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# Book review

*Chromatographic Analysis of Pharmaceuticals* (Second Edition, Revised and Expanded Edition, Chromatographic Science Series, Vol. 74), edited by John A. Adamovics; Contributors: James C. Eschbach, David L. Farb, Nirdosh K. Jagota, Shelley R. Rabel, James T. Stewart, John F. Stobaugh. Publisher: Marcel Dekker, Inc., New York, NY, USA. ISBN 0-8247-9776-0; X+527 pp; US$ 165.00

Following the success of the first edition published in 1990, the second edition of Chromatographic Analysis of Pharmaceuticals is revised and expanded from classical to modern analytical methods worldwide in use in pharmaceutics and biotechnology. Understanding and application of chromatographic methods have increased rapidly, as updated material as well as current literature references are provided in this edition.

The book's purpose is to provide a concise, comprehensive and practical overview of analysis of bulk and formulated drug products, describing basic instrumentation, theories and method development of consolidated as well as new analytical techniques. Most major analytical separation techniques used in pharmaceutics are covered, including liquid chromatography, gas chromatography, thin-layer chromatography, capillary electrophoresis and supercritical fluid chromatography, as well as robotics and sample preparation.

The editor, John A. Adamovics, opens with a useful presentation of regulatory considerations, updated from U.S. Pharmacopoeia 23, Chinese, British and European Pharmacopoeias. Variables to consider in developing an accurate and rugged chromatographic method before starting an analysis are outlined with emphasis on specificity, accuracy, precision, sensitivity and stability.

In the second, particularly instructive chapter (by the editor), the topic is an exhaustive discussion on advantages and disadvantages of various manual and automated sample pretreatment procedures for several pharmaceutical formulations (i.e., parenterals, tablets, liquid dosage forms), which can strongly affect analytical performances. Throughout the text is an helpful, extensive table showing a compilation of published procedures of column liquid-solid extraction of pharmaceutical formulations, that provides some practical hints and suggestion concerning method development.

In the third chapter, by John A. Adamovics and James C. Eschbach, paper and thin-layer chromatography (TLC) are presented. For many years TLC has held a prominent position in the various fields of drug analysis, including the quality control of pharmaceuticals and forensic applications. Notwithstanding the decline in the use of TLC observed with the development of high-performance liquid chromatography (HPLC), chromatographers still show some interest in this analytical method to be used alongside HPLC, gas chromatography (GC) and, more recently, capillary electrophoresis (CE). The revival of TLC is very much due to new instrumentation and automation, which allow higher reproducibility together with the traditional advantages such as rapidity, versatile detection possibilities and cost. Instrumental TLC has, so far, been focused not only in qualitative applications, but also for quantitative work using commercial available densitometers. The


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authors briefly and clearly discuss TLC developments and detection procedures required to enhance selectivity and detection limits.

A longer fourth chapter (by John A. Adamovics and James C. Eschbach) on chromatographic analyses of pharmaceutical compounds is dedicated to gas chromatography (GC), the oldest of the instrumental analytical techniques, developed from the early 1950s to the present with many concurrent innovations. The authors concisely introduce the gas chromatographic stationary phases, one of the most critical topics of this technique, pointing out the important features of each with reference to experimental results. This is followed by a detailed presentation of the GC hardware, including injectors, capillary columns and detectors. Various analytically crucial factors such as injection system, column size and temperature are considered with respect to maximizing analytical performance. A subsection highlights most of the detectors available and outlines useful and interesting applications for different classes of compounds. In the chapter's conclusion, several GC applications (i.e. separation of enantiomers, excipients, preservatives and pharmaceuticals) and headspace methods for the analysis of residuals (i.e. ethylene oxide, ethanol, oxygen) prove most valuable for analysts working with GC. More than 200 references, at the end, give the reader the possibility of getting more detailed information on GC methods.

In the following chapter (by John A. Adamovics and David L. Farb) the aim of the authors is to present a comprehensive and practical overview of high-performance liquid chromatography (HPLC), the most popular chromatographic technique in the pharmaceutical laboratories. From the late 1950s to the 1990s, analysts have progressively refined and improved liquid chromatography instrumentation: the most significant advances have occurred in the column sorbent technology, detection and automation. The strategy adopted by the authors is to briefly survey all the necessary information for approaching current HPLC and coupled techniques (for example HPLC–mass spectrometry). In fact, the chapter avoids to describe exotic methods, but focuses on method development for a broad range of interesting compounds, including enantiomers, biomolecules, drugs containing basic functionality and neutral drugs.

Chromatograms and a number of helpful tables supplement explanations in the text (for example, the table for chiral derivatization reagents for separation of enantiomers as diastereomers). As a reference source, the paper is really updated and the reader who is developing methods, is directed to many literature references (about 350) for more detailed information.

In this book, a new chapter (the sixth, by Shelley R. Rabel and John F. Stobaugh), lacking in the previous edition, is devoted to capillary electrophoresis (CE), a recent but rapidly expanding technique in analytical chemistry. Since the commercial introduction in the late '80s, the use of CE has spread across a number of applications for all analyte types (such as metal ions, inorganic anions, nucleic acids, amino acids, proteins, peptides etc.). In many pharmaceutical laboratories CE is now accepted as an orthogonal and, therefore, complementary method to traditional instrumental separation techniques, such as HPLC and GC. CE has been utilized in the quantitation of drug-related impurities, stability studies, chiral analysis and formulation analysis. This paper introduces the knowledge behind the technique, by a satisfactory account of the fundamental electrophoretic/electrokinetic concepts of different modes of CE separations; a second section covers the basic instrumental aspects of CE. The presentation of detection modes, however, is very concise, as the last section of the chapter which surveys rapidly some approaches to method development with only few examples on proteins and small molecules.

In the seventh chapter, the authors James T. Stewart and Nirdosh K. Jagota present supercritical fluid chromatography (SFC), a column chromatographic technique in which a supercritical fluid (such as $CO_2$) is used as the mobile phase. Recent technological advances in instrumentation now make it possible to exploit the high speed, efficiency and resolution of this technique, that will emerge as an alternative analytical method to HPLC, for example in chiral separation and purity analysis. A first part of the chapter briefly describes the SFC apparatuses (sample introduction, pumps, oven, columns, mobile phases and detectors), then in 25 pages the reader

can find several published applications of SFC to bulk and formulated pharmaceuticals, including benzodiazepine, non-steroidal anti-inflammatory drugs, polar compounds, prostaglandins, antibiotics, fat-soluble vitamins.

The second half of the book (about 250 pages) is meant to complement the preceding chapters; it contains a wide, comprehensive tabulation of supplementary up-to-date references (as many as 1,167!), covering analytical procedures (mode of analysis, sample pretreatment, sorbent, mobile phase, type of detection and comments) for detecting and characterizing drug substances and related impurities not discussed previously, but considered relevant to the scope of the book.

In conclusion, this book edited by John A. Adamovics offers a concise and well-organized overview of modern chromatography in pharmaceutics and an interesting discussion of newer analytical technologies and methodological developments, together with an impressive literature on these subjects.

The book should be recommended to any chemist and biochemist working in the different pharmaceutical fields to update his knowledge, but looks particularly useful for Ph.D. students approaching the challenging world of modern pharmaceutical analysis.

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