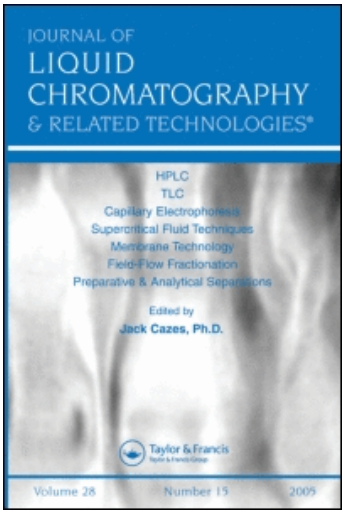

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A Review of: Ion Exchange and Solvent Extraction

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THE BOOK CORNER

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION, by M. Swartz and I. S. Krull, Marcel Dekker, Inc., New York, 1997, 96 pages. Price: $35.00

Analytical Method Development and Validation is a concise guide which deals mainly with optimization and validation of HPLC-based analytical methods. Although the title of the book does not specify that it is mostly based on HPLC methodology, this guide gives enough information that can be useful when other analytical methods are required. This 92 page booklet gives:

* step-by-step procedures for methods development and optimization, including guidelines for the future development of analytical techniques, as well as discussing instrument qualification and system suitability;

* validation protocols for new and improved analytical methods, aiding in the creation of validated procedures and revealing which steps can be employed today;

* regulatory agency guidelines, assessing methods validation criteria stipulated by the USP and the FDA, and undergoing final approval by the ICH;

* examples of complete methods development and validation, illustrating what a final validated analytical method contains and how it is presented; and

* pathways by which acceptable, validated methods of analysis are built, providing directions for the development of assays.

The book is divided into an Introduction, which discusses the validation process of software, hardware, the method, and system suitability. Chapter 2 deals with method development and optimization. The authors ask the question, "How does the analyst really know that the new method has been adequately optimized and is ready for actual sample applications?" They

answer the question by the following: "To verify that the optimized method satisfies the goals of unequivocal analyte identification and quantitation, improved quantitative accuracy and precision, faster sample turnaround time, absence of interference, and automation." In the past, we defined HPLC optimization as the set of conditions which give baseline resolution of the solutes of interest, in the minimum amount of time, with maximum detection and ease. Chapter 3 is a discussion of method validation, a requirement of the Food & Drug Administration, an important part of a pharmaceutical Quality Control Laboratory. The discussion centers around the USP eight-step method validation and the ICH method validation. System suitability (Chapter 4), method validation protocol (Chapter 5), and method transfer (Chapter 6) are very brief. The book ends with Chapter 7, which is a summary and conclusions. The book is well written, easy to read, and will be useful to analytical chemists, especially those in the pharmaceutical industry.

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STATIC HEADSPACE - GAS CHROMATOGRAPHY THEORY AND PRACTICE, by B. Kolb and L.S. Ettre, Wiley-VCH, New York, 1997, 298 pages. Price: $79.95

This book deals with an important topic, headspace analysis, which, in essence, is a clean-up procedure employing an inert gas for the extraction of highly volatile compounds. A gas extract is ideally suited for gas chromatography, and this combination is called headspace analysis by gas chromatography (HS-GC). Gas extraction techniques, the authors tell us, can be carried out in several variants: as a single step (static headspace) or by

stepwise repeating of the extraction (multiple headspace extraction) and, also, by stripping the volatiles (dynamic headspace) by a continuous flow of an inert purge gas. All these gas extraction techniques are called headspace techniques for historical reasons (the name "headspace" was originally given to the gas content of the bulge that forms at the top of a can of food whose composition had to be analyzed).

