

Miniaturization of Analytical Systems: Principles, Designs and Applications

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A John Wiley and Sons, Ltd, Publication

This edition first published 2009
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John Wiley & Sons Ltd, The Atrium, Southern Gate, Chichester, West Sussex, PO19 8SQ,
United Kingdom

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Library of Congress Cataloging-in-Publication Data

Rios Castro, Angel.

Miniaturization of analytical systems : principles, designs and applications / Angel Rios, Alberto Escarpa, Bartolome Simonet.

p. cm.

Includes bibliographical references and index.

ISBN 978-0-470-06110-7

1. Chemistry, Analytic. 2. Miniature electronic equipment. I. Escarpa, Alberto. II. Simonet, Bartolome. III. Title.

QD75.3.R56 2009

543--dc22

2009016260

A catalogue record for this book is available from the British Library.

ISBN 978-0-470-06110-7

Set in 11/13pt Times by Thomson Digital, Noida, India.

Printed and bound in Great Britain by CPI Antony Rowe Ltd, Chippenham, Wiltshire.

Contents

<i>Preface</i>	xi
1 Miniaturization in Analytical Chemistry	1
1.1 Introduction	1
1.2 Miniaturization as One of the Critical Trends in Modern Analytical Chemistry	2
1.3 Evolution in the Field of Analytical Miniaturization	8
1.4 Classification of Miniaturized Analytical Systems and Definition of Terms	10
1.5 Theory of Miniaturization	15
1.6 Features of Miniaturized Analytical Systems	17
1.7 Incidences of Miniaturization in the Analytical Process	19
1.7.1 Miniaturization of the Steps of the Analytical Process	20
1.7.2 Integrated (Micro)systems for the Performance of the Entire Analytical Process	30
1.8 Outlook	33
References	36
2 Tools for the Design of Miniaturized Analytical Systems	39
2.1 Introduction	39
2.2 Miniaturized Analytical Processes: The Downsizing and Integrating Phenomena	40
2.2.1 Transport within Microfluidic Systems	42
2.2.2 Microsystem Design from Transport Parameter Information	45
2.3 Microfluidic Devices	45
2.3.1 Microvalves	45
2.3.2 Moving Liquids in Miniaturized Systems	51
2.3.3 Mixers	59
2.3.4 Volume-dispensing and Sample-introduction Devices	63
2.3.5 Detection Systems for Analytical Microsystems	64
2.4 Microtechnology	66
2.4.1 Computer Simulations in Microfluidics	66
2.4.2 Micromachining	68
2.4.3 Packaging of Microsystems	74

2.5	MEMS and NEMS	74
2.5.1	Fabrication and Characterization	75
2.5.2	Functionalization	76
2.5.3	Detection Methods	81
2.6	Outlook	84
	References	88
3	Automation and Miniaturization of Sample Treatment	93
3.1	Introduction	93
3.2	Simplification of Sample Treatment: Microextraction Techniques	94
3.2.1	Calibration in Microextraction Processes	94
3.2.2	Solid Phase Microextraction (SPME) Techniques	95
3.2.3	Liquid Phase Microextraction (LPME) Techniques	114
3.2.4	Comparison of Solid and Liquid Phase Microextraction Techniques	117
3.3	Simplification of Sample Treatment: Continuous Flow Systems	119
3.3.1	Coupling to Gas Chromatography	120
3.3.2	Coupling to Liquid Chromatography	123
3.3.3	Coupling to Capillary Electrophoresis (CE)	127
	References	135
4	Miniaturized Systems for Analytical Separations I: Systems Based on a Hydrodynamic Flow	139
4.1	Introduction	139
4.2	The Earliest Example of Miniaturization of a Gas Chromatograph and Some Other Developments	141
4.3	Capillary Liquid Chromatography (CLC)	144
4.4	Liquid Chromatography on Microchips	150
4.4.1	The Agilent HPLC Chip	150
4.4.2	Other Approaches to Microchip HPLC	155
4.4.3	Some Selected Applications	157
	References	163
5	Miniaturized Systems for Analytical Separations II: Systems Based on Electroosmotic Flow (EOF)	165
5.1	Introduction	165
5.2	CE on the Microchip Format	168
5.3	Modes and Theories of CE Microchips	170
5.4	Microfabrication Techniques	175
5.4.1	Microfabrication of Glass CE Microchips	177
5.4.2	Microfabrication of Polymer CE Microchips	178

