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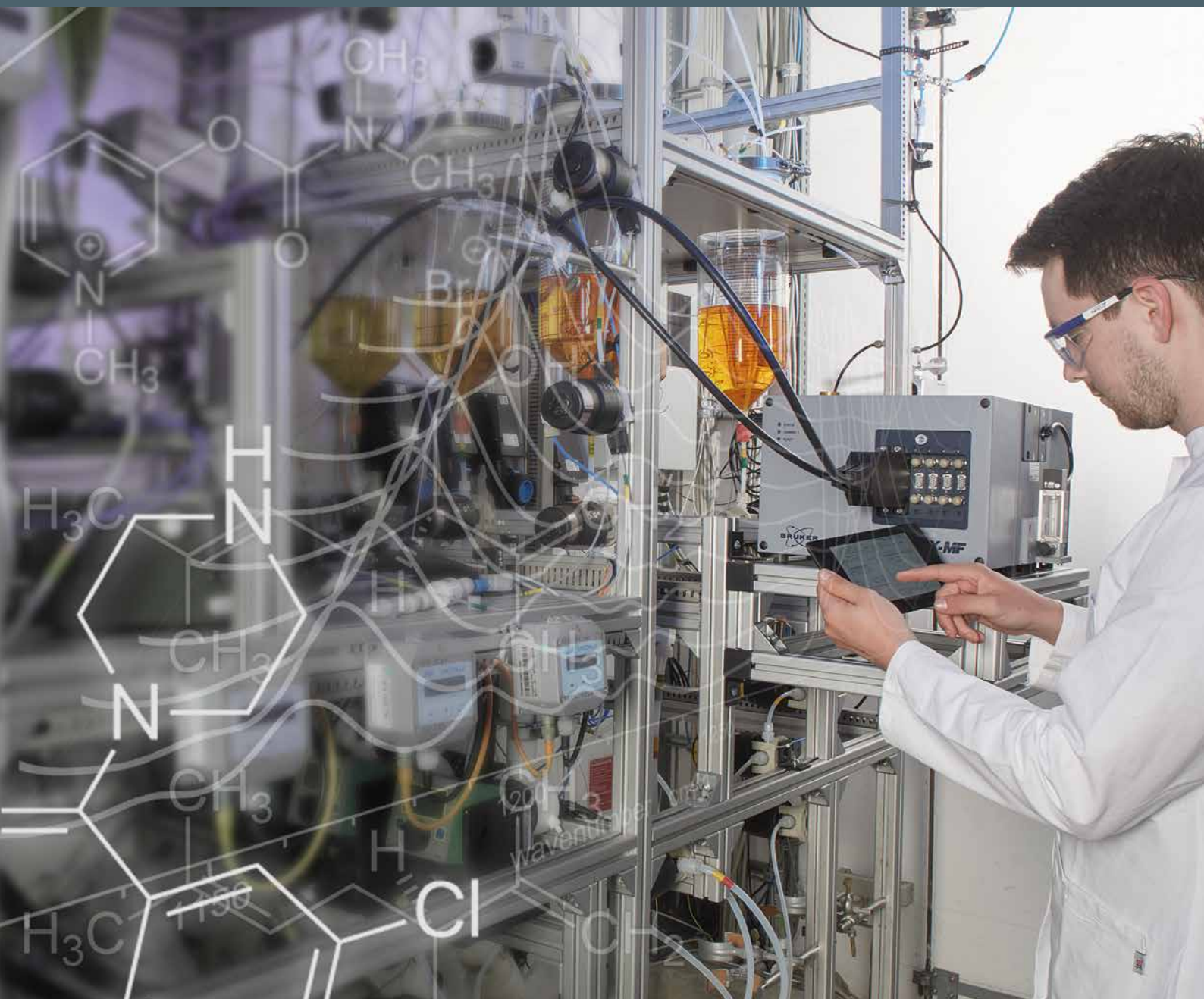
ICT

