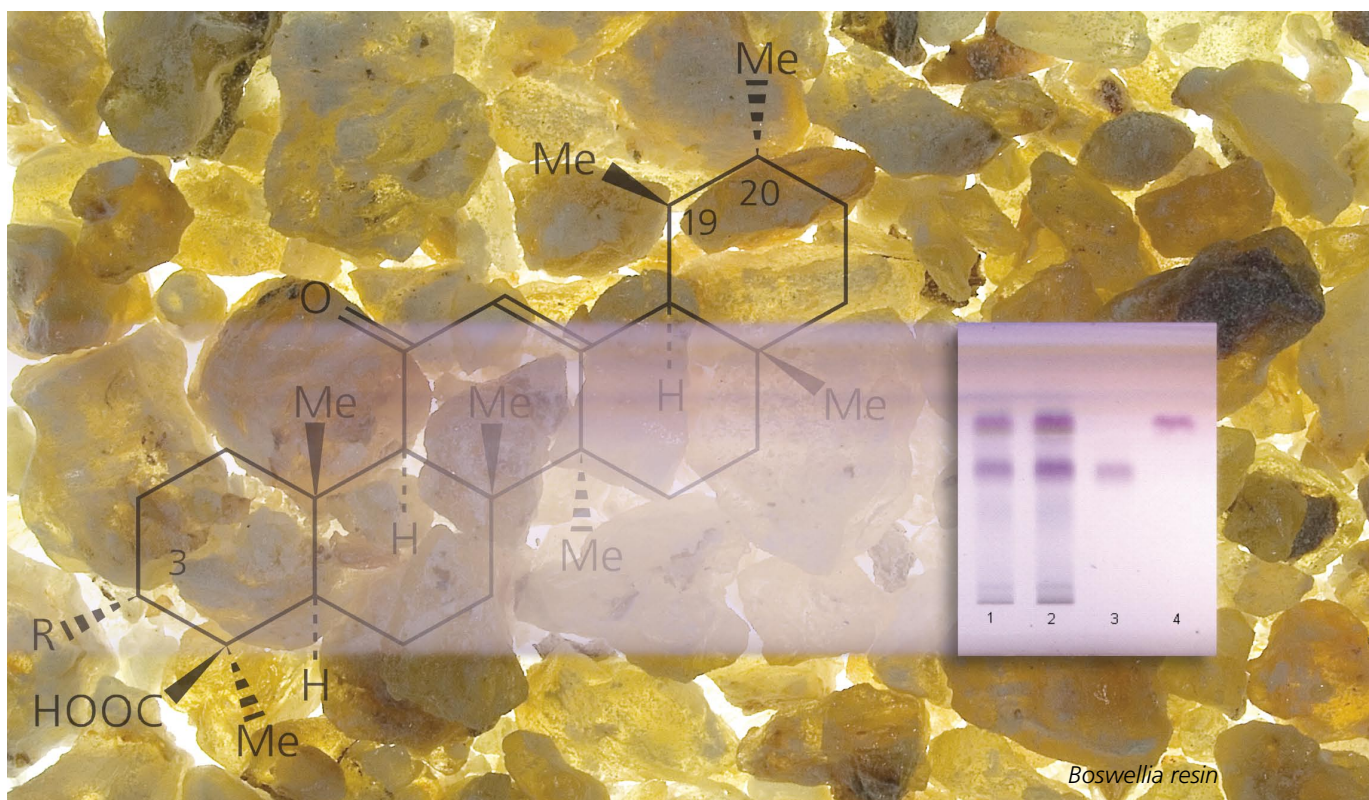


CBS

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**Analytical tasks –
efficiently solved by HPTLC**

CAMAG 104

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Planar Chromatography in Practice

A comparison between HPLC and HPTLC for the separation and quantification of boswellic acids in *Boswellia serrata* extracts



Left to right: Prof. Alexander Shikov*, Dr. Olga Pozharitskaya, Dr. Svetlana Ivanova, Prof. Valery Makarov, Dr. Vera Kosman

The Department of Standardization and New Technologies of the Saint Petersburg Institute of Pharmacy was introduced in CBS 102. In their daily routine, HPTLC is especially helpful for the optimization of the extraction, determination of antiradical activity, or the study of the kinetics of active metabolites in plasma. They currently use more than 20 validated, quantitative HPTLC methods, such as quantification of chlorogenic, caffeic and ferulic acids in coffee bean extracts, curcuminoids in plasma and dosage forms, bile acids and cholesterol in the bile of rats, etc.

Introduction

Boswellia serrata Roxb. (Indian frankincense) is an Indian medical plant and its gum resin is used as an antipyretic, analgesic, diuretic, or antidysenteric traditional medicine, among other usages. The anti-inflammatory and antiarthritic activity of its extracts has been tested in animal experiments and in clinical trials [1, 2]. The main active compounds of *Boswellia serrata* extracts are triterpenic acids [2] like β -boswellic acid (BA), acetyl- β -boswellic acid (ABA), 11-keto- β -boswellic acid (KBA) and acetyl-11-keto- β -boswellic acid (AKBA). For the analysis of boswellic acids, a nonaqueous titration method [3], HPLC [4], GC-MS [5], LC-MS [6], and HPTLC methods [7] have been reported.

