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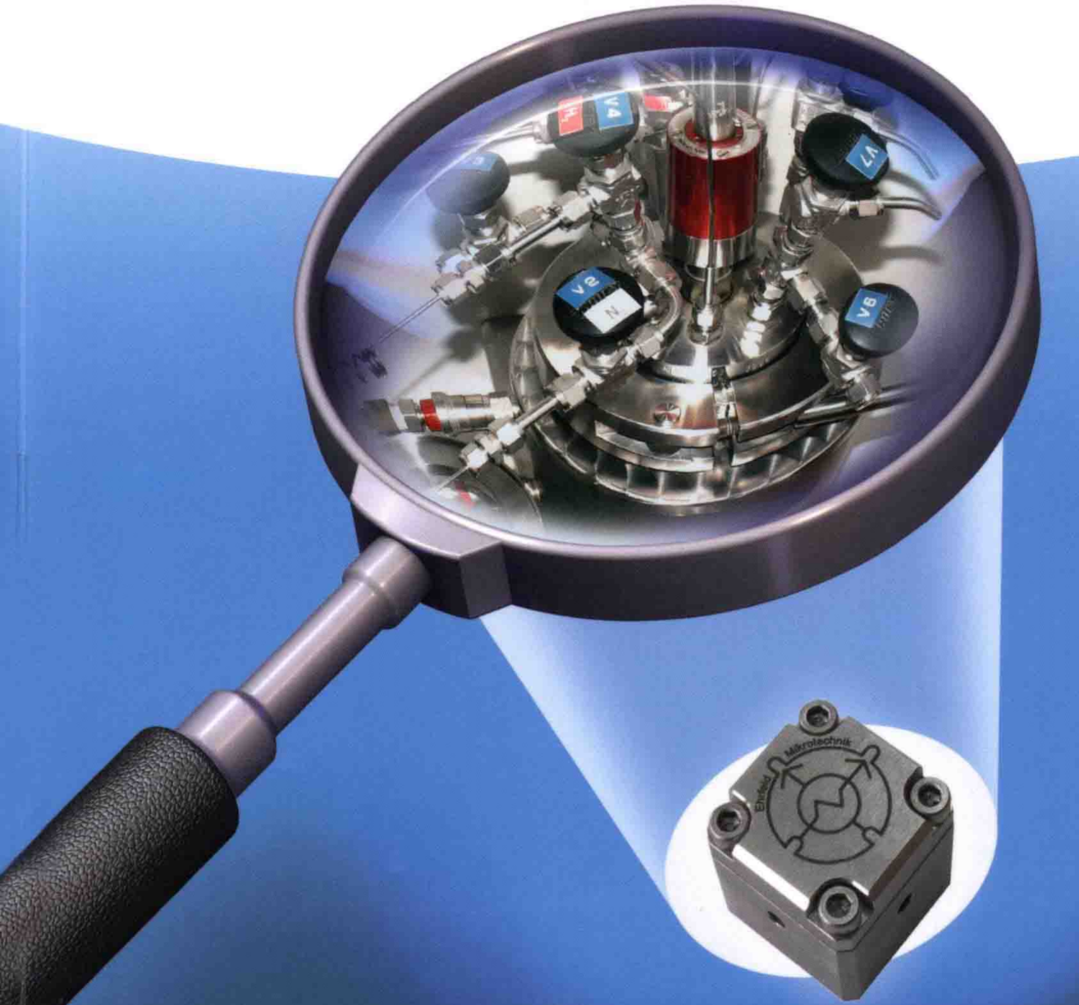
Microreactors in Preparative Chemistry

Practical Aspects in Bioprocessing, Nanotechnology, Catalysis and more



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Preface

At the beginning of the twenty-first century, the transfer of microreaction technology to the industrial sector remains in focus. Knowledge about the rate of chemical reactions as well as about heat and mass transfer processes is particularly essential. Since less time is required for the production of the desired product in the given reaction volume, a higher space–time yield – a measure of the reactor performance and consequently of the efficiency of the process guiding – can be obtained. Nevertheless, in spite of a large number of organic syntheses, which were successfully carried out in microstructured reactors, polymerization reactions, biocatalytic and electrocatalytic conversions as well as heterogeneously catalyzed reactions, or syntheses of inorganic nanoparticles still leave a lot to be desired. Moreover, the handling with this technology, especially in the area of the preparative chemistry, has not yet been described in sufficient detail up to now.