FRAUNHOFER INSTITUTE FOR CHEMICAL TECHNOLOGY ICT

CONTINUOUS CHEMICAL PROCESSING
DESIGN AND OPTIMIZATION OF CHEMICAL PROCESSES FOR FLEXIBLE PRODUCTION



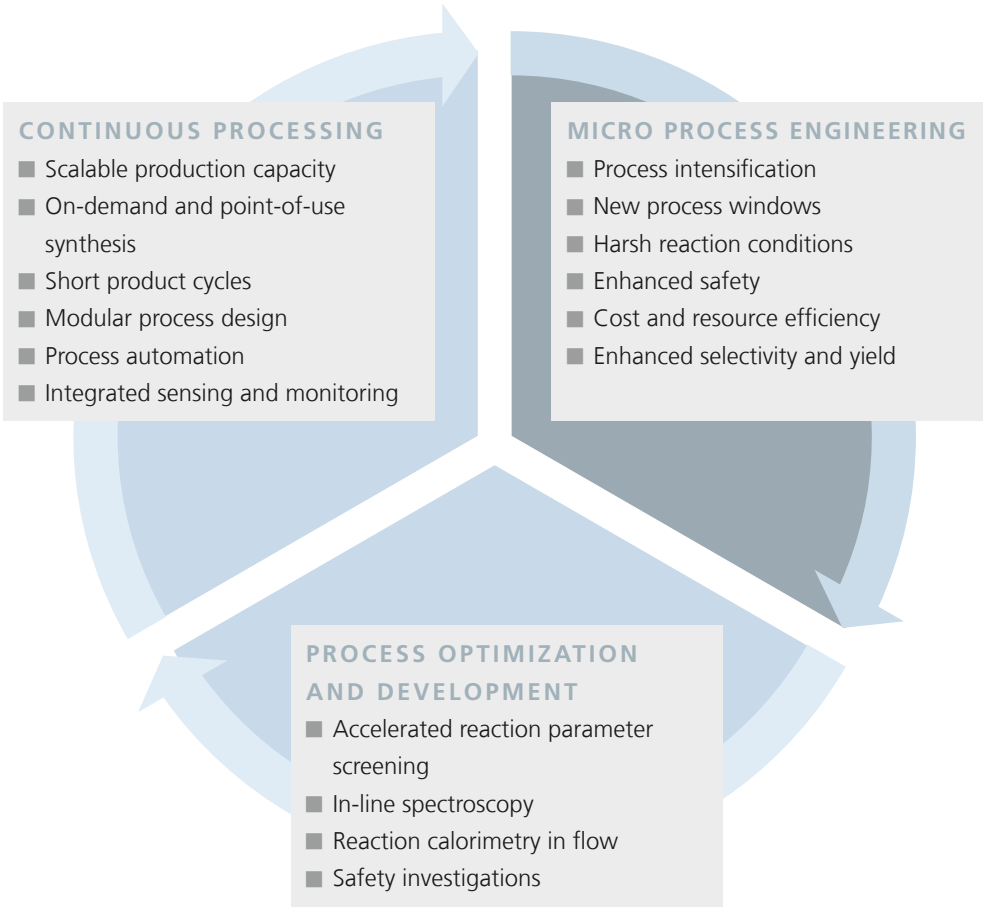
CONTINUOUS CHEMICAL PROCESSING
DESIGN AND OPTIMIZATION OF CHEMICAL PROCESSES FOR FLEXIBLE PRODUCTION

Versatile customer requirements, strong competition and a dynamic market within the chemical industry have led to a demand for flexible but yet increasingly efficient processes and rapid process development.

Continuous chemical processing, which makes use of intensified and microstructured process equipment, is a key element in the future production concepts of chemical high-value products.