The aim of this work was to compare the capabilities of two chromatographic methods, HPTLC and HPLC, for identification and quantification of the four major boswellic acids in *Boswellia serrata* extracts.

Chromatogram layer and HPLC column

- HPTLC plates silica gel 60 F₂₅₄, 10 × 10 cm (Merck).
- HPLC column Luna C18 (150 × 4.6 mm, 5 μm, Phenomenex, USA) and a SecurityGuard 20-mm precolumn with the same sorbent

Standard solutions

Methanolic solutions (0.5 mg/mL) of BA, ABA, KBA, and AKBA were used.

Sample preparation

Commercially available standardized *Boswellia serrata* extracts were a kind gift from New Ambadi Estates Pvt. Ltd. (Chennai, India). Sample solutions were prepared by dissolving of the *Boswellia serrata* extracts in methanol (10 mg/mL).

Sample application and HPLC injection

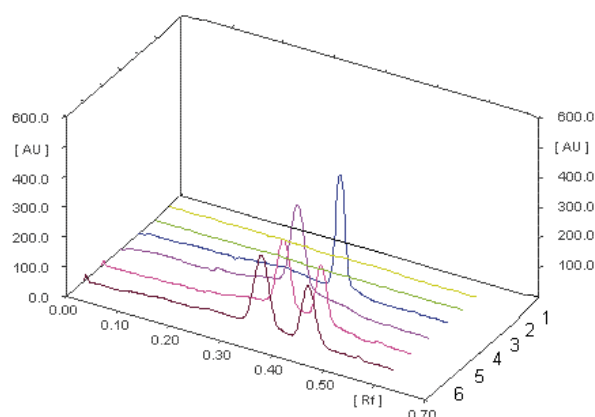
- Bandwise with Linomat 5, band length 6 mm, distance from lower edge 10 mm, distance on both sides min. 10 mm, application volumes 1–20 μL
- Injection volume of 20 μL for HPLC analysis

Chromatography

- In the Twin-Trough Chamber pre-saturated (for 15 min, using wetted filter paper) with n-hexane – ethyl acetate – glacial acetic acid 16:5:1 up to a migration distance of 7 cm (ca. 10 min)
- HPLC (Waters Inc., USA) consisting of two 510 series pumps, an automated gradient controller and a 484 model tunable absorbance detector. Separation was carried out at room temperature with a flow rate of 1 mL/min using a 20-min linear gradient from 75 % to 100 % acetonitrile (other phase: 0.03 % aqueous trifluoroacetic acid solution), followed by a 15-min isocratic step.

Densitometry

- Absorption measurement at 254 nm for KBA and AKBA, and after derivatization at 560 nm for BA and ABA using TLC Scanner 3 and winCats software.
- HPLC-UV detection at 254 nm for KBA and AKBA and at 210 nm for BA and ABA.

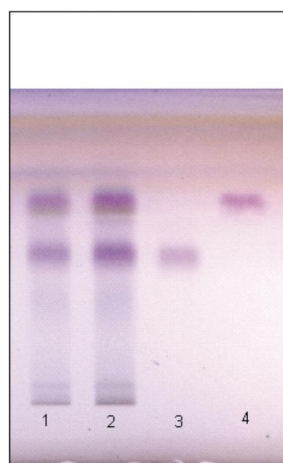


HPTLC densitogram at 254 nm of BA (track 1), ABA (track 2), AKBA (track 3), KBA (track 4), and two *B. serrata* extracts (track 5 and 6)

Post-chromatographic derivatization

For visualization of BA and ABA, the plate was manually dipped in anisaldehyde sulfuric acid reagent (1 mL anisaldehyde and 2 mL conc. sulfuric acid in 100 mL glacial acetic acid) followed by heating at 110 °C for 5 min on the TLC plate heater.

Note (editor): Using automated immersion by the TLC Chromatogram Immersion Device (immersion time 1 s, vertical speed 3.5 cm/s) will improve the precision.



Visualization by anisaldehyde sulfuric acid reagent: tracks 1 and 2 *B. serrata* extract at 5 and 10 μg/band, track 3 BA, and track 4 ABA

Results and discussion

For both methods, quantification of boswellic acids by external standard calibration was performed, and both methods were validated according to the ICH guidelines on the validation of analytical methods. Using a 20-μL volume for injection in HPLC, the limits of detection (LOD) for KBA and AKBA were

6–8 ng and for BA and ABA 60–80 ng. In HPTLC just a 2- μ L volume was applied. LOD obtained were 150 ng for KBA and AKBA and 100 ng for BA and ABA. If required, LOD can be improved using a higher volume for application. Regarding precision, the relative standard deviations (%RSD) of boswellic acids varied from 6 to 18 % using HPLC. For HPTLC, %RSD of AKBA and KBA were ≤ 2 %, and after derivatization, for BA and ABA about 10 %, which could be improved by automated immersion.

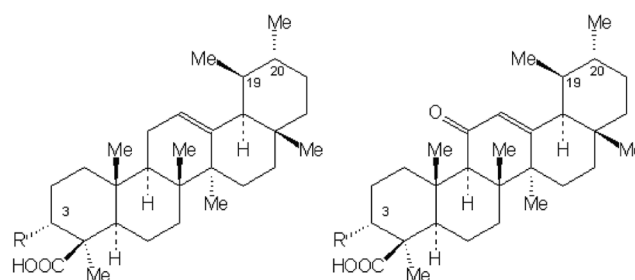
Two differently marketed, dry *Boswellia serrata* extracts were analyzed in triplicate using both methods.

