



## Book reviews

*Chromatographic Characterization of Polymers*, Advances in Chemistry Series 247, edited by Theodore Provder, Howard G. Barth and Marek W. Urban, American Chemical Society, 1995, 294 pp., \$124.95, ISBN 0-8412-3132X.

This volume covers some of the more significant advances in areas related to the characterization of polymeric materials, and is especially focused on hyphenated chromatographic separation methods. Topics include size-exclusion chromatography, liquid chromatography, and a variety of field-flow fractionation techniques. The book is divided into three main sections which cover general considerations and background, topics related to light scattering and viscometry, and the analysis of compositional heterogeneity. The various chapters tend to be somewhat long and to be heavily weighted with research results from the laboratories of the contributing authors.

The first section contains four chapters which outline important general considerations associated with the chromatographic characterization of polymers. Discussed in this section is the state of hyphenated polymer separation techniques (H.G. Barth), where particular emphasis was placed on the use of hyphenated multidimensional separation and detection techniques. The analysis by liquid chromatography of polymeric solutes at the limit of their solubility was examined (D.J. Hunkeler, M. Janco, V.V. Guryanova and D. Berek), and the advantages and disadvantages of this approach provided. Various experimental configurations for isoperichoric focusing field-flow fractionation and thin-layer isoperichoric focusing were presented (J. Janca), and uses for the new

methodology suggested. The first section ends with a paper describing the interfacing of a size-exclusion chromatograph of a mass spectrometer using electrospray ionization (W.J. Simonsick, Jr., and L. Prokai).

The second section covers the use of light scattering and viscometry molecular size-sensitive detectors, and issues pertaining to multidetector calibration. The practice of using multiple detectors with size-exclusion chromatography is critically evaluated (C. Jackson and H.G. Barth), and the balance between instrumental complexity and amount of information available is discussed. A computer simulation of size-exclusion chromatography-viscometry-light scattering is described (C. Jackson and W.W. Yau), and data presented for polymers with a Florey-Schulz molecular weight distribution. The next contribution deals with size-exclusion chromatography and a method for eliminating the need to estimate empirical lag times (K.G. Suddaby, R.A. Sanayei, K.F. O'Driscoll, and A. Rudin). A study is described where thermal field-flow fractionation was used to characterize ultrahigh molecular weights and sizes using light scattering (S. Lee, and O.-S. Kwon). In the next chapter, results are presented of a largely theoretical analysis of molecular characterization, which stimulated a unified refractive index-light scattering detector (R. Frank, L. Frank and, N.C. Ford). A method is then described for the analysis of data from a size-exclusion chromatography detector that measures elastic light-scattering intensities at two angles (T.H. Mourey and H. Coll). The solubility problems associated with the characterization of high molecular weight natural

polymers have been addressed through the use of a nondegrading solvent consisting of dimethylacetamide and lithium chloride (J.D. Timpa), and subsequent characterization by size-exclusion chromatography with viscometry and refractive index detectors. The utility of size-exclusion chromatography with multi-angle light scattering, viscosity, and refractive index detectors as a means to characterize pullulans and dextrans was studied (W.S. Bahary, M.P. Hogan, M. Jilani, and M.P. Aronson). The second section ends with a presentation dealing with the characterization of star-branched model copolymers using refractive index and viscometric detection with an on-line light-scattering detector (J. Lescq, M. Millequant, M. Patin, and P. Teyssie).

The third and final section contains topics concerned with the analysis of compositional heterogeneity in copolymers and blends. A method was presented for obtaining the molecular weight and composition of copolymers using thermal field-flow fractionation and viscometry (M.E. Schimpf). The use of coupled techniques with chromatographic columns and multidetectors is discussed, primarily as related to the practice of size-exclusion chromatography (J.V. Dawkins). The advantages of using various nonexclusion chromatography methods in conjunction with size-exclusion chromatography to provide accurate compositional information on copolymers was presented (S. Mori). Methodology for the totally automated characterization of complex copolymers and blends by two-dimensional liquid chromatography-size-exclusion chromatography has been developed (P. Kilz, R.-P. Kruger, H. Much, and G. Schulz), and applied to the analysis of a 16-component mixture of a star block copolymer. The problems caused in size-exclusion chromatography by the superimposition of chemical composition distributions onto molecular weight distributions were addressed through the use of ultraviolet absorption and differential refractive index detectors (E. Meehan and S. O'Donohue). An interface between gel permeation chromatography and infrared spectroscopy has been developed, and applied to the analysis of copolymers (J.N. Willis and L. Wheeler). The final contribution to this section discusses the use

of a solvent-evaporative interface to isolate the fractions obtained after the performance of size-exclusion chromatography onto a surface suitable for infrared spectral analysis (P.C. Cheung, S.T. Balke, and T.C. Schunk).

This volume presents the results of a number of investigations, where advances in the field continue to extend the arsenal of analysis methods available for the characterization of polymers. Viewed within this context, the book fulfils its mission to encourage and catalyze additional activity and method development in hyphenated chromatographic separation techniques for polymer characterization. However, research papers are not review articles, and those seeking such presentations on the main characterization methods, or on the hyphenated hybrids, will need to search elsewhere.

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*Validation Compliance Biannual, 1996-1997*, The International Validation Forum, Marcel Dekker, 1996, 848 pp., \$195.00, ISBN 0-8247-9746-9.

Validation has become one of the most important issues within the pharmaceutical industry, and the range of associated activities increases practically with each new issue of the Federal Register. This present book seeks to encapsulate the essential elements of this ever-growing field into a single volume which provides a concise summary of current expectations. The primary focus is that of the United States Food and Drug Administration, although some information is provided regarding other venues. The editors have recognized that validation is a developing and changing creation, and thus have chosen to update their series on a periodic basis. As indicated by the title, the latest validation compliance volume seeks to remain current for the 1996–1997 time period.

Each section of the volume follows the same general pattern, which facilitates updating of the work from previous editions. The editors begin with a very short overview of a defined subject matter, and follow with a voluminous reproduc-