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Book Reviews *

Preparative Chromatography of Fine Chemicals and Pharmaceutical Agents. Edited by Henner Schmidt-Traub. Wiley-VCH: Weinheim. 2005. 458 pp. £90.00. ISBN 3-527-30643-9.

Over the past decade or so, preparative (process-scale) chromatography has evolved from what many process chemists considered a costly last resort into a powerful and cost-effective tool in the production of active pharmaceutical ingredients and organic chemical intermediates.

This book provides a timely and detailed review of the state of the art, aimed at both chemists and engineers. The contributing authors come from both academic and industrial backgrounds, emanating from Merck KGaA, Johnson & Johnson, BASF, BP, Bayer CropScience AG, Bayer Technology Services GmbH, University of Dortmund, and the Ohm Fachhochschule Nurnberg.

The introductory chapter provides a short history of liquid chromatography and provides context for the reader, while Chapter 2 presents the basic principles of chromatography. Chapters 3 and 4 focus on columns and stationary phases and the selection of chromatographic systems. Chapter 5 "Process Concepts" is a particularly interesting section, covering a number of different chromatographic approaches in addition to the now well-established simulated moving bed (SMB) methodology.

Chapters 6 and 7 provide in-depth accounts of the role of computational modelling in the design and optimisation of chromatographic separation processes, while Chapter 8 describes the less familiar concept of chromatographic reactors. The final chapter, perhaps a little out of sequence, is devoted to the advanced control of simulated moving bed processes.

The layout and style of the book maintain the attractive standards we have come to expect from Wiley-VCH, and the division of each chapter into bite-sized chunks makes for relatively easy reading, albeit resulting in a contents listing extending to eight pages.

Although the authors emphasise the importance of the partnership between chemistry and engineering, the emphasis on chemistry is relatively light, which is perhaps to be expected in a field in which engineering issues are a major consideration. As a result, the book will appeal more to specialists. Nevertheless, generalists will find a number of the chapters to be of interest and value. A notable shortcoming of this book is the glaring omission of any discussion relating to issues of solvent evaporation and recovery (these terms cannot even be found in the index). Cost issues are addressed in a theoretical modelling sense, but a discussion of absolute costs is also absent.

In conclusion, this is a comprehensive reference text, which should find its way into the libraries of all companies who are serious about process-scale preparative chromatography, whether internally or via outsource contracts.

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Green Separation Processes: Fundamentals and Applications. Edited by Carlos A. M. Afonso and Joao G. Crespo. Wiley-VCH: Weinheim. 2005. 363 pp. £100.00. ISBN 3-527-30985-3

This book looks at green and sustainable chemistry from an unusual perspective, focussing, as the title indicates, on separation processes. The author listing is truly international, and there are no less than 37 contributors, from a variety of backgrounds, both industrial and academic (although the latter predominate).

The book is divided into four parts. Part 1, "Green Chemistry for Sustainable Development", is essentially an introduction, providing a technological and political context for what follows. Part 2, "New Synthetic Methodologies and the Demand for Adequate Separation Processes" has three subsections, covering Microreactor Technology for Organic Synthesis, Solventless Reactions (predominantly using microwaves), and Combinatorial Chemistry on Solid Phases (again mediated largely by microwaves). Part 3, "New Developments in Separation Processes", is the real meat of the book, covering topics ranging from distillation, chromatography, and fluid extraction (encompassing supercritical fluids, fluorinated solvents, and ionic liquids and superheated water) through to membrane processes, enantiomer separation by inclusion complexation, and nanostructures (dendrimers and functionalised magnetic particles). The book closes with a very brief Part 4 entitled "Concluding Remarks".

This is an eclectic collection of accounts, some engineering based, and others more chemically oriented. Chemists will find the example-packed chapter on "Green Enantiomeric Separations by Inclusion Complexation" to be of particular interest.

While the green aspects of each of the topics and technologies may be obvious, it must be said that the green credentials do not shine through particularly strongly in a number of the chapters. Certainly the book feels slightly lacking in critical discussion relating to the green agenda.

*Unsigned book reviews are by the Editor.

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There is undoubtedly merit in this collection, but it is unlikely to be at the top of the process chemist's shopping list.

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Organic Light-Emitting Devices: Synthesis, Properties and Applications. Edited by K. Mullen and U. Scherf. Wiley-VCH: Weinheim. 2006. xvii + 410 pp. £139. ISBN 3-527-31218-8.

Organic Light-Emitting Devices (OLEDs) are already making big inroads into markets such as mobile phones, digital cameras, and notebooks, although they have yet to penetrate the potentially vast market for flat screen TVs, which is dominated by liquid crystal displays. As a result, chemical companies are naturally interested in this new market, and collaborations with universities have increased, as OLEDs try to topple LCD technology for displays.