Results of HPLC and HPTLC quantification of boswellic acids in *Boswellia* extracts

Compound	Sample 1 (% w/w) ^a		Sample 2 (% w/w) ^a	
	HPLC	HPTLC	HPLC	HPTLC
BA	18.9 \pm 1.2	19.0 \pm 1.7	22.4 \pm 0.7	23.4 \pm 1.9
ABA	11.9 \pm 1.2	12.4 \pm 1.4	11.6 \pm 0.3	12.3 \pm 1.2
KBA	3.8 \pm 0.6	3.4 \pm 0.1	8.0 \pm 0.1	7.7 \pm 0.1
AKBA	3.9 \pm 0.7	3.6 \pm 0.1	5.1 \pm 0.2	5.2 \pm 0.1
Total	38.5	37.9	47.1	48.6

^a \pm standard deviation ($n = 3$) expressed on dry weight

The two methods treated as paired data were compared by the matched pair Student's t-test. The calculated t-values were between 2.16 and 2.45 for the different boswellic acids and below the t-table value of 2.57. Hence, it was concluded that both methods gave identical results for the analysis of boswellic acids in the extracts. There was no statistically significant difference ($P = 99$ %) between the mean values for all extracts. Considering the fast analysis time, low solvent consumption and high sample throughput capacity, HPTLC is the method of choice for routine analysis.



Boswellic acids:

BA with R = α -OH

KBA with R = α -OH

ABA with R = α -CH₃COO AKBA with R = α -CH₃COO

Further information is available on request from the authors.

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Validated HPTLC method for skin lipids



Karin Rothenbühler

In cooperation with the University of Basel and under supervision of Prof. Matthias Hamburger, Ms. Karin Rothenbühler has worked on her master thesis [1] in Pharmaceutical Biology at the CAMAG laboratory.

Introduction

Even though numerous literature references indicate that TLC/HPTLC is the method of choice for skin lipid analysis, no validated quantitative HPTLC method was found. Following the recent publication on the HPTLC analysis of phospholipids [2], Ms. Rothenbühler developed and validated a HPTLC method for the most important markers of non-polar lipid classes of the human stratum corneum: squalene, triolein, palmitic acid, 1,2-dipalmitoyl-sn-glycerol, stearyl palmitate, cholesteryl palmitate, and cholesterol.

In combination with appropriate sampling procedures the method was rapid, robust and reliable for use by the R&D laboratories of cosmetic industry. The advantages of HPTLC, in comparison to RP-HPLC, are its simple derivatization as a prerequisite for lipid detection and the separation based on functional groups.

Sample preparation

Skin samples were obtained from the inner forearm of volunteers either by direct extraction with ethanol, abrasion of a defined area with a razor blade or with Epiglu, a tissue adhesive for medicinal use.

Standard solutions

2 mg each of squalene, triolein, palmitic acid, 1,2-dipalmitoyl-sn-glycerol, stearyl palmitate, cholesteryl palmitate, and cholesterol were dissolved in 10 mL of

chloroform – methanol 1:1. For quantification 1 mL of this solution was diluted with 4 mL of chloroform – methanol 1:1.

Chromatogram layer

HPTLC plates LiChrospher silica gel 60 F₂₅₄ (Merck), 20 × 10 cm, pre-washed by developing with methanol followed by drying in an oven at 120 °C for 20 min.

Sample application

Bandwise with ATS4, band length 8 mm, track distance min. 10 mm, distance from lower edge 8 mm, distance from left edge min. 15 mm, application volumes 2–30 µL of samples and 2.5–10 µL of standard solutions.

Chromatography

In the ADC2 with chamber saturation for 20 min first with toluene to a migration distance of 80 mm followed by drying for 5 min, then with *n*-hexane, *t*-butyl methyl ether, acetic acid 80:20:1 to 45 mm. Separation was affected by the plate activity, therefore plates were conditioned prior to both developments at 33 % relative humidity for 10 min using a saturated solution of magnesium chloride.

Post-chromatographic derivatization

Copper(II) sulfate reagent was prepared by dissolving 20 g Copper(II) sulfate pentahydrate in 200 mL of cooled methanol and then adding 8 mL of sulphuric acid 98 % and 8 mL of orthophosphoric acid 85 %. With the Immersion Device III the plate was dipped for 6 s into the reagent, then dried for 30 s with cold air and finally heated on a plate heater for 30 min at 140 °C.

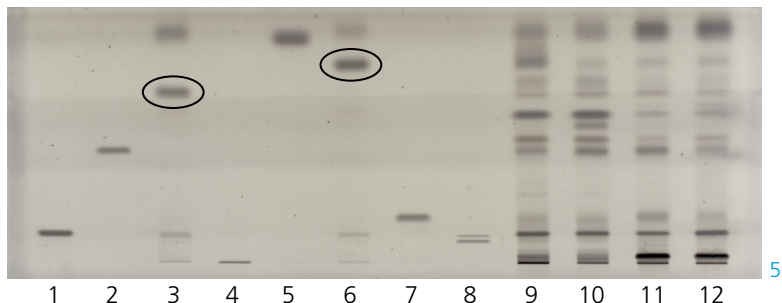
Densitometry

TLC Scanner 3 with winCATS software, absorption measurement at 350 nm.