HS-GC is simple, can be automated, is sensitive and quantitative. I agree with the authors that the simplicity of static HS-GC is unsurpassed by any other cleanup techniques: the sample (either a gas, a liquid, or a solid) is filled into the headspace vial, which is closed immediately and remains closed until an aliquot has been withdrawn from the closed vial and transferred directly to the gas chromatographic system, thus guaranteeing sample integrity. This simplicity enabled the early automation of the whole procedure. Also, as far as sensitivity and the possibilities for quantitative analysis are concerned, one would, at first, favor dynamic HS-GC. Its inherent purpose is to perform an exhaustive extraction, contrary to static HS-GC and, therefore, the composition of the resulting gas extract is often considered to be the same as that of the original sample. As this book shows, however, the modern techniques of cryogenic focusing also allows the sensitivity range to be extended to determine concentrations down to the level of parts per trillion, or even parts per quadrillion (ppt, $1{:}10^{-12}$; ppq, $1{:}10^{-15}$).

The authors also state that, although this book emphasizes techniques, methods, and procedures, rather than applications, we have selected the many practical examples to cover, at least, the most important applications of static HS-GC in environmental, polymer, and food analysis, and in some other interesting fields of application. Static Headspace-Gas Chromatography provides a thorough and current treatment available on this technique. The book covers the theory of headspace sampling as related to selection of the operational parameters, describes quantitative calibration techniques, and presents detailed methods, all adapted for automation. The book is well written, clear, and is a valuable reference.

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4. **Sample Handling in HS-GC**, (117).

5. **Headspace Methods for Quantitative Analysis**, (149).

6. **Method Development in HS-GC**, (211).

7. **Nonequilibrium Static Headspace Analysis**, (227).

8. **Qualitative Analysis by HS-GC**, (235).

9. **Special Measurements**, (251).

ION EXCHANGE AND SOLVENT EXTRACTION, Volume 13, Edited by J. A. Marinsky and Y. Marcus, Marcel Dekker, Inc., New York, 1997, 416 pages. Price: $195.00

This volume of Ion Exchange and Solvent Extraction deals mainly with advances in solvent extraction. Six chapters which present novel approaches to solvent extraction make up the book. Chapter 1 shows how mixed extractants such as mixed hydrophobic acids and bases can be used to extract divalent metal salts.

Chapter 2 deals with the extraction of acids by acid-base-coupled extractants. When weak organic acids are to be extracted or when several strong and weak acids are to be separated, the advantages presented by the use of the coupled extractants are displayed. The mechanisms involved in the processes, involving ion exchange, ion pairing, and hydrogen bonding phenomena, depending on the conditions and reagents used, are demonstrated. Possible industrial applications are illustrated with several cases, including the removal of acids from waste streams, the production of potassium nitrate, and the purification of acids.

Host-guest associates, in a "hot" subject in many fields of chemistry; it turns out to also have an impact on solvent extraction. Organic compounds, including bio-organics, can be extracted as guests by means of suitable hosts, and the host-guest associates can be transported in membranes.

Chapter 3 explores the possibilities opened by these processes. The preferred hosts are macrocyclic compounds, such as crown ethers, aza-crowns, and many other kinds of molecules.

A completely different aspect is covered in Chapter 4. Although not many laboratories have studied aqueous biphasic extraction systems, there are many advantages when such systems are employed, including the avoidance of volatile organic compounds. The extractants are made up from polyethylene glycols, which are made immiscible with aqueous phases having fairly high concentrations of electrolytes. Since the major component in each phase is water, the hydrophilicity of the solutes to be separated plays the most important role.

Another interesting procedure is to impregnate a porous polymeric resin with chemically active extractants. The methods of resin impregnation and the mechanisms through which this is achieved are discussed, and a physicochemical characterization of the resulting solvent-impregnated resins (SIRs) is provided in Chapter 5.

Finally, Chapter 6 deals with the extraction of the alkali metal cations. These cations do not form extractable complexes with most of the useful extractants. However, they can be extracted by certain reagents, such as crown ethers, if a suitable diluent takes care of the co-extracted anion or the alkali metal cation is exchanged for another cation.

The book is written by authorities in their areas, and is well illustrated and presented.

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6. **Principles of Solvent Extraction of Alkali Metal Ions: Understanding Factors Leading to Cesium Selectivity in Extraction by Solvation**, B. A. Moyer and Y. Sun, (295).

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