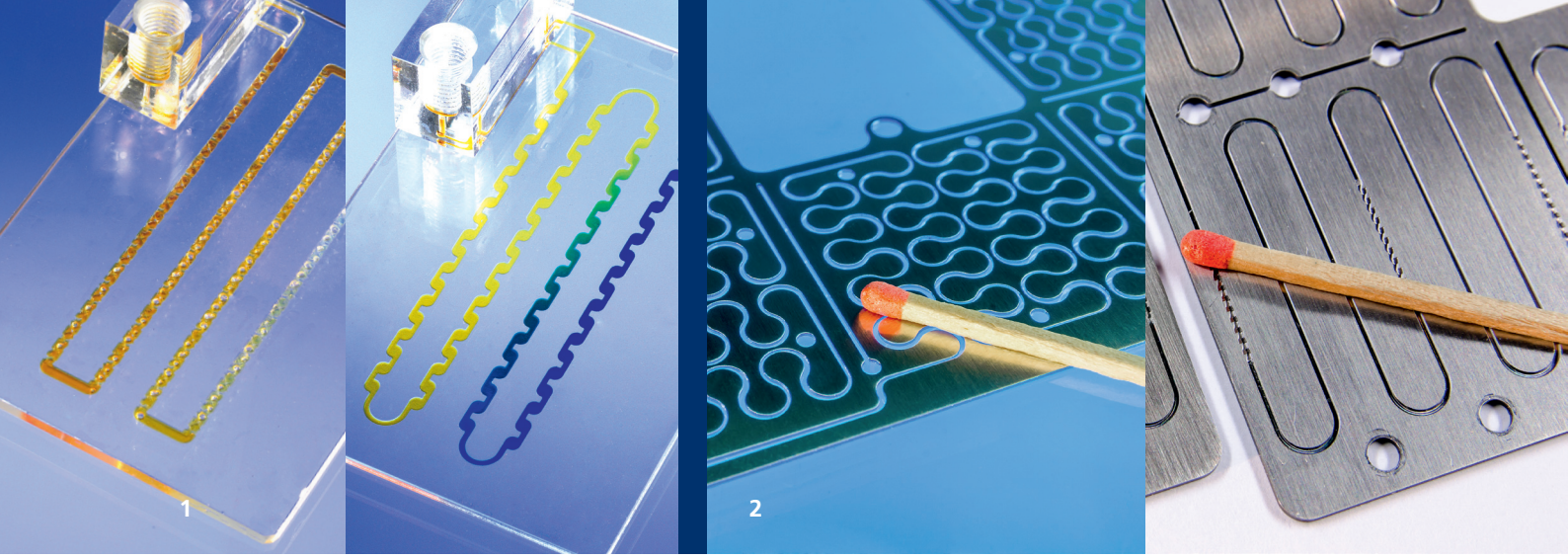
FRAUNHOFER INSTITUTE FOR CHEMICAL TECHNOLOGY ICT

REACTION CALORIMETRY IN MICROREACTORS
FAST REACTION SCREENING AND PROCESS DESIGN





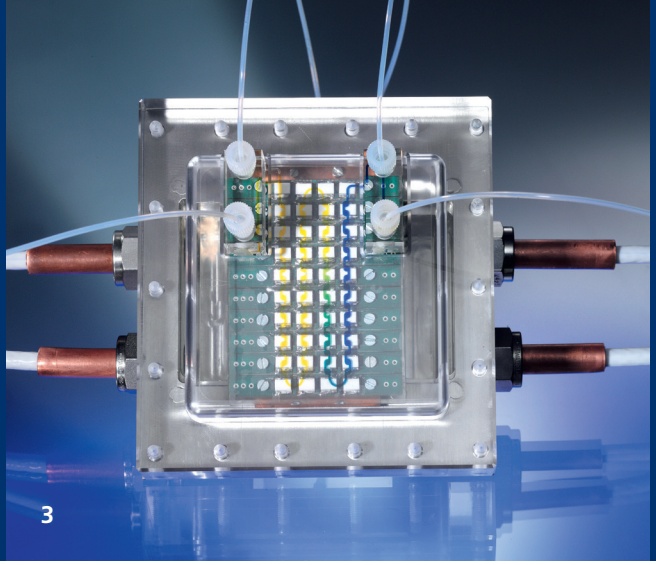
To keep the time to market as short as possible, detailed knowledge about a chemical reaction system must be available in an early phase of process development, facilitating dependable decisions about synthesis strategies and process management. The analytical methods used in fast reaction screenings should therefore offer a high volume of information within a short experimental period. The combination of microreaction technology with calorimetric measurement technology is an extremely powerful process analytical technique to identify key features of the process.