Results and discussion

Since there was no standardized methodology for sampling of human skin samples, various techniques were evaluated. Skin samples from the inner forearm of volunteers were obtained by use of various commercial tapes, medicinal tissue adhesive,

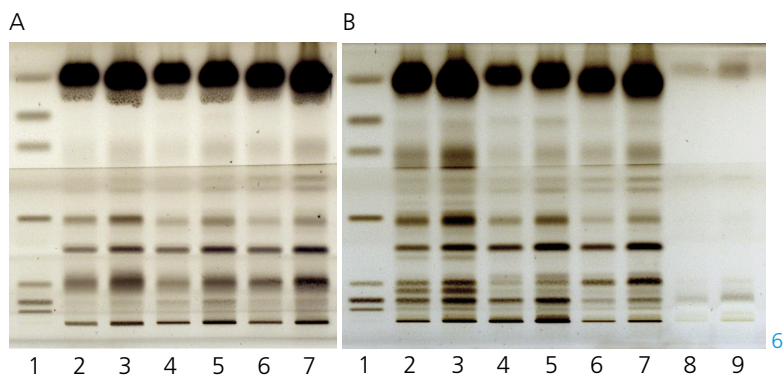
abrasion with a razor blade, cold wax, aluminium foil, cling film or by direct application of organic solvents.



Standards (track 1: cholesterol, 2: triolein, 3: stearyl palmitate*, 4: ceramid VI, 5: squalene, 6: cholesteryl palmitate*, 7: palmitic acid, 8: 1,2 dipalmitoyl-sn-glycerol) and various extracts of skin lipids (track 9: using ethanol, 10: cyclohexane – ethanol 1:9, 11: cyclohexane – ethanol 1:4, 12: n-hexane – methanol 2:3)

*encircled, other zones are impurities

The major challenge involved with the use of tapes and glues was to selectively extract the skin lipids from the carrier material, whereas the direct application of organic solvents was limited to ethanol to avoid skin irritations. Three sampling methods (direct extraction with ethanol, abrasion, Epiglu) were suitable for quantitative investigations, as neither skin nor glue matrix interfered with the lipid zones. However, standardization of the sampling procedure was still difficult because the depth of skin removed (number of stratum corneum layers) was unclear and the area of treated skin was difficult to define; further investigations are under way.



Sampling A) by direct abrasion and B) with Epiglu; track 1: lipid standard mix, 2–7: skin lipid samples, 8–9: Epiglu blanks

The method was validated regarding stability, robustness, precision of R_f values and standard determinations as well as linear range. All lipid standards were stable for at least 3 h in chloroform – methanol 1:1 and on the plate prior to development. Standards as well as extracted skin samples were stable during chromatography. The derivatized chromatogram was stable for at least 2 h. The influence of relative humidity on the

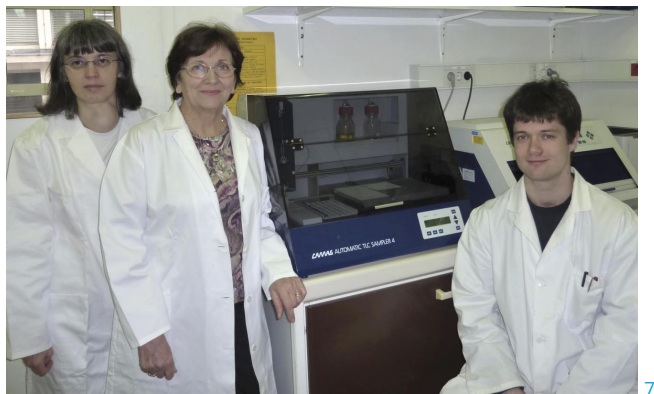
separation was evaluated at 5 %, 33 %, 47 %, and 75 % using the ADC 2. For reproducible selectivity, the relative humidity must be maintained between 33 % and 47 %. The precision of the R_f values of seven compounds was determined between plates (3 plates developed on the same day, followed by one plate each on two different days). Differences of R_f values were <0.03 and thus well below the acceptance criterion of <0.05 . Precision of measurement ($n = 9$) was compared on various HPTLC silica gel 60 plates of different layer thickness (100 and 200 μm) and particle shapes (irregular and spherical particles LiChrospher). The HPTLC LiChrospher Si 60 F_{2545} plates with a thickness of 200 μm showed the lowest relative standard deviations ($\%RSD \leq 5\%$) for all investigated compounds. The linear range for all but two of the selected markers was between 100 and 350 ng/band (40–160 ng for cholesterol, 100–280 ng for 1,2 dipalmitoyl-sn-glycerol). Calibration curves showed correlation coefficients of $r > 0.9975$.

Further information is available on request from the authors.

[1] K. Rothenbühler, Master thesis, Institute for Pharmaceutical Biology, University of Basel, 2009

[2] D. Handloser, V. Widmer, E. Reich, J. Liq. Chromatogr. Rel. Technol. 31 (2008) 1857

Separation of common plant triterpenoids by HPTLC



Left to right: Dr. Irena Vovk*, Dr. Breda Simonovska and Mitja Martelanc

The Laboratory for Food Chemistry at the National Institute of Chemistry in Ljubljana (www.ki.si) is the leading research group in the field of planar chromatography in Slovenia, providing consulting for industry and other institutions. Research and development activities are primarily focused on the field of nutraceuticals and the development of analytical methods based on chromatographic techniques.

