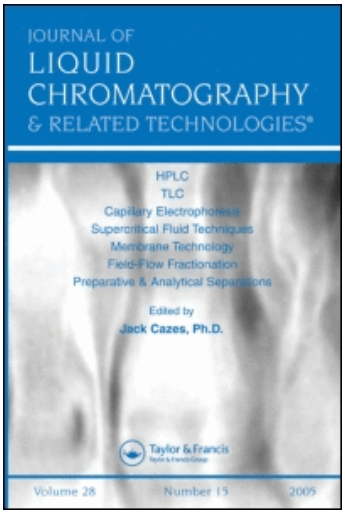

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

FOREWORD

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Online publication date: 04 May 2010

To cite this Article Sherma, Joseph and Fried, Bernard(2010) 'FOREWORD', Journal of Liquid Chromatography & Related Technologies, 33: 7, 875 — 879
To link to this Article: DOI: 10.1080/10826071003788942
URL: http://dx.doi.org/10.1080/10826071003788942

Journal of Liquid Chromatography & Related Technologies, 33:875–879, 2010

ISSN: 1082-6076 print/1520-572X online
DOI: 10.1080/10826071003788942

FOREWORD

This is the 12th special issue of the *Journal of Liquid Chromatography & Related Technologies* on thin layer chromatography (TLC) that we have guest edited, beginning in 1999 upon invitation of the Editor-in-Chief, Dr. Jack Cazes. The papers were submitted by recognized experts in TLC working in the United States of America, Hungary, Poland, Romania, Germany, and Bulgaria.

Literature searches via Chemical Abstracts and ISI Web of Science indicate continuing high research activity involving TLC on a worldwide basis, with over 1000 publications on that topic abstracted in 2009 in *Chemical Abstracts*. These publications indicated the continued movement to greater use of high performance TLC (HPTLC) rather than TLC plates, and of automated instruments such as sample applicators and densitometers for quantitative analysis and coupled mass spectrometry (MS).

The papers in this issue are examples of the most important current technique and application areas of TLC as verified in the 2010 Planar Chromatography biennial review written by Sherma and published in the ACS journal *Analytical Chemistry* (January 7, 2010; DOI: 10.1021/ac902643v).

In Paper 1, Oros and Cserháti report a study of the relationship between calculated physicochemical parameters and retention behavior of a group of phenylbenzamide derivatives of carboxamide fungicides carried out on silica gel and alumina layers impregnated with paraffin oil [reversed phase (RP) TLC]. Mobile phases were mixtures of methanol, acetonitrile, tetrahydrofuran, or acetone with water, and compound detection was made under ultraviolet (UV) light. The results indicated the importance of using both computed and measured parameters in quantitative structure retention relationship (QSRR) and quantitative structure activity relationship (QSAR) studies. The ability to impregnate layers with various reagents to improve selectivity, along with the great variety of available commercial precoated plates, is an important factor in the versatility and wide applicability of TLC for a great range of analytes. Pesticide QSAR and QSRR studies; identification and characterization of plant pesticides; pesticide metabolism, degradation, mobility, and lipophilicity studies; and pesticide residue and formulation analysis are important application areas of TLC that are reviewed biennially by Sherma in the *Journal of Environmental Science and Health, Part B.*

In Paper 2 by Kamińska and Choma, five amphoteric piperazynyl fluoroquinones were analyzed on RP octly bonded (C8) silica gel plates developed in a horizontal sandwich chamber, rather than the common ascending development in classical N-chambers, using acetonitrile-acidic aqueous mobile phases containing various concentrations of potassium perchlorate to control retention. Increasing concentrations of perchlorate ion increased retention up to 10–20 mM.

In Paper 3, Sârbu and Briciu evaluated a variety of oil- and fat-impregnated silica gel plates in lipophilicity and retention mechanism studies of natural sweeteners. Mobile phases with five organic solvent modifiers were used, and silver nitrate-sodium hydroxide reagent for detection. No significant differences between the oil- and fat-impregnated plates were indicated.