5.5	Basic Fluidic Manipulation/Motivation: Electrokinetic Injection and Separation Protocols	181
5.6	Electrochromatography in Microchip Format: Designs and Applications	184
5.7	Comparison of Hydrodynamic and Electroosmotic Flow-driven Miniaturized Systems	187
5.8	Analytical Applications	189
5.8.1	DNA Analysis	190
5.8.2	Protein Analysis	196
5.8.3	Small-molecule Analysis	200
5.9	Outlook	206
	References	208
6	Detection in Miniaturized Analytical Systems	213
6.1	Introduction	213
6.2	Laser-induced Fluorescence (LIF) Detection	214
6.2.1	Lamp-based Fluorescence Detection	218
6.2.2	Fluorescence Excited by Light-emitting Diodes	218
6.2.3	(Electro)chemiluminescence Detection	219
6.3	Electrochemical Detection (ED)	220
6.3.1	End-channel Detection	221
6.3.2	In-channel Detection	224
6.3.3	Off-channel Detection	225
6.3.4	Electrode Materials	228
6.3.5	Modes of Detection	230
6.4	Microfluidics–MS Interfacing	234
6.4.1	Microfluidics-based Electrospray Ionization (ESI) Sources	234
6.4.2	Microfluidics–MALDI-MS Interfacing	243
6.5	Unconventional Detection Methods	248
6.5.1	Absorbance Detection	248
6.5.2	Surface Plasmon Resonance (SPR) Detection	249
6.5.3	Thermal Lens Detection	251
6.5.4	Other Detection Methods	253
6.6	Outlook	254
	References	254
7	Miniaturization of the Entire Analytical Process I: Micro(nano)sensors	263
7.1	Introduction	263
7.2	Evolution of Sensors with Nanotechnology	264

7.3	Micro(nano)sensors	266
7.3.1	Optical Sensors	266
7.3.2	Electrochemical Sensors	270
7.3.3	Magnetic Sensors	272
7.3.4	Mass Sensors	273
7.4	Nanoprobes for <i>In Vivo</i> Bioanalysis	275
	References	278
8	Miniaturization of the Entire Analytical Process II: Micro Total Analysis Systems (μTAS)	281
8.1	μ TAS, Microfluidics and Lab-on-a-Chip: Concepts and Terminology	281
8.2	Basic Concepts of Microfluidics: The Design of Analytical Microsystems	283
8.2.1	Types of Transport	284
8.2.2	Laminar and Turbulent Flows	285
8.2.3	The Design of Microfluidic Systems	286
8.3	The Basics of Downscaling in Microsystems	290
8.4	Microfluidic Platforms: Types, Principles and Classification	293
8.4.1	Capillary-driven Test Strips	294
8.4.2	Microfluidic Large-scale Integration (LSI)	296
8.4.3	Centrifugal Microfluidics	297
8.4.4	Electrokinetic Platforms	299
8.4.5	Droplet-based Microfluidic Platforms	300
8.5	Microfluidic Devices for Analytical Lab-on-a-Chip Applications	302
8.5.1	DNA Analysis Integrated on Microfluidic Devices	305
8.5.2	Real Clinical Sample Analysis on Microfluidic Devices	310
8.5.3	Real Environmental and Related Sample Analysis on Microfluidic Devices	323
8.5.4	Real Food Sample Analysis on Microfluidic Devices	328
8.6	Outlook	338
	References	339
9	Portability of Miniaturized Analytical Systems	345
9.1	Introduction	345
9.2	Portable Gas Analysers	346
9.3	Portable Electrochemical Analysers	351
9.4	Portable Optical Analysers	351
9.5	Portable Lab-on-a-Chip Analysers	352
	References	354