Introduction

Triterpenoids represent a large class of secondary metabolites commonly present in plants, especially in leaf and fruit epicuticular waxes. Some of them exhibit beneficial activities, such as anti-inflammatory ones. Therefore, they are of emerging importance as constituents of food supplements and functional foods.

In general HPLC is used for determination of triterpenoids, but due to their lack of chromophores and numerous possible isomeric structures, the choice of detector and mobile phases has limitations. On the other hand, HPTLC is indispensable in the investigation of triterpenoids, because it provides unique separations with a wealth of potential mobile phases and with possibilities for in situ derivatization, which results in characteristic specific coloration and fluorescence of the separated bands [1, 2]. In the investigation of triterpenoids in plant extracts, the proposed HPTLC methods enable easy, fast and inexpensive screening. Depending on the sample characteristics, quantification is possible.

Standard solutions

Standard solutions and mixtures were prepared in *n*-propanol (0.1 mg/mL).

Sample preparation

Fresh tomato fruit and fresh leaves of cabbage, rosemary and sage were immersed into dichloromethane. Pulverized oak bark was extracted with the same solvent. After filtration and evaporation of the solvent, final test solutions were prepared in *n*-propanol with concentrations of 5 mg/mL (cabbage and rosemary leaves), 30 mg/mL (sage leaves), 10 mg/mL (oak bark) and 2 mg/mL (tomato fruit) [2].

Chromatogram layer

HPTLC plates silica gel 60 (Merck), 20 × 10 cm, pre-washed by developing in chloroform – methanol 1:1 and HPTLC plates RP18 (Merck), 20 × 10 cm, pre-washed by developing in acetone, followed by drying on the TLC plate heater at 110 °C for 10 minutes.

Sample application

Bandwise with Linomat, band length 6 mm, distance from lower plate edge 5 mm, from left edge 12 mm, track distance 10 mm, application volumes as follows: 6 µL (cabbage leaves), 7 µL (rosemary leaves), 5 µL (sage leaves), 4 µL (oak bark), and 6 µL (tomato), for standard solutions 2-8 µL.

Chromatography

In the Horizontal Developing Chamber with *n*-hexane – ethyl acetate 5:1 on silica gel plate (developing time 15 min), whereby the conditioning tray was covered with the developing solvent, and with ethyl acetate – acetonitrile 3:2 or acetone – acetonitrile 5:1 on RP18 plate (developing time 17 min), running distance 8 cm

Post-chromatographic derivatization

Dried plates were dipped for 2 s in anisaldehyde sulfuric acid reagent (16 mL sulfuric acid and 1 mL *p*-methoxybenzaldehyde were added to 20 mL acetic acid and 170 mL methanol during cooling with ice water) using the Chromatogram Immersion Device (vertical speed 3.5 cm/s), dried in a stream of warm air and heated at 110 °C with a TLC Plate Heater for

continuation on page 9

Online Coupling of HPTLC-MS on the road to success



The one-day seminars announced in CBS 102 met with great interest. Between 30 and 35 analysts attended each of the seminars held in 2009 at Langenau, Offenburg, Münster, Berlin, Basle and Vienna. The participants came from companies and institutes where TLC/HPTLC analysis is a part of the daily routine in foods, pharmaceuticals, water, organic synthesis and natural products' research. The seminar's aim was to keep the participants informed on the innovative technique of online coupling and its potential.

Competent speakers reported of their own experience: Dr. H. Luftmann (University of Münster), who developed the interface, reported of the history and background and his experience with its application in organic synthesis. Dr. W. Schulz (Zweckverband Landeswasserversorgung in Langenau) presented the use of HPTLC-MS coupling for identification of organic trace substances in raw and drinking water, Prof. Dr. H.-R. Schmutz (University Northwest Switzerland in Muttenz) lectured on HPTLC-MS of drug compounds and plant ingredients and PD Dr. Gerda Morlock (University of Hohenheim in Stuttgart) covered applications in the field of food and pharmaceutical analysis. A wide range of analytes of different structural groups was shown such as atrazine and its metabolites, metoprolol and its

degradation products, caffeine, paracetamol, acetylsalicyl acid, ginsenoides, isopropylthioxanthone, food dyes, ergotamine, pyridinol and harmane.

In the afternoon there were practical demonstrations of the interface with the MS systems available in the respective laboratories. Some participants brought sample chromatograms which were successfully processed. All participants were convinced of the great potential of the new coupling technique with respect to time saving, cost effectiveness and convenience.

Success obliges!

CAMAG will offer the same type of seminars in 2010

- 16.04.2010 University of Oldenburg, Prof. Dr. Christoffers, 26111 Oldenburg, Germany
- 03.06.2010 University of Leiden, Prof. Dr. Overkleeft, 2333 CC Leiden, The Netherlands
- 04.06.2010 University of Gent, Prof. Dr. De Spiegeleer, 9000 Gent, Belgium
- 29.06.2010 Technical University of Munich, Prof. Dr. Schieberle, 85345 Munich, Germany

If you are interested please register by E-mail (info@camag-Berlin.de). You will receive detailed information. Attendance is free of charge.