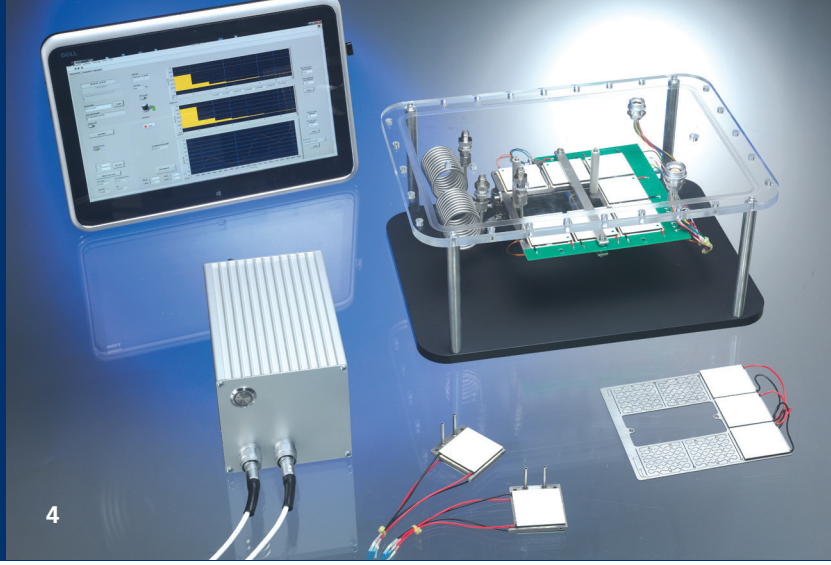
The Fraunhofer ICT develops continuously operating reaction calorimeters based on microreactors, which permit rapid screening of thermokinetic key data. At the heart of the calorimeter are sensor arrays based on Seebeck elements for the localized, quantitative characterization of heat flows. The sensor arrays consist of up to 40 individual sensors, which can collect data concerning the reaction heat generated in a microreactor with a high degree of temporal and spatial resolution. The measurement of the heat flows has the advantage that the heat release rate of the reaction is directly proportional to the reaction rate. This allows easy access to basic kinetic and thermodynamic data of chemical reactions (heat of reaction, reaction rate and conversion). The microreactors themselves may be made of various materials; moreover, they may be exchanged to adapt the most appropriate microstructured reactor to the reaction being analyzed, for example in terms of residence time or mixing performance. The measurement software, based on LabVIEW, provides graphic visualization of the measured heat flows.

During parameter screenings, the influence of individual process parameters (e. g. concentration, stoichiometry, use of alternative reactants, temperature, residence time, etc.) on the reaction rate can be followed directly by observing the altered heat signal – both in a qualitative and quantitative manner. The measurement of the heat flows is in real-time; time-consuming calibrations for the heat transfer are not required. Due to the large surface-to-volume ratio of microstructured reactors and the resulting small time constants of just a few seconds, it is also possible to perform isothermal calorimetric measurements for rapid exothermic reactions. The smallest test volume is about 100 µl, reducing material consumption to a minimum, which is particularly important in the case of expensive chemicals. Even targeted investigations and quantitative analysis of the energetic potential of critical process conditions (worst case scenarios) can be conducted safely.

The combination of continuous microreaction technology and efficient heat flow sensors based on Seebeck elements thus paves the way toward a rapid and extremely efficient screening of reaction and safety-related parameters. The measurement of heat flows is therefore a core technology for developing new and rapid process analytical techniques.

1 *Flow reactors with different mixing characteristics are available, covering a broad range of chemical reactions.*
2 *Reaction plates with different mixing-structures: Dean and caterpillar mixer (Photo: Fraunhofer IMM).*





Important advantages at a glance

- Reaction calorimetry in real-time
- Determination of thermokinetic and safety-relevant key data
- Rapid screening of reaction and process conditions
- Isothermal measurements even for rapid, strongly exothermic processes
- No costly and time-intensive calibrations, simple to operate
- Small test volumes (<100 µl): minimal substance consumption
- Safe analysis of potentially dangerous reactions, even critical process conditions can be investigated
- The measurement principle can be adapted to a wide range of flow reactors and process conditions (temperature up to 200 °C; pressure up to 100 bar, reaction volume up to 10 ml)

Our products and services

We provide our customers and project partners with reaction calorimetry solutions based on microreactor technology for the investigation and analysis of their chemical processes. We determine the thermokinetic parameters for their reactions and test the behavior of their reaction systems under critical process conditions. Based on parameter screenings, we determine relevant process parameters for process design and optimization.

We supply calorimetric measurement systems with the requested specifications and equipment to enable our customers to perform their own research. For special reaction processes and new areas of application, we develop customer-specific solutions. The spatial and temporal resolution of the heat flow measurement can be adapted to suit the intended application.

In addition, we offer a broad portfolio of other calorimetric measurement techniques as well as extensive R&D services in the areas of chemical process development, process optimization and safety technology.

3 *Calorimetric setup: The sensor array consists of up to 40 Seebeck elements for the calorimetric monitoring of the reaction progress in the microreactor. The sensor array can be adjusted to the dimensions of the microfluidic component being used.*

4 *Reaction calorimetry for real-time measurement of heat flows in microreactors. The modular concept allows adaptation to a wide range of flow reactors and process conditions, for example in terms of residence time or mixing performance. The measurement software, based on LabVIEW, provides graphic visualization of the measured heat flows.*

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