In Paper 4, Kalász et al. compared development in classical chambers and by the forced flow technique for 12 different pyridinium aldoximes. Overpressured layer chromatography (OPLC) on silica gel layers gave a four-times faster development with constant mobile phase velocity. Zones were detected by UV absorption at 254 nm on the layers containing a fluorescent phosphor (F-layers), which is undoubtedly the most commonly used detection method in TLC.

In Paper 5, Sajewicz, Wojtal, et al. used analytical and preparative silica gel plates, low temperature development in a sandwich chamber with toluene-ethyl acetate (95:5) mobile phase, and densitometric evaluation at 340 nm for fingerprint analysis of sesquiterpenes in essential oils originating from sage species. In this work, sample application with an autosampler, rather than manually with a microsyringe, and the most common type of densitometry (slit scanning in the reflection mode) were used. This is an example of the use of TLC in phytochemical analysis, which is a very important application area of TLC that was covered in the book "Thin Layer Chromatography in Phytochemistry," edited by Waksmundzka-Hajnos, Sherma, and Kowalska, which is Volume 99 in the Chromatographic Science Series edited by Dr. Cazes for CRC/Taylor & Francis. A companion book titled "HPLC in Phytochemical Analysis," edited by Waksmundzka-Hajnos and Sherma and to be published in the same Series, is currently in press.

In Paper 6 by Broszat et al. a videodensitometric quantification method with a charge coupled device (CCD) camera was reported for five triazine herbicides on silica gel HPTLC plates with a layer having spherical particles (rather than the usual irregular particles from which commercial plates are most commonly prepared) using vertical development with methyl-*t*-butyl ether-cyclohexane (1:1) mobile phase and detection based on a derivatization reaction using chlorine and starch-iodine. The method was found to be inexpensive, rapid, and reliable. The great number of available universal

or selective and sensitive postchromatographic spray and dip reagents, many of which can be used in sequence on a plate containing multiple chromatograms, is a significant advantage of TLC for analyte detection compared to column liquid chromatography (HPLC).

In Paper 7, Aranda and Morlock developed and validated a high throughput method for pyritinol quantification in a solid pharmaceutical formulation. Samples were spotted automatically onto a silica gel HPTLC plates that was developed in a twin trough chamber (TTC) with dichloromethane-methanol-formic acid (9:1:1), followed by slit scanning densitometry at 300 nm. The TTC is a widely used special N-chamber that requires very little mobile phase volume and provides two compartments, one for plate development and the other for conditioning the vapor phase. Peak purity and identity were determined by UV spectrometry and MS coupled directly to an electrospray ionization single quadrupole mass spectrometer through an interface. The combination of TLC and MS is being much more widely reported for qualitative and quantitative analysis of a variety of analytes than in the past. There are more papers reporting the TLC determination of synthetic and natural (herbal) drugs, most with the use of densitometry, than any other application area.

TLC with biological detection is becoming increasingly important, and Papers 8 and 9 report utilization of this approach. In Paper 8, Ueta et al. showed that short neck clam extract is an excellent source of vitamin B12 for persons with a deficiency of this nutrient. Silica gel TLC with 1-butanol-2-propanol-water (10:7:10) and 2-propanol-20% ammonia-water (7:1:2) mobile phases and bioautography with vitamin B12 dependent *Escherichia coli* 215 were used in the study.

In Paper 9, Baumgartner and Schwack applied HPTLC coupled with bioluminescence detection using the luminescent bacterium *Vibrio fischeri* for quantitative analysis of wastewater on silica gel HPTLC plates with automated sample application. Chromatogram images containing black spots on a bright background were quantified using videoscanning and VideoScan and ChromQuest computer programs.

Paper 10 by Antosch et al., an example of the determination of an herbal drug, reports development and validation of a method for quantification of the marker compound xylose in *Plantago ovata Forssk* raw material and finished product. After automatic sample application onto silica gel plates, the mobile phase acetonitrile-water (90:10) was used for ascending double development. 4-Aminobenzoic acid derivatization reagent detected xylose as a red-brown zone that was scanned at 366 nm.