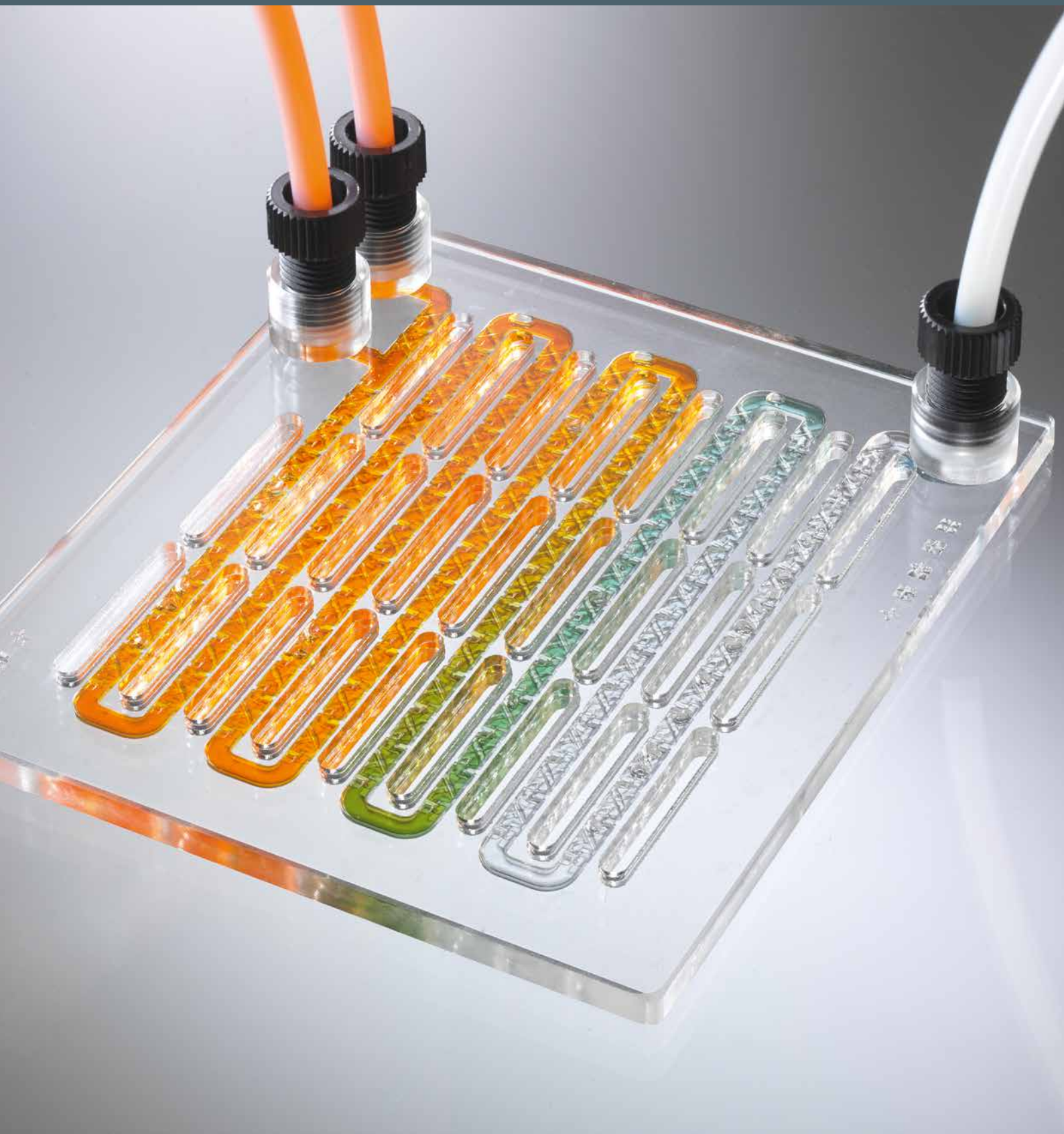
PHOTO LEFT

Spatially resolved spectroscopic process analysis in microreactors using fiber optics.



MICRO PROCESS ENGINEERING

FRAUNHOFER ICT HAS BEEN DEVELOPING MICROREACTION PROCESSES FOR 20 YEARS, FOR PROCESS OPTIMIZATION AND PRODUCTION



CHARACTERISTICS

- High suface-to-volume ratio
- Channel dimensions in the sub-millimeter range
- Short residence times
- Small hold-up
- Intensified multiphase processing

BENEFITS

- Significant improvement of heat and mass transfer characteristics
- Precisely adjustable process conditions
- Higher selectivity and yield
- Enhanced process safety
- Flexible, on-demand production