10 Analytical Performance of Miniaturized Analytical Systems	357
10.1 Introduction	357
10.2 Quality Control in Miniaturized Systems	358
10.3 Validation of Microsystems	360
10.4 Qualification of Microsystems	361
10.5 Robustness of Microsystems	362
Further Reading	363
Index	365

Preface

Without question, the main drivers of modern analytical techniques are the simplification of procedures and the improvement of measurement quality. To reach these goals, modern analytical techniques try to reach lower detection limits, improve selectivity and sensitivity, and achieve faster analysis time, higher throughput and less expensive analysis systems with ever-decreasing sample volumes. These very ambitious goals are exacerbated by the need to reduce the overall size of the device and the instrumentation. These items are termed ‘analytical miniaturization concepts’.

The miniaturization of analytical systems is a rapidly growing area. It is associated with performing the analytical process on a small scale (sometimes, a very small scale). Different terms have been used to represent this idea: ‘miniaturized analytical systems’, ‘analytical micro(nano)systems’ and, more ambitious, ‘micro total analysis systems’ (μ TAS), also called ‘lab-on-a-chip’.

Originally, microsystems came from the need to perform online control and monitoring of industrial processes. This mainly resulted in the development of (bio)chemical sensors. However, after this initial phase, society’s needs increased and sensors became insufficient to respond to new specific problems, as they suffer from poor selectivity for many applications. As a consequence, the concept of the total analysis system appeared in analytical chemistry. The idea of such a system is to integrate all the steps of analytical process (sampling, sample treatment, separation of analytes and detection) in the same device. More recently, the interest in portable instruments, allowing field tests to be carried out with ease, has increased the practical usefulness of miniaturized analytical systems. Between sensors and lab-on-a-chip devices, a wide range of microsystems, which can affect either the entire analytical process or only a part of it, have been described.

This exciting challenge has guided our effort to offer a book with a general approach to miniaturization in analytical chemistry, including the principles, designs and applications of miniaturized systems. Through ten chapters, the different issues characterizing such systems are critically discussed.

The first two chapters include the basic concepts behinds miniaturization in analytical chemistry, as well as the mechanical and electronic tools needed for designing and fabricating these systems. It is very important to give an integrated classification of the systems and to define the different terms associated with miniaturization, in order to provide a systematic view of both the different levels of miniaturization and the main objectives of the downsizing developments.

Chapters 3 to 6 represent the solid core of the book. Taking as their basis the analytical process, these chapters deal with: the miniaturization of sample treatment (including the consequent automation), with sections devoted to the problems associated with sample introduction in micro(nano)systems; miniaturized systems for analyte separation, divided into two chapters according to the forces involved in moving the flow; and detection in micro-size environments. Through these chapters, practical aspects such as the representativeness of the portion of sample analysed, the analytical potential of micro- and nanochromatography, the advantages of miniaturized capillary electrophoresis with special attention to microchip format, and both well-established and new approaches to detection in miniaturized systems, are comprehensively studied.

Chapters 7 and 8 deal with the miniaturization of the entire process: from the sample introduction to the generation of the corresponding analytical results. Thus, when possible, Chapter 7 considers the use of sensors and biosensors in an online approach, or as micro(nano)probes, very useful for *in vivo* analyses. The objective of this chapter is not to give a wide report on the sensor field (different books address this topic), but to cover the integration of micro(nano)sensors in miniaturized technology. Chapter 8 covers the miniaturization of the entire analytical process under the philosophy of the μ TAS approach. From a practical point of view, it is clear that μ TAS entails many different challenges, but this trend in analytical chemistry is very attractive and will be the reality of future analytical work (both inside and outside the lab). Microfluidic concepts and lab-on-a-chip systems will make up the content of this chapter, in which a rich discussion of real samples is offered.