Lipids are very often determined by TLC because of the lack of a chromophore group in the compound structures allowing easy detection of the compounds by HPLC or suitable volatility for GC. Papers 11 and 12 report lipid TLC analyses. In Paper 11, Bolstridge et al. used HPTLC on silica gel

plates with channels and a preadsorbent zone to study the effects of temperature on the neutral lipid content of the medically important snail *Biomphalaria glabrata*. The Mangold mobile phase, petroleum ether-diethyl ether-glacial acetic acid (80:20:1), phosphomolybdic acid detection reagent, and densitometry at 610 nm were employed. Laned preadsorbent plates are a great advantage for quantitative analysis with manual sample application.

In paper 12, Marekov et al. used silver ion TLC to determine minor triacylglycerols as indication of the adulteration of virgin olive oils. Continuous mobile phase development of silica gel G plates was carried out in open containers, and zone detection by charring was performed using consecutive treatment with bromine and sulfuryl chloride vapors. Quantification was by zigzag scanning in the reflection mode at 450 nm.

The increasing use of TLC in the analysis of biological samples is again illustrated in Paper 13, in which Vasta et al. determined estivation induced changes in the amino acid content of *B. glabrata* snails. Analyses were carried out on silica gel and cellulose HPTLC layers with optimized mobile phases, and separated zones were detected by spraying with ninhydrin reagent followed by heating. Quantification was by densitometry at 610 nm.

In Paper 14, Parys and Pyka used silica gel layers developed with methanol-benzene (50:50) and acetone-hexane (40:50) mobile phases and densitometry at analyte maximum UV absorption wavelengths to study the chemical stability of nicotinic acid and its esters upon 1–7 hours of heating at 120°C. The most unstable compounds were found to be ethyl and methyl nicotinate.

In Paper 15, Sajewicz, Kronenbach, et al. investigated of the oscillatory in vitro chiral conversion of *R-beta*-hydroxybutyric acid. It was found that this compound can undergo the conversion, similar to other *alpha*-hydroxy acids that were studied earlier, despite the methylene spacer between the carboxyl and hydroxyl groups in its structure. A book titled "Thin Layer Chromatography in Chiral Separations and Analysis," edited by Kowalska and Sherma, Volume 98 in the Cazes Chromatographic Science Series, covers this field comprehensively.

After submitting the group of 15 papers to Dr. Cazes, we were greatly saddened to learn of his death at The Villages, Lady Lake, FL, on February 16, 2010, at the age of 76. Dr. Cazes was a leading expert in the theory, practice, and applications of liquid chromatography, particularly gel permeation chromatography in which field he published numerous journal articles and taught an American Chemical Society short course. In addition to serving as editor of this journal for over 30 years, he was also editor of *Instrumentation Science & Technology, Preparative Biochemistry & Biotechnology*, and the *Journal of Immunoassay and Immunochemistry*, as well as the *Chromatographic Science Series* with over 100 volumes, the *Encyclopedia of Chromatography, Third Edition*, and *Ewing's Analytical Instrumentation Handbook, Third*

Edition, titles all formerly published by Marcel Dekker, Inc., since acquired and currently published by CRC Press/Taylor & Francis Group. Dr. Cazes was a consultant in chromatography and analytical chemistry, and he passed on his great knowledge to students by teaching an organic chemistry course at Rutgers University, New Brunswick, NJ, and later special topics graduate level courses as a Visiting Scholar and Adjunct Professor at Florida Atlantic University, Boca Raton. Under Dr. Cazes' editorship, this journal has become one of the premier sources of new research and review articles on liquid chromatography and related methods in the world today.

We will begin to solicit papers in September, 2010, for our 2011 guest edited special TLC issue. We invite readers to send us feedback on this and our past special issues, as well as suggestions for topics and contributors for the next issue. We also encourage the submission of papers on TLC and HPTLC for regular issues of the *Journal of Liquid Chromatography & Related Technologies*.

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March, 2010