TOOLS

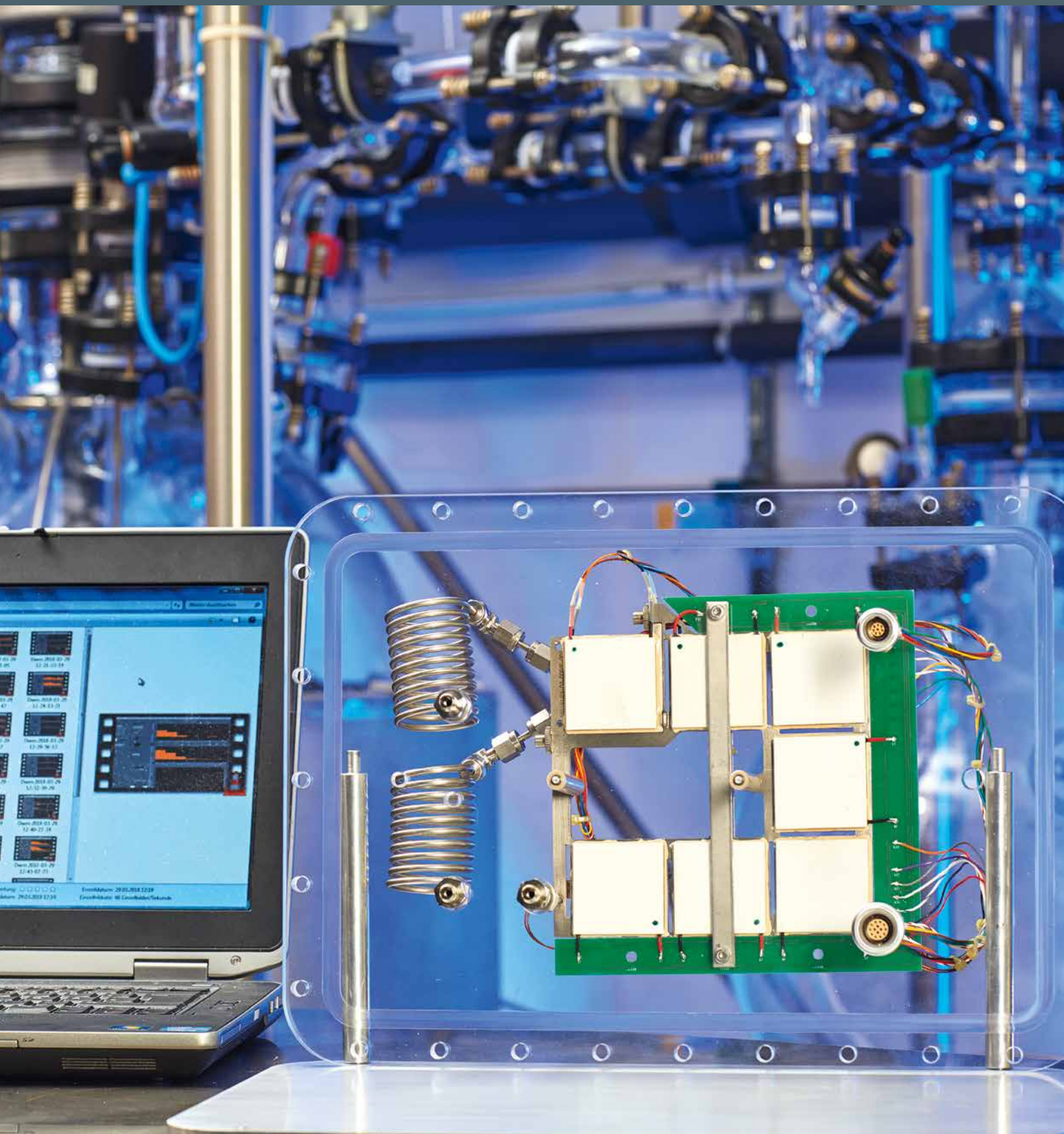
- Microstructured reactors ranging from lab to production scale
- Process analytical technology (PAT) with high temporal and spatial resolution
- Integrated downstream processing
- Simulation and modeling

Customers and project partners from the chemical, pharmaceutical and process technology industries can now access a wide range of products, processes and services in the area of micro process engineering and microreaction technology, extending from the analysis, design and optimization of chemical processes to the synthesis of specialty and fine chemicals, and the development of tailored microreaction systems for use in laboratories and production plants.

PHOTO LEFT

Passive mixing structures intensify mass transport.

FROM PROCESS OPTIMIZATION TO PRODUCTION



Micro process technology and flow chemistry are key tools for process optimization and process development for future flexible production. The knowledge obtained can be transferred effectively to production tasks on a technical scale.

DRIVERS AND GOALS FOR PROCESS OPTIMIZATION

- Economically attractive synthesis routes
- Reduction of reaction steps
- High yields and selectivities
- Minimizing hazardous potential
- Applying 'forbidden' reaction conditions
- Catalytic processes
- Photo chemistry

PROCESS OPTIMIZATION AT LABORATORY SCALE

- Modular lab systems for fast reaction and parameter screenings
- Wide selection of micro-structured reactors
- Reactor prototype manufacturing by ultrashort pulse laser ablation
- Fully automated lab systems with data logging
- Systematic and automated experimental plans based on design of experiments (DoE)
- Integrated spectroscopic and calorimetric online analysis
- Remote control for processes with high hazardous potential