I, the editor, was interested to review this volume on OLEDs since my first R&D job with ICI was on discovery and development of organic electroluminescent materials for OLEDs, and I wished to see how far the technology has moved over the last 30 years.

The editors have brought together a wide range of experts in the field, including well-known names, such as Heeger and Friend, to review each area of the field. Of course, this is a multidisciplinary science and includes both inorganic and organic materials and is at the boundary of chemistry and physics.

For organic devices, many of the chapters cover polymer-based OLEDs, and there is one chapter on the synthesis of electroluminescent polymers.

Other chapters of interest to organic chemists are Dendrimer Light-Emitting Devices, Amorphous Molecular Materials, Cross Linkable Organic Semiconductors for OLEDs, Polymer Electrophosphorescence Devices, and Low-Threshold Organic Semiconductor Lasers.

The technology has moved on since the pioneering work of the Friend group at Cambridge, who published their results on light-emitting precursor poly(phenylene vinylene), PPV, in 1990. This book on OLEDs surveys the advances, particularly over the last 15 years, on new materials and properties and covers technological advances, problems to be solved, and large-scale fabrication issues as the focus moves towards commercialisation of OLEDs.

Early chapters cover the fundamentals of OLEDs and their development, whilst later chapters focus on the materials themselves, and their processing. The later chapters are looking to the future and where the new developments will come from.

The editors have done an excellent job on providing an up-to-date account of the current state of the art in this fast-moving field. The importance of industry−university collaborations in this area is apparent, and whilst there is much discussion of fundamentals, the focus is towards devices which can be commercialised and understanding the properties of new materials.

Highly recommended.

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Preparative Chromatography of Fine Chemicals and Pharmaceutical Agents. Edited by Henner Schmidt-Traub. Weinheim: Wiley-VCH. 2005. xxvii + 458 pp. $170. ISBN 3-527-30643-9 (hardcover).

The compilation volume from Wiley-VCH, *Preparative Chromatography of Fine Chemicals and Pharmaceutical Agents*, edited by Henner Schmidt-Traub (Universität Dortmund), offers nine chapters and more than 400 pages on this important and fast-changing topic. At a price of $145 (amazon.com), the volume is still potentially affordable to the individual scientist. While the volume possesses some of the unevenness and overlap that are almost inevitable with a compilation volume, it provides a useful introduction to the subject of preparative chromatography, with a particularly strong chemical engineering focus.

Preparative chromatography is a vast field that means different things to different people. Historically, preparative chromatography has been heavily used in the biochemistry and natural products areas, and is now increasingly being used in the small molecule areas of drug discovery, development, and even manufacturing. For a general process research chemist looking for a quick introduction to the field, this volume may feature a little too much engineering and not enough chemistry. Nevertheless, basic principals are soundly treated, and a generally useful overview of the field is presented.

A chapter dealing with fundamentals and general terminology is presented by E. Merck's Michael Schulte, who also authors or co-authors three additional chapters in the volume. An overview of columns, packings, and stationary phases is next presented by renowned expert, Klaus Unger. Only a brief overview of commercial materials is offered, which is unfortunate, given that most preparative chromatographers are simply buying and utilizing existing commercial equipment, columns, and stationary phases.

A compilation chapter next tackles the problem of selecting chromatographic systems, i.e. preparative chromatographic method selection and development. The subject is treated in something of a piecemeal fashion, but much useful information can be found. The thin-layer chromatography (TLC) approach to method development utilized by Jules Dingenen (Johnson & Johnson, Beerse, Belgium) is presented here in some detail. Few young chemists are trained in the art and science of TLC method development, and Dingenen does a good job of presenting a useful systematic approach to help guide method development for flash or HPLC purifications on silica, probably still the most frequent type of preparative chromatography carried out by process chemists. Process

concepts are covered in the next chapter, with an overview of various preparative chromatography components and separation systems.

The remainder of the volume is devoted largely to process modeling, simulation, and mathematical optimization, which are important topics for industrial-scale separations, but of only limited value to the process chemist looking to purify a few kilograms of material. An interesting chapter on chromatographic reactors presents the theory and equations relating to this emerging technology.

Overall, the coverage is a bit uneven, some of the material seems a little dated, and the focus of the book is not particularly well suited for introduction of the topic of preparative chromatography to the organic process research chemist. Nevertheless, the volume does compile some useful material and may be a good addition to the library of practicing preparative chromatographers.

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The Organic Chemistry of Sugars. Edited by D. E. Levy and P. Fügedi. CRC/Taylor and Francis: Boca Raton, Florida. 2006. xxiv + 880 pp. £115. ISBN 0-8247-5355-0.