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Liebe Freunde

Das effiziente Lösen analytischer Fragestellungen mit der HPTLC ist Schwerpunkt dieser Ausgabe. Voraussetzung ist, dass das gesamte Instrumentarium optimal eingesetzt wird – eine Folgerung, die auch im Beitrag Coran (S. 5) gezogen wird.

Die HPTLC ist heute – 35 Jahre nach Einführung dieser Schichten – längst in der Praxis angekommen. Unsere zahlreichen Kundenkontakte zeigen allerdings auch, dass ein großer Teil der Prüfvorschriften in Industrie und Kontrollinstituten noch auf den klassischen DC-Schichten basiert. Dies liegt einerseits am mangelnden Kenntnisstand eines Teils der Anwender, andererseits am bürokratischen Aufwand, der erforderlich ist, eine DC-Methode auf HPTLC umzustellen. Aber wäre es nicht besser, die noch vorhandenen DC-Methoden Schritt für Schritt durch die HPTLC mit ihrem großen Potential zu ersetzen, als sich jahrelang im Routinebetrieb mit einer unzulänglichen Prüfvorschrift abzufinden? Bürokratie darf kein Hemmschuh für den analytischen Fortschritt sein!

Vorreiter sollten eigentlich die Universitäten sein. Doch gerade dort wird, wenn überhaupt, den Studierenden die Dünnschicht-Chromatographie oft nur in ihrem rudimentären Stand nahe gebracht. CAMAG ist bemüht, durch Seminare den Wissensstand zu aktualisieren. Wenn Sie als Lehrperson daran interessiert sind, melden Sie sich bitte unter info@camag-berlin.de

Eine Gelegenheit, Fortschritte in der HPTLC zu erfahren, ergibt sich beim International Symposium on High-Performance Thin-Layer Chromatography in Basel vom 6.–8. Juli 2011 (letzte gelbe Seite). Bitte reservieren Sie sich diesen Termin.

Mit freundlichen Grüßen

Gerda Morlock

Gerda Morlock
cbs@camag.com

Dear friends

The efficient solution of analytical tasks by HPTLC is the focus of this issue. An absolute prerequisite is a sound knowledge of the state-of-the-art technique – a conclusion drawn also by Professor Coran (p. 5).

HPTLC is now clearly established in the laboratory, 35 years after the introduction of the high-performance layers. But in the field, industry as well as regulatory laboratories, we meet many customers still using classical TLC layers and old fashioned procedures. To a large extent this is due to the fact that standard operating procedures are based on classical TLC layers, and that substituting an established procedure with HPTLC requires a lot of bureaucracy. Nonetheless, even this appears much better than struggling along for years with inadequate procedures and more importantly, suboptimal results.

Outriders for teaching state-of-the-art knowledge should be university institutes. But unfortunately, nowhere is it more apparent than at universities that TLC is just rudimentarily presented to students, if at all! CAMAG strives to upgrade the knowledge of contemporary HPTLC at universities by offering seminars. If you, as a teaching person are interested in a CAMAG seminar at your institute, contact us via info@camag.com.

An opportunity to see first hand the progress in HPTLC is the attendance of the International Symposium on High-Performance Thin-Layer Chromatography in Basle, July 6th–8th, 2011 (last yellow page). Make a note!