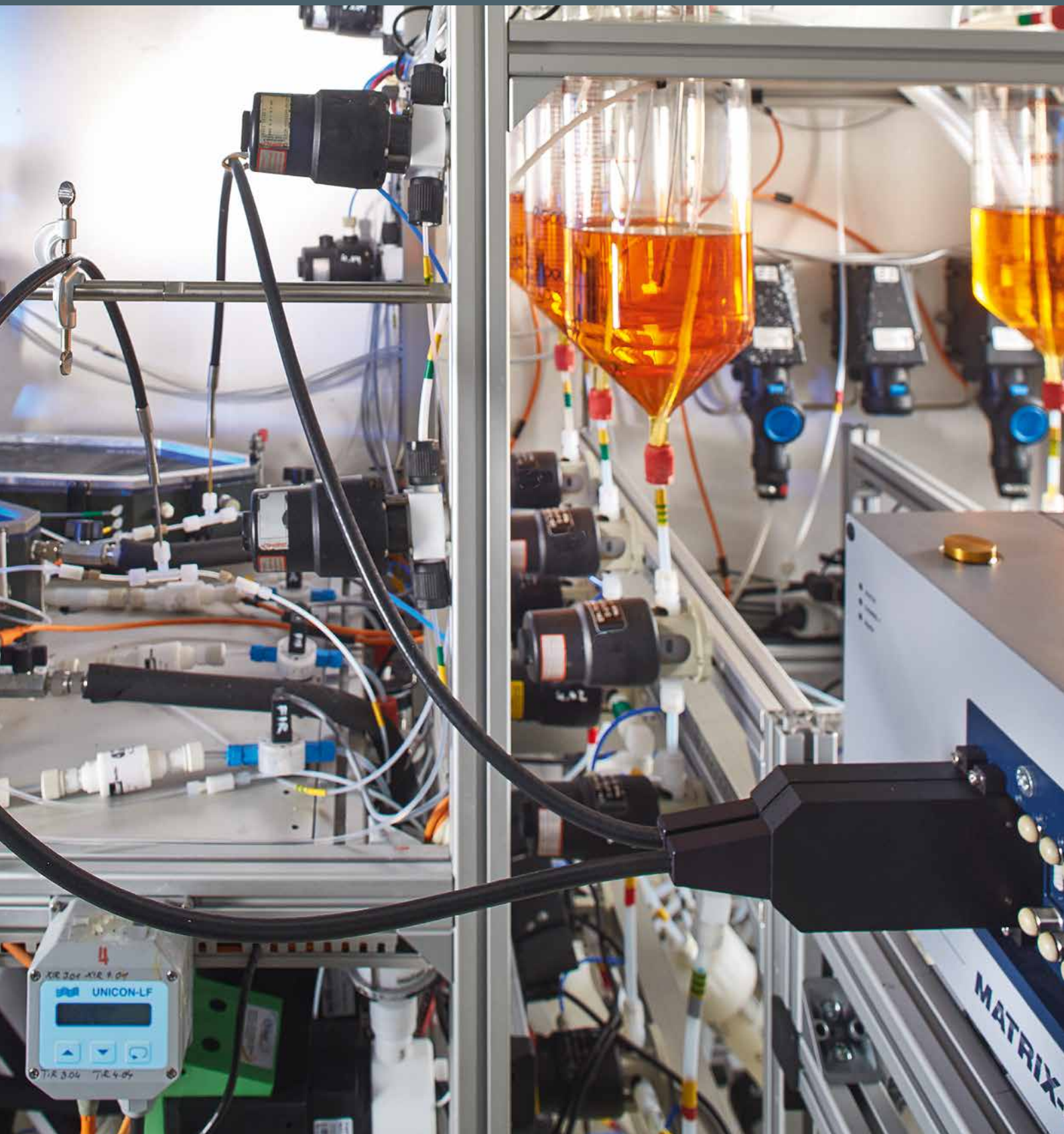
PRODUCTION AT TECHNICAL SCALE

- Transfer of continuous processes from lab to technical kg-scale
- Tailored design of micro-structured reactors
- Customer specific processes with high-throughput
- Continuous downstream processing and purification
- Scalable on-site production

PHOTO LEFT

Time and spatially resolved reaction calorimetry in microreactors using heat flow sensor arrays.

SPECTROSCOPIC AND CALORIMETRIC PROCESS ANALYSIS AND DATA-DRIVEN CONTROL OF PROCESSES



At Fraunhofer ICT process analysis is an essential tool for the design, diagnostics, optimization and control of chemical processes. Spectroscopic and calorimetric process analysis are adapted to continuous and microreaction processes as inline, online or at-line measurement technology. In particular, techniques with a high degree of temporal and spatial resolution are used:

PUSHBROOM-IMAGING (UV/Vis, NIR)

Spectroscopic monitoring in real-time with a high spatial resolution in a selected section of a reactor or at discrete measuring points along flow direction

MULTIPLEX SPECTROSCOPY (MIR, NIR, RAMAN)

Tracking of chemical processes at multiple measuring points with high chemical selectivity

QUANTUM CASCADE LASER SPECTROSCOPY (MIR)

New semiconductor lasers with high spectral brightness for next-level infrared process analyzers

SURFACE ENHANCED RAMAN SPECTROSCOPY (SERS)

Online and at-line Raman analysis with superior sensitivity and selectivity for screening applications

SPATIALLY RESOLVED FLOW CALORIMETRY

Real-time calorimetric monitoring of processes regarding thermokinetic data, reaction enthalpies and safety data

Advanced process control practices and data-driven control based on integrated sensors are crucial for future continuous processes. We use all our process analysis tools to create closed loop control for adaptable process automation. Smart control strategies contribute significantly to product quality, resource efficiency and process safety.

