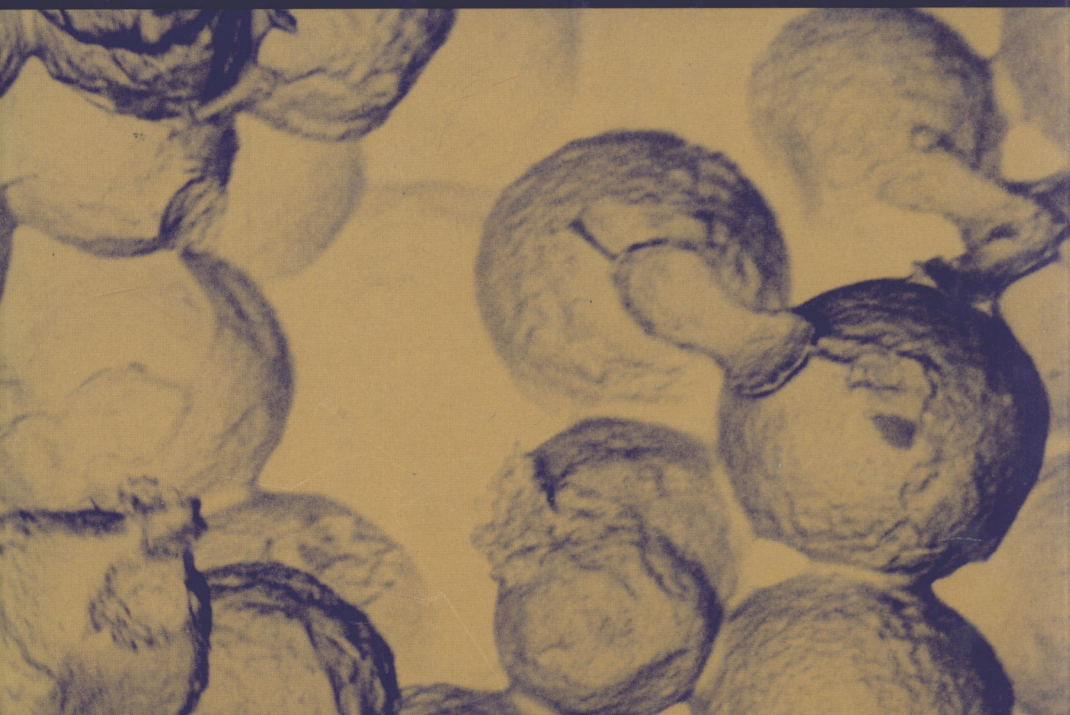


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Sample Preparation Techniques in Analytical Chemistry

Edited by Somenath Mitra



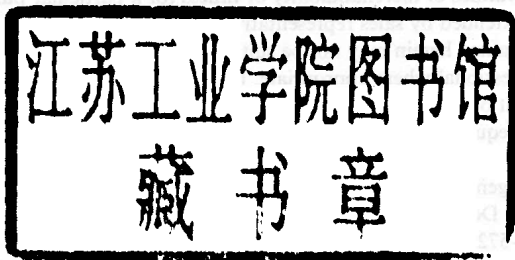
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Sample Preparation Techniques in Analytical Chemistry

Edited by

SOMENATH MITRA

Department of Chemistry and Environmental Science
New Jersey Institute of Technology



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To the hands in the laboratory
 and
 the heads seeking information

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PREFACE

There has been unprecedented growth in measurement techniques over the last few decades. Instrumentation, such as chromatography, spectroscopy and microscopy, as well as sensors and microdevices, have undergone phenomenal developments. Despite the sophisticated arsenal of analytical tools, complete noninvasive measurements are still not possible in most cases. More often than not, one or more pretreatment steps are necessary. These are referred to as *sample preparation*, whose goal is enrichment, cleanup, and signal enhancement. Sample preparation is often the bottleneck in a measurement process, as they tend to be slow and labor-intensive. Despite this reality, it did not receive much attention until quite recently. However, the last two decades have seen rapid evolution and an explosive growth of this industry. This was particularly driven by the needs of the environmental and the pharmaceutical industries, which analyze large number of samples requiring significant efforts in sample preparation.

Sample preparation is important in all aspects of chemical, biological, materials, and surface analysis. Notable among recent developments are faster, greener extraction methods and microextraction techniques. Specialized sample preparations, such as self-assembly of analytes on nanoparticles for surface enhancement, have also evolved. Developments in high-throughput workstations for faster preparation–analysis of a large number of samples are impressive. These use 96-well plates (moving toward 384 wells) and robotics to process hundreds of samples per day, and have revolutionized research in the pharmaceutical industry. Advanced microfabrication techniques have resulted in the development of miniaturized chemical analysis systems that include microscale sample preparation on a chip. Considering all these, sample preparation has evolved to be a separate discipline within the analytical/measurement sciences.

The objective of this book is to provide an overview of a variety of sample preparation techniques and to bring the diverse methods under a common banner. Knowing fully well that it is impossible to cover all aspects in a single text, this book attempts to cover some of the more important and widely used techniques. The first chapter outlines the fundamental issues relating to sample preparation and the associated quality control. The

remainder of the book is divided into three sections. In the first we describe various extraction and enrichment approaches. Fundamentals of extraction, along with specific details on the preparation of organic and metal analytes, are presented. Classical methods such as Soxhlett and liquid-liquid extraction are described, along with recent developments in widely accepted methods such as SPE, SPME, stir-bar microextraction, microwave extraction, supercritical extraction, accelerated solvent extraction, purge and trap, headspace, and membrane extraction.

The second section is dedicated to the preparation for nucleic acid analysis. Specific examples of DNA and RNA analyses are presented, along with the description of techniques used in these procedures. Sections on high-throughput workstations and microfabricated devices are included. The third section deals with sample preparation techniques used in microscopy, spectroscopy, and surface-enhanced Raman.

The book is intended to be a reference book for scientists who use sample preparation in the chemical, biological, pharmaceutical, environmental, and material sciences. The other objective is to serve as a text for advanced undergraduate and graduate students.

I am grateful to the New Jersey Institute of Technology for granting me a sabbatical leave to compile this book. My sincere thanks to my graduate students Dawen Kou, Roman Brukh, and Mahesh Karwa, who got going when the going got tough; each contributed to one or more chapters.

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SOMENATH MITRA

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