Sincerely,

Gerda Morlock

Gerda Morlock
cbs@camag.com



CAMAG

**MARCH
2010 104**

THE CBS CLASSIFICATION SYSTEM

- 1. Reviews and books**
 - a) Books on TLC
 - b) Books containing one or several chapters on TLC
 - c) Books containing frequent TLC information spread over several chapters of other information
- 2. Fundamentals, theory and general**
 - a) General b) Thermodynamics and theoretical relationship
 - c) Relationship between structure and chrom. behaviour
 - d) Measurement of physico-chemical and related values
 - e) Optimization of solvent systems
 - f) Validation of methods
- 3. General techniques** (unless they are restricted to the application within one or two classification sections)
 - a) New apparatus/techniques for sample preparation
 - b) Separation material
 - c) New apparatus for sample application/dosage
 - d) New apparatus/techniques for chromatogram development
 - e) New apparatus/techniques for pre- or post-chromatographic derivatization
 - f) New apparatus/techniques for quantitative evaluation
 - g) New apparatus/techniques for other TLC steps (distinguished from section 4)
- 4. Special techniques**
 - a) Automation of sample preparation/application
 - b) Automation of complex chromatogram developing techniques
 - c) Automation, computer application in quantitative chromatogram evaluation
 - d) Combination of TLC with other chromatographic techniques
 - e) Combination of TLC with other (non-chromatographic) techniques...MS, IR...etc.
- 5. Hydrocarbons and halogen derivatives**
 - a) Aliphatic hydrocarbons
 - b) Cyclic hydrocarbons
 - c) Halogen derivatives
 - d) Complex hydrocarbon mixtures
- 6. Alcohols**
- 7. Phenols**
- 8. Substances containing heterocyclic oxygen**
 - a) Flavonoids
 - b) Other compounds with heterocyclic oxygen
- 9. Oxo compounds, ethers and epoxides**
- 10. Carbohydrates**
 - a) Mono- and oligosaccharides, structural studies
 - b) Polysaccharides, mucopolysaccharides, lipopolysaccharides
- 11. Organic acids and lipids**
 - a) Organic acids and simple esters
 - b) Prostaglandins
 - c) Lipids and their constituents
 - d) Lipoproteins and their constituents
 - e) Glycosphingolipids (gangliosides, sulfatides, neutral glycosphingolipids)
- 12. Organic peroxides**
- 13. Steroids**
 - a) Pregnane and androstane derivatives
 - b) Estrogens
 - c) Sterols
 - d) Bile acids and alcohols
 - e) Ecdysones and other insect steroid hormones
- 14. Steroid glycosides, saponins and other terpenoid glycosides**
- 15. Terpenes and other volatile plant ingredients**
 - a) Terpenes
 - b) Essential oils
- 16. Nitro and nitroso compounds**
- 17. Amines, amides and related nitrogen compounds**
 - a) Amines and polyamines
 - b) Catecholamines and their metabolites
 - c) Amino derivatives and amides (excluding peptides)
- 18. Amino acids and peptides, chemical structure of proteins**
 - a) Amino acids and their derivatives
 - b) Peptides and peptidic proteinous hormones
- 19. Proteins**
- 20. Enzymes**
- 21. Purines, pyrimidines, nucleic acids and their constituents**
 - a) Purines, pyrimidines, nucleosides, nucleotides
 - b) Nucleic acids, RNA, DNA
- 22. Alkaloids**
- 23. Other substances containing heterocyclic nitrogen**
 - a) Porphyrins and other pyrroles
 - b) Bile pigments
 - c) Indole derivatives
 - d) Pyridine derivatives
 - e) other N-heterocyclic compounds
- 24. Organic sulfur compounds**
- 25. Organic phosphorus compounds** (other than phospholipids)
- 26. Organometallic and related compounds**
 - a) Organometallic compounds
 - b) Boranes, silanes and related non-metallic compounds
 - c) Coordination compounds
- 27. Vitamins and various growth regulators** (non-peptidic)
- 28. Antibiotics, Mycotoxins**
 - a) Antibiotics
 - b) Aflatoxins and other mycotoxins
- 29. Pesticides and other agrochemicals**
 - a) Chlorinated insecticides
 - b) Phosphorus insecticides
 - c) Carbamates
 - d) Herbicides
 - e) Fungicides
 - f) Other types of pesticides and various agrochemicals
- 30. Synthetic and natural dyes**
 - a) Synthetic dyes
 - b) Chloroplasts and other natural pigments
- 31. Plastics and their intermediates**
- 32. Pharmaceutical and biomedical applications**
 - a) Synthetic drugs
 - b) Pharmacokinetic studies
 - c) Drug monitoring
 - d) Toxicological applications
 - e) Plant extracts, herbal and traditional medicines
 - f) Clinico-chemical applications and profiling body fluids
- 33. Inorganic substances**
 - a) Cations
 - b) Anions
- 34. Radioactive and other isotopic compounds**
- 35. Other technical products and complex mixtures**
 - a) Surfactants
 - b) Antioxidants and preservatives
 - c) Various specific technical products
 - d) Complex mixtures and non-identified compounds
- 36. Thin-layer electrophoresis**
- 37. Environmental analysis**
 - a) General papers
 - b) Air pollution
 - c) Water pollution
 - d) Soil pollution
- 38. Chiral separations**

XX. (abstract number underlined) refers to HPTLC related publication or application using HPTLC materials

1. Reviews and books

- 104 001 Tara MCGLINCHEY*, P.A. RAFTER, Fiona REGAN, D. GILLIAN, P. MCMAHON (*Department of Agriculture, Fisheries & Food, Central Meat Control, Backweston Laboratory Complex, Youngs Cross, Celbridge, Co., Kildare, Ireland): A review of analytical methods for the determination of aminoglycoside and macrolide residues in food matrices. *Anal. Chim. Acta* 624 (1), 1-15 (2008). Aminoglycosides and macrolides are important antibiotics for veterinary medicine and are widely used in the treatment of bacterial disease, and as feed additives for growth promotion. As a result the European commission set strict criteria for monitoring residues and requires testing for low levels of aminoglycosides and macrolides in foods. Therefore the development of fast, reliable, and sensitive methods for the extraction and subsequent analysis of these antibiotics is of great interest. The review discusses analytical methods for both extraction and determination of antibiotics in various food matrices focusing on the last 10 years. Extraction and clean-up methods such as deproteinization and solid-phase extraction are described, and various screening methods including TLC, EI, CE, microbiological assays, and LC combined with MS are reviewed. agricultural, food, analysis, HPTLC, review 1, 28