PHOTO LEFT

Multiplex IR-spectroscopy based on optical fibers in a continuous microreactor process at technical scale.

SAFE PRODUCTION OF HAZARDOUS PRODUCTS

A special field of research at Fraunhofer ICT is the development of processes for the safe management of reactions that are potentially explosive or otherwise hazardous. We have more than 50 years of expertise in the area of explosive synthesis as well as the associated infrastructure and safety equipment.

Advantages of micro process engineering for processes with increased hazard potential:

- Fast removal of strong reaction heat
- Suppression of unwanted side or decomposition reactions
- Small hold-up
- Safer handling of toxic, explosive or otherwise unstable products and intermediates
- Point of use and on-demand production of hazardous intermediates

Fraunhofer ICT has developed special multipurpose plants at technical scale for both the continuous synthesis of liquid explosive materials and their subsequent, continuous downstream processing in relevant production capacities. Microreactors have been specially developed for these processes, allowing high throughput for both synthesis and downstream processing. A modular process concept allows the flexible adjustment of process equipment to match the synthesis product and throughput required.

All plants have a wide range of safety features and every aspect is controlled and monitored remotely. Multipoint in-line spectroscopic measurement can be implemented as an option for the closed-loop control of crucial process variables, contributing significantly to process safety and the efficient consumption of reagents.

Multipurpose microreactor plants are used for the production of a variety of explosive substances and are set up according to customers' specifications. Typical throughputs are in the range of several hundred grams per minute.