Both of the editors have written or edited books related to carbohydrate chemistry, and together they have presented short courses on this topic for the American Chemical Society, so they are well-known experts in the field. They have now found the time—in addition to their day jobs—to not only edit this compendium but also to write some of the chapters.

After a most interesting historical overview and an introduction to carbohydrates, chapters on "Protective Group Strategies", "Glycosylation Methods", and "Oligosaccharide Synthesis" complete the first section of the book. The second part, subtitled "From Sugars to Sugar-Like Structures to Non-Sugars" is perhaps the most interesting section for the non-carbohydrate chemist and includes chapters on Functionalisation of Sugars, Strategies Towards C−Clycosides, From Sugars to Carbosugars, and Sugars with Heteroatoms.

Part III, entitled Sugars as Tools, Chiral Pool Starting Materials and Formidable Synthetic Targets is perhaps less of interest to the industrial chemist since chiral auxiliaries and natural product synthesis are relatively unimportant. However, two chapters in this section on "Sugars as Starting Materials in Synthesis" and "Asymmetric Synthesis of Monosaccharides" are well worth reading.

The final section, subtitled "Additional Topics" covers combinatorial carbohydrate chemistry, glycopeptides, and carbohydrate mimics in drug discovery.

Overall, this is an excellent compendium but could have included more on furanoses and nucleosides, and on the industrial chemistry of sugars.

A major disadvantage of many of the chapters is that the latest references are only to 2002, and some chapters have hardly any references after 2000. Occasional references to 2004 and 2005 appear in one chapter, but these look as if they have been added in the proofs. I hope the editors will consider updating this excellent work in a few years time.

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Statistical Design: Chemometrics. By R. E. Bruns, I. S. Scaramino, and B. De Barros Neto. Elsevier: Amsterdam. 2006. x + 412 pp. 130 Euro/£90/$145. ISBN 0-444-52181-X.

This is the latest title (No. 25) in Elsevier's exhaustive series of texts on Data Handling in Science and Technology and is a welcome addition to the growing choice of textbooks for newcomers to chemometric techniques. This book concerns itself solely with Design of Experiments (DOE); it does not deal with the more advanced chemometric methods such as multivariate analysis.

The authors are academic chemists from various universities in Brazil, and indeed the book is a translation of an original text in Portuguese. The quality of the translation is excellent; very clear and readable expositions are provided for all of the concepts. The pain of learning the statistics is ameliorated by the authors' almost whimsical style, and their choice of deceptively trivial examples such as counting beans or optimising journey times from home to work.

After an introductory overview, the first main chapter is devoted to statistics, introducing the fundamental concepts such as variance, standard errors, and the most relevant types of distribution (normal, *t*-, χ^2-, and F-distributions). The theory is broken up by examples, where the reader is invited to verify conclusions and, indeed, to establish fresh results for him-/herself, with helpful answers being provided at the back.

There follows a chapter on full factorial designs. The emphasis here is on the analysis and interpretation of the acquired data, with little advice on how to choose the factors or factor ranges to begin with. It explains how to derive the effects of each factor and their interactions and also how to use the data to estimate confidence intervals for these effects—either from replicated experiments or from redundant higher-order interactions—drawing on the statistical theory carefully established in the first chapter. It deals with the construction of a statistical model from the data—although some familiarity with matrix algebra is assumed for this purpose. The reader is equipped with the means to work everything out with a simple calculator. (In practice, of course, the bulk of that number-crunching would be done using computer software.)

The next chapter deals with the use of fractional factorial designs to investigate the influence of larger numbers of variables with a practical number of experiments. The important concepts here are design generators, aliasing (confounding) of effects, and design resolution. Alternative

saturated designs, such as the Plackett−Burman and Taguchi approaches, are also briefly discussed.

Before progressing to the next logical stage—response surface modelling—there is an interpolated chapter devoted to the mathematics behind empirical model-building that covers least-squares regression, analysis of variance (ANOVA), confidence intervals, statistical significance, and the difference between pure error and lack of fit.

The following chapter deals with methods for exploring the response surface, including steepest ascent, central composite designs, Box−Behnken designs, Doehlert designs, and D-optimal designs. There is some discussion on the best choice of star distance, using criteria of either rotatability or orthogonality, and on how many centre points to run. The use of desirability functions for the joint optimisation of conflicting responses is also considered.

The final two chapters deal respectively with designs for optimising mixtures (including the use of pseudocomponents in constrained systems) and with simplex optimisation, covering the basic simplex method and also the modified and super-modified versions.

All of the techniques—apart from the simplex methods—are illustrated by real-life examples, annotated with the authors' own comments and with references to the original literature sources where available. However, most of the examples come from analytical chemistry—the growing body of published synthetic organic applications has been comprehensively ignored.

All in all, a very competent and thorough introduction to this important area.

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