

# Advances in Lipid Methodology – Five

Edited by

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## Preface

Welcome to *Advances in Lipid Methodology*. This is the fifth volume in the *Advances* series, with a new editor (replacing the irreplaceable William Christie) and a new publisher (PJ Barnes & Associates, Bridgwater, Somerset, UK), but the objectives of this series have not changed. As noted in the prefaces of previous volumes, the goal of the *Advances* series is “to provide readable, up-to-date reviews of rapidly expanding areas of lipid analysis and practical examples which should be of immediate use to lipid analysts”. These have been, are and will continue to be the goals of the authors and Editors of this series.

As in previous volumes of *Advances*, the Editor has attempted to choose leading international experts to author individual chapters. Volume 5 contains four chapters of specific methodologies of lipid analysis. Professor Yakoob D. Che Man (Faculty of Food Science & Biotechnology, Universiti Putra, Serdang, Selangor, Malaysia) describes the application of differential scanning calorimetry to study the physical properties of individual fats and oils and fats and oil blends, to relating fatty acid unsaturation and chain length to an oil’s physical properties, to the detection of adulteration, to monitoring the oxidative stability of an oil and characterization of antioxidant effects in heated oils. The analysis of lipids by silver ion chromatography, a topic originally reviewed by Dr Boryana Nikolova-Damvanova (Institute of Organic Chemistry, Bulgarian Academy of Sciences, Sofia, Bulgaria) in *Advances* 1 (1992), has been expanded and updated by the author in *Advances* 5. Recent developments relating changes in sample retention patterns/resolution(s) with silver ion/solvent (Ag ion-related vs. normal phase effects)/solute form (fatty acids, fatty acid esters, TAG, etc.)/substrate interactions and the mechanisms of silver ion bonding have been applied to thin layer (TLC; one- and two-dimensional), high performance (Ag-HPLC), reversed phase (RP-HPLC), and supercritical fluid (SFC) chromatography. Specific examples of separation are included, as are such topics as TLC plate preparation/impregnation and methods of sample visualization.

The historical development of atmospheric pressure chemical ionization-mass spectrometry (APCI-MS), its utilization with HPLC as a “hyphenated technique”, and its application to the characterization of complex mixtures of lipids are reviewed in depth by Dr Craig Byrdwell, Florida State University, Boca Raton, FL, USA. Areas of APCI-MS discussion include instrument

configuration, theory and applications, the latter including both the qualitative and quantitative analysis of complex TAG configurational isomer mixtures, oxidized TAG, fatty acids, phospholipids and other lipid classes such as carotenoids, vitamins, and cholesterol. Concepts such as dual parallel APCI and APCI plus electrospray (ESI) MS, multiple ion probe MS, and atmospheric pressure photoionization (APPI) MS are discussed.

Another methodology discussed in *Advances 1*, supercritical fluid chromatography (SFC), has been updated and expanded by Dr Jerry King, Los Alamos National Laboratory, Los Alamos, New Mexico, USA. The advantages of SFC relative to GC or HPLC and its use in sample preparation and as a coupled technique are discussed, as are recent advances in analytical (both capillary and packed column), preparative and plant scale SFC. Specific applications include the characterization of TAG formulations, the isolation of lipid classes, the profiling of marine-derived oils, the detection and/or selective removal/isolation of minor seed oil components (such as estolides, cholesterol, phospholipids, free fatty acids, fat soluble vitamins), as well as their oxidation/degradation products (polymers, peroxides) and for detection of miscellaneous contaminants including herbicides, organochlorine pesticides, and antibiotics. The utilization of analytical SFC with a variety of detectors (ELSD, UV) and the inter-relationship of sorbent type with supercritical CO<sub>2</sub> pressure and flow rates is discussed. The chapter also includes an extensive listing of SFC-related books and internet sites.

Genetically modified oils (GMOs), their regulation and perceived health problems, continue to be a "hot topic". Dr Jan Hazebroek (Pioneer Hi-Bred International, Johnston, IA, USA) provides an overview of GMOs, with emphasis on methods of analysis, including recent developments in chromatographic and spectrophotometric/colorimetric methods (GC, TLC, HPLC, MS, NMR, NIR) to characterize GMO fatty acid composition/structure (TAG profiling), as well as phospholipids, tocopherols, tocotrienols and other unexpected fatty acids. Methods of seed sampling/seed oil extraction/derivatization (including potential sources of error) and utilization of the data to define a GMO's biological variability and potential molecular markers are also discussed.

The use of fatty acid profiling in the characterization of metabolic diseases is detailed by Professor Armand Christophe, University Hospital, Department of Endocrinology, Metabolic Diseases, Ghent, Belgium. Professor Christophe discusses the utilization of human plasma, serum or red blood cell phospholipid fatty acid profiling to diagnose such diseases as fatty acid deficiency, Refsum disease, cystic fibrosis, and extrahepatic biliary atresia. Problems in the extraction, storage and fractionation of blood lipids and lipid classes, in their derivatization and with methods of expressing the analytical results are discussed. Specific examples of fatty acid profiles and the effects of such variables as subject gender, age and season are included.

The potential consolidation of at least some of the many standard methods for analysis of fats and oils is currently an issue under serious discussion in Europe and North America. Dr James K. Daun (Program Manager, Canadian Grain Commission, Grain Research Laboratory, Winnipeg, Canada) and Mr Richard Cantrill (Technical Director, American Oil Chemists' Society, Champaign, IL, USA) present an overview of this topic, examining the purpose and types of standard methods, and their development, including sources of error, cost (instrumentation, time), formulation of standards, and validation. Other issues are discussed, such as sponsorship, formal protocol development (ISO), approval and professional associations and organizations for publication of "Official Methods" (AOAC, AOCS, IUPAC, IOC). Extensive listings of addresses, e-mail contacts (current officers, secretariats, and so on) and websites of standards writing organizations, of reference material sources, participating national standards organizations for analysis of animal and vegetable fats and oils (ISO TC 34/SC 11) are included, as is an intercomparison of methods table.

As exemplified by the chapter on silver ion chromatography, rapid advances in analytical methodologies have resulted in the re-evaluation of chapters published in previous volumes of *Advances*, often as recently as 8 to 10 years ago. Future volumes of *Advances* will strive to update or address such wide-ranging topics as: the chiral separation of lipids, lipid analysis by  $^{13}\text{C}$  and  $^2\text{H}$ -NMR, the analysis and the characterization of phospholipids, the application of stable isotopes to lipid studies, the characterization/preparation of structured lipids, government regulations and method development for *trans* fatty acids in foods, characterization of low-*trans*/low-fat formulations, GMO regulation/worldwide impact, lipids as phytonutrients, data acquisition software in analytical chemistry and Internet resources for the analytical chemist.

We, as authors and Editor, hope we have succeeded in our continuing efforts to "provide compact readable texts on all aspects of lipid chemistry and biochemistry". In this and future volumes, we hope to provide relevant, up-to-date and extensively-referenced examples of analytical methodologies and their application to the analysis and characterization of lipids. As envisioned by Bill Christie more than a decade ago, the *Advances* series will continue to strive to be a combination of "cookbook" and overview of lipid analytical methodologies. As always, your comments and suggestions are both solicited and welcome.

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