The last part of the book (Chapters 9 and 10) deals with two aspects directly connected to the usefulness of miniaturized analytical systems: the design of portable miniaturized systems (very interesting for the performance of field tests) and how to assure the practical reliability of micro(nano)systems (quality control tests, performance and validation activities, as well as the robustness of the miniaturized depicted systems). The ruggedness of micro(nano)systems is briefly discussed and related to the tools used for the design and fabrication described in the first chapters of the book.

Finally, the authors wish to thank the broad group of researchers who have contributed to analytical miniaturization developments over the past two decades. They have been cited throughout this book, where their works have been selected, studied and strategically related between them in order to give the reader a novel textbook on analytical miniaturization as a whole. The authors hope that the reader enjoys this book and finds it useful in their own teaching and research developments.

1

Miniaturization in Analytical Chemistry

1.1 Introduction

Miniaturization is rapidly growing, with novel ideas emerging in recent years. Like other fields, analytical systems have been affected by this new technology. Concretely, the capacity to carry out laboratory operations on a small scale using miniaturized devices is very appealing. *Micro total analysis systems* (μ TAS), also called *lab-on-a-chip*, have renewed interest in scaling laws in the last 10–15 years. A small scale reduces the time required to synthesize and analyze a product, as greater control of molecular interactions is achieved at the microscale level. In addition, reagent cost and the amount of chemical waste can be very much reduced.

Now, at the beginning of the twenty-first century, it is clear that the lab-on-a-chip approach is starting to be considered as a potential analytical tool in many application fields. Nevertheless, additional efforts must be addressed to two main points: (i) the laws at nanometre scale must be established, as basic physical and chemical fundamentals cannot be applied; and (ii) more applications demonstrating the real use of these systems must be developed, particularly in the area of complex samples analysis. There is no doubt that miniaturized chemical analysis systems have a tremendous potential. For instance, it is foreseeable that such devices will allow the study and analysis of complex cellular processes, facilitate the development of new diagnostic abilities that could revolutionize medicine, and have applications in environmental monitoring, food analysis and industry.

Some miniaturized analytical systems, such as capillary gas chromatography, microliquid chromatography and microcapillary electrophoresis – which can be

The book describes a general vision of the miniaturization of the analytical systems, including their principles, designs and applications. Through ten chapters the different aspects characterizing the miniaturized systems are developed. Thus, the two first chapters include the basic concepts behind miniaturization in analytical chemistry, as well as the mechanical and electronic tools needed for designing and fabricating miniaturized analytical systems. Chapters 3 to 6 CAD for Miniaturized Electronics and Sensors Computer-aided design of nanoscale devices and sensors is a cost effective way to infuse emerging nanoelectronics technologies in on-board information processing. + Read More. Carbon Nanotube Field Emitters We are developing Carbon Nanotube (CNT) field emitters to improve their efficiency and durability. Current densities of $\sim 1\text{A}/\text{cm}^2$ have been measured from these emitters. + Read More. Nanoengineered Heat Sink Materials Advanced thermal materials will radically improve the performance of devices and instruments such as high-performance computers and The book describes a general vision of the miniaturization of the analytical systems, including their principles, designs and applications. Through ten chapters the different aspects characterizing the miniaturized systems are developed. Thus, the two first chapters include the basic concepts behind miniaturization in analytical chemistry, as well as the mechanical and electronic tools needed for designing and fabricating miniaturized analytical systems. 1.3 Evolution in the Field of Analytical Miniaturization. 1.4 Classification of Miniaturized Analytical Systems and Definition of Terms. 1.5 Theory of Miniaturization. 1.6 Features of Miniaturized Analytical Systems. 1.7 Incidences of Miniaturization in the Analytical Process. 1.8 Outlook. References.