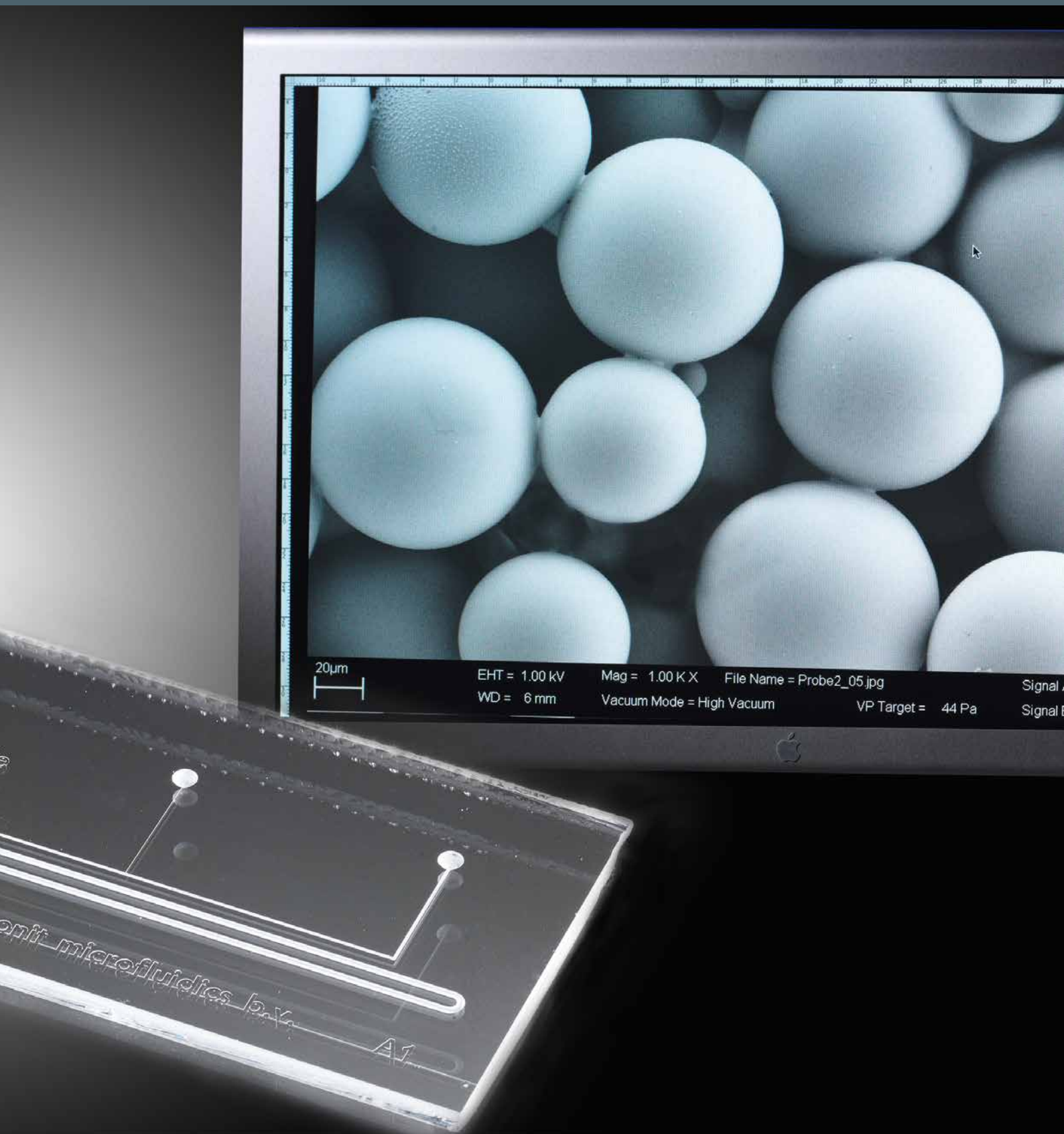
PHOTO LEFT

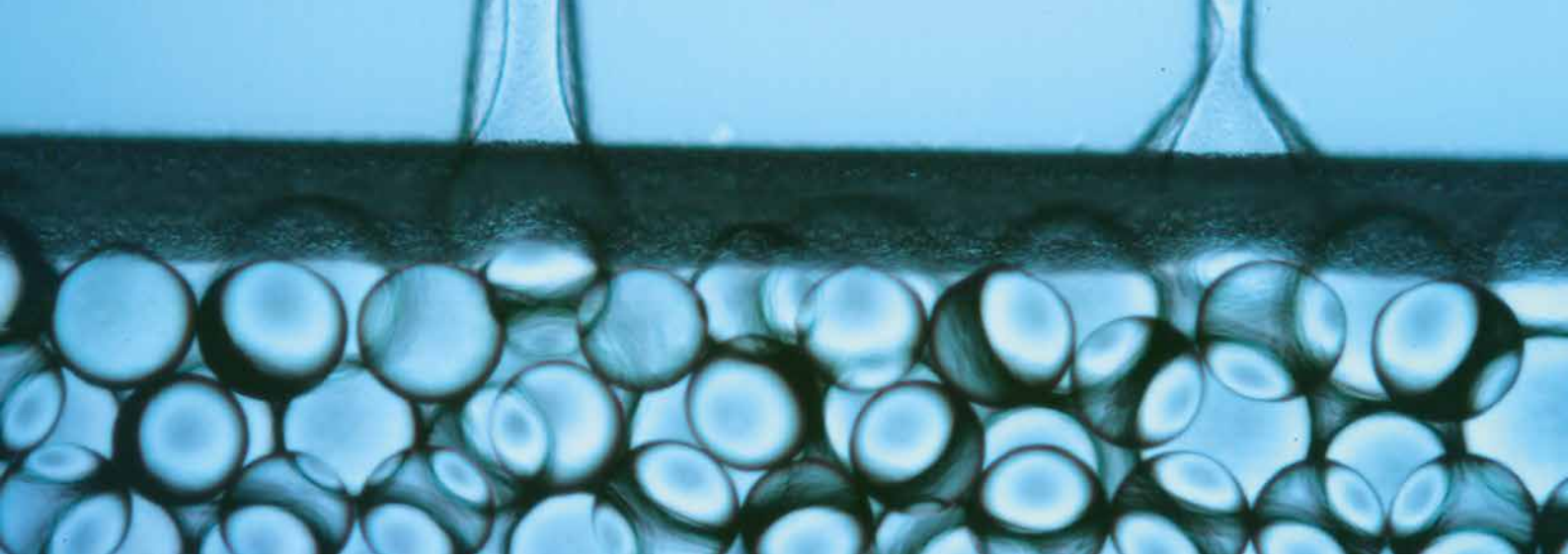
Pilot plant for the synthesis of explosive substances under safe conditions.

PHOTO ABOVE

Production of hazardous substances in a remotely controlled continuous process.

MULTIPHASE PROCESSING
PARTICLE SYNTHESIS AND MICROENCAPSULATION





Microfluidic structures can also be used for the high-precision processing of multi-phase systems in the form of segmented flows and unimodal emulsions, allowing the synthesis of particle size-controlled solid materials with properties superior to those achieved by conventional processing.

For example, precise control of the droplet size in emulsions is used to synthesize monomodal spherical polymer particles and microcapsules which can be filled with a wide range of active ingredients during the process.

A current field of research at Fraunhofer ICT is the increase of throughput which is attained by parallelization of microfluidic channel elements while maintaining exceptional properties regarding droplet / particle size control and dispersitivity.