This book should help to clear out these existing deficits and give useful information for anyone to consider the application of microreaction technology regarding problem solving in preparative chemistry. Therefore, this book includes not only a number of reaction types that have already been described in the original literature and patents, but also a balance between the well-chosen research highlights and the general practical aspects resulting from it. Thus, careful consideration to the basic theoretical principles of the reaction in microreactors is given, so that the book appeals not only to specialists, but also to those who have just begun to deal with the application of the microreaction technology for preparative purposes. Moreover, specific instructions and test procedures for verified product syntheses are provided and therefore facilitate the collection of own practical experiences with the microreactor equipment. Hence, the topics discussed in the book assume a form that makes the practical discussion of research- and development-oriented problems comprehensible for both the specialist and the newcomer. Readers will obtain not only an understanding of the advantages of microstructured reactors, but also guidance as to the demands concerning used chemicals, production, pressure loss, and blockage danger. In addition, information is provided in matters of computer-supported measuring, regulation of temperature, pressure, flow rate, concentration, and quantitative proportions of the reactants even up to the special demands of miniaturized analysis systems such as the "lab-on-a-chip." Ultimately integrated modular microsystems are described, which consist of microreactors, separation units, and analytic components presenting adaptable tools for the preparative chemist. Faster as well as economically and ecologically more favorable routes for the synthesis of new products and materials under optimum reaction terms are discussed.

After a short introductory chapter, the progress in the microreaction technology over the past 20 years is reviewed and emphasis put on the fact that implementation into microreactors often leads to better yield, higher safety, and less time and cost of materials involved. Single chapters are summarized according to greatest possible cohesion, that is, in groups by related reactions. Correspondingly, the main focus of the book is directed to the preparative side, for example, to the application of microreactors for organic syntheses, polymer reactions, biocatalytic and electrocatalytic as well as heterogeneously catalyzed conversions, and syntheses of nanoparticles. Besides, practice-oriented solutions are described in conjunction with economical and ecological aspects of the optimum reaction management. At the end of every chapter, the verified synthesis examples of the typical approach, the microreactor test equipment, and analysis techniques are provided in combination with straightforward calculation methods. Especially beginners should be able to obtain a first impression about the world of preparative chemistry in such microstructured apparatuses, preparing them optimally for the later process development.

I would like to thank all authors for their contribution to this book, and also on behalf of the authors I hope that we succeed in reaching a wide range of readers in academia and industry. I thank Wiley-VCH publishers for the invitation to edit this book and comprehensive support in the preparation of this book. Special thanks go to Dr.-Ing. Ekaterina Borovinskaya and Dr. Alexander Rüfer for carefully checking parts of the manuscript.

Dresden
December 2012

Wladimir Reschetilowski

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1
Principles of Microprocess Technology

Wladimir Reschetilowski

1.1
Introduction

The microreactor technology is nowadays the key technology for process intensification. Manufacturers of microreactor systems bring their products to market with slogans like "A Chemical Factory in a Briefcase" or "Lab-on-a-chip." Due to the small dimensions of microstructures, which do not exceed 1 mm, microreactors contribute to the minimization of material in terms of production as well as raw material and energy consumption during exploitation. Moreover, due to the intensification of heat and mass transfer, the productivity of plants with microreactors is in a number of cases significantly higher than that with classical batch reactors applied in industry.

Extensive research efforts have been made incessantly in this field during the past few years. Recent advances in the design and fabrication of microreactors, micromixers, microseparators, and so on show that they represent a cheap alternative for the production of special fine chemicals by a *continuous* process to observe simpler process optimization and rapid design implementation. It is possible to predict that in the near future chemical, pharmaceutical, and biological laboratories will change radically toward considerable improvement of process and synthesis efficiency at essential miniaturization of reactor devices.

One of the key moments in the microprocess technology is the effective way to increase the process productivity by the so-called reproduction (numbering-up) of continuous microreactor systems, that is, a series of continuous reactors works simultaneously. Hereby the dimensions of microreactors and their efficiency in heat exchange do not change, when transferring processes from laboratory to pilot and production scales. Due to the facility to change the process parameters (temperature, pressure, flow velocity, ratio of reagents, use of catalysts, etc.) rapidly and accurately, the microreactor systems can be predestined as an ideal tool for effective and fast optimization of investigated reactions. The full automation of such systems interfaced with integrated analytical devices in real time (online analytic) gives an opportunity to receive high-grade information about optimal parameters of multistage reactions within only a few hours.

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