In segmented flow processing, individual fluid segments function as closed reaction vessels, and have no chemical interaction with the transport phase. Within the fluid segments – each with a volume of just a few nanoliters – the syntheses of high value products can be performed by suppressing cross-contamination, dilution and dispersion effects caused by convection and diffusion. Moreover, mixing of the reagents in the nanoliter segment is strongly intensified by advection, without the need for complex static mixing structures which are usually required in microreactors. As a result, even liquids containing solid matter can be handled. We use segmented flows to synthesize nanoparticles and other crystalline materials, e.g. metal-organic frameworks (MOFs), a new class of solid microporous materials that are characterized by extremely large specific pore volumes, large specific surfaces and the possibility to precisely tune their chemical and physical properties.

On the other hand, segmented flow processing can also be used to deliberately intensify interaction between two-phase systems. By providing large interfacial areas we can significantly accelerate mass transport over the phase boundary layers compared to macroscopic processes. Typical applications are phase transfer catalysis and other two-phase organic syntheses.

PHOTO LEFT

Microreactor for manufacturing spherical polymer particles.

Parallelized microfluidic channels for high-throughput droplet generation.

DESIGN, FABRICATION AND LASER STRUCTURING OF MICROSTRUCTURED REACTORS

One essential element in the development of chemical processes is the design of tailored reactors, process apparatuses and microfluidic components. Fraunhofer ICT uses both mathematical methods and numerical simulation tools as well as standardized experimental measurement methods to obtain quantitative and qualitative data concerning important performance characteristics, for example flow distribution, mixing efficiency, residence time behavior, etc.

The design of tailored reactors and microfluidic components requires flexible micro-structuring techniques allowing the fast development and testing of microfluidic prototypes and a rapid re-designing of microfluidic components. Laser ablation is a powerful technique for the rapid generation of microstructures in various substrates. Focused picosecond laser pulses allow controlled and well-defined material removal on a micro-scale. The microstructures exhibit an excellent geometric precision and can be obtained with high aspect ratios. In contrast to conventional processes for generating microfluidic structures, such as wet etching or sandblasting, no masks are required, saving time and resources in the process development. Re-designs of a microfluidic structure can be quickly executed by simply adjusting the 3D-CAD data according to the new specifications.

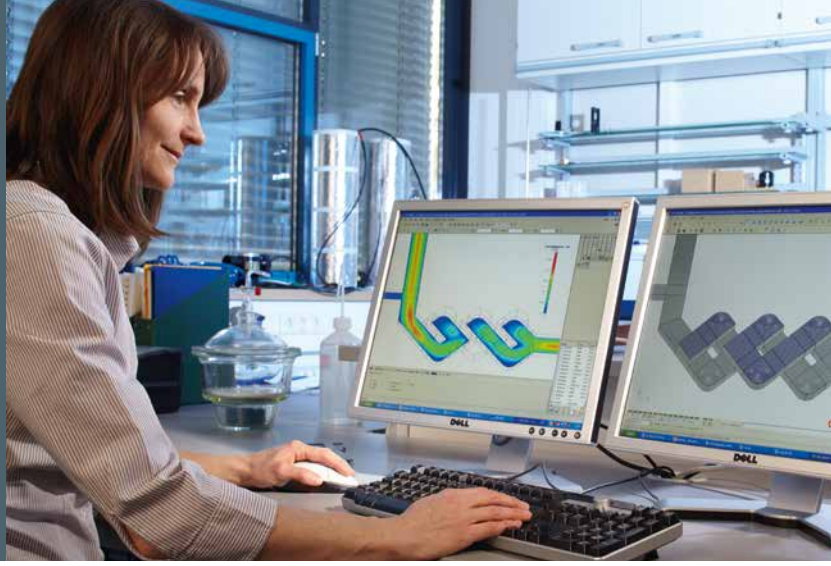
At Fraunhofer ICT glass is mainly used as the substrate due to its transparency, chemical inertness and mechanical strength. Nevertheless, various substrates such as ceramics, polymers and metals are also processed by the same laser system.

PHOTO LEFT

Micro-structuring of glass reactors by ultrashort pulse laser ablation.

PHOTO RIGHT

Visualization of the fluid dynamics in threedimensional microchannel structures using CFD.



OUR PRODUCTS AND SERVICES

We provide our customers and project partners with rapid and comprehensive access to the diverse applications of continuous processing, micro process engineering and flow chemistry.

Fraunhofer ICT offers a broad variety of solutions in the fields of chemical synthesis and process development, process optimization and process analytical technology.

As R&D services, we also offer feasibility studies, rapid parameter screenings and targeted analysis of individual process steps as well as detailed safety investigations.

Based on our 20 years of experience, we develop tailored microreactor processes for customer-specific tasks in all areas from the laboratory to production scale.

To enable our customers to perform their own research, we supply complete laboratory systems for synthesis, process analysis and calorimetry.

Finally, we develop products together with our customers in the areas of fine and speciality chemicals as well as manufacturing of microcapsules and micro/nano particles.

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