2. Fundamentals, theory and general

- 104 002 Tatjana DJAKOVIC-SEKULIC*, Nada PERISIC-JANJIC, Evgenija DJURENDI (*Department of Chemistry, Faculty of Sciences, University of Novi Sad, Trg Dositeja Obradovi a 3, 21000 Novi Sad, Serbia): Retention data from reverse-phase high-performance thin-layer chromatography in characterization of some bis-salicylic acid derivatives. *Biomed. Chromatogr.* 23 (8), 881-887 (2009). HPTLC investigation of salicylic acid derivatives on RP-phase with various methanol - water and dioxane - water binary mixtures in order to establish relationships between chromatographic data and selected physico-chemical parameters that are related to ADME (absorption, distribution, metabolism and elimination). A linear correlation between RM values and the volume fraction of mobile phase modifier was observed. The obtained RM0 values were correlated with lipophilicity, solubility, human intestinal absorption, plasma-protein binding, and blood-brain barrier data. HPTLC, QSRR, lipophilicity 2
- 104 003 L. KOMSTA*, K. SZTANKE, P. MACZKA, M. UCHEREK, R. SKIBINSKI, A. GUMIENICZEK (*Department of Medicinal Chemistry, Faculty of Pharmacy, Medical University of Lublin, Jaczewskiego 4, 20-090 Lublin, Poland; lukasz.komsta@am.lublin.pl): Determination of the lipophilicity of twenty-seven imidazo[2,1-c][1,2,4]triazine derivatives with strong biological activity by reversed-phase TLC. Comparison with results obtained by use of computational algorithms *J. Planar Chromatogr.* 22, 327-331 (2009) TLC of 27 novel imidazo[2,1-c][1,2,4]triazine derivatives together with 12 compounds with known literature log P values (as reference calibration data) on RP-18 with methanol - water binary mobile phases containing different proportions of methanol in horizontal chambers without chamber saturation. Detection under UV 254 nm. Using principal-component analysis the obtained results were compared with results from nine computational methods. pharmaceutical research, qualitative identification 2c
- 104 004 T. SAMUEL*, A. MANIKANDAN, A. SINGH (*CGPSG College of Pharmacy, Peelamedu, Coimbatore, India): Standardisation of herbal medicine by high-performance thin-layer chromatography. Abstract No. 9923, IHCB (2009). HPTLC of several herbal formulations (tablets extracted with methanol) on silica gel with toluene - ethyl acetate 19:1. The fingerprint method was suitable for correct identification and for routine quality control of the herbal extracts. pharmaceutical research quality control, herbal, HPTLC 2a
- 104 005 M. WAKSMUNDZKA-HAJNOS, D. MATOSIUK, A. PETRUCZYNIK, U. KIJKOWSKA-MURAK* (*Medical University of Lublin Department of Inorganic Chemistry 20-081 Lublin Poland): Determination of the lipophilicity of selected isoquinoline alkaloids by RP-TLC. *Acta Chromatographica* 24 (4), 563-573 (2008). TLC of nine alkaloids on RP-18 with mixtures of

aqueous acetone or aqueous dioxane with various mobile phase additives (ammonia, diethylamine, or tetrabutylammonium chloride) in order to suppress ionization of the alkaloids and/or reduce ionic interactions with surface silanol groups. Ion-pair TLC on RP-18 with aqueous acetone mixtures and various mobile phase additives (pentane sulfonic acid, octane sulfonic acid, or di-(2-ethylhexyl)orthophosphoric acid). Investigation of relationships between RM values and modifier concentration using a linear semilogarithmic equation for experimental data to calculate lipophilicity values RMW (RM for pure water), the slope, and the intercept with the x-axis. Comparison with retention data of standards with known lipophilicity (log P).

doping

2d, 22

3. General techniques

- 104 006 V.G. BEREZKIN*, N.Y. KULAKOVA, S.S. KHREBTOVA (*A. V. Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, Leninsky pr. 29, Moscow, 119991, Russia, berezkin@ips.ac.ru): Three-dimensional thin-layer chromatography on plates with open and closed adsorption layers. *J. Planar Chromatogr.* 22, 313-319 (2009). Presentation of two different methods of three-dimensional TLC using plates with open and sealed (closed) adsorption layers. In the suggested method the components of the test dye mixture initially migrate in the first direction, then in the second direction (different from the first) and in the third direction (different from first and second). TLC of dye mixture 1 (crystal violet, xylen cyanol, neutral blue, bromothymol dark blue, methanyl yellow, acridine orange, indophenol, ariabel red, Sudan blue II, Sudan IV, dimethylaminoazobenzene) with ethanol - acetic acid 10:1 (1D), acetone (2D), and toluene (3D) and of mixture 2 (dark violet, bright orange, yellow, dark red, violet) on silica gel with tetrahydrofuran - benzene 9:1 (1D), dichloromethane - benzene 3:1 (2D), and toluene (3D).
comparison of methods 3d

- 104 007 X. LIU*, T. KUBO, H. DIAO, J. BENJAMAS, T. YONEMICHI, N. NISHI (*College of Materials and Textiles, Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University, Xiasha Higher Education Zone, Hangzhou 310018, China): DNA/polyvinyl alcohol interpenetrating polymer network as stationary phase for thin-layer chromatography. *Anal. Biochem.* 393 (1), 67-72 (2009). A DNA/polyvinyl alcohol interpenetrating polymer network was produced by cross-linking polyvinyl alcohol with glutaraldehyde and subsequent cross-linking of DNA by UV irradiation. The polymer was then used to coat the surface of porous silica particles for TLC. Three typical DNA-binding compounds and eight amino acid enantiomers were used as model chemicals to investigate the chromatographic behavior of the modified TLC phase. Both classes of chemicals provided high separation efficiency. DNA-modified TLC phases could be used for various application fields, including efficacy evaluation of a medicine, toxicity assessment of a pollutant at the molecular level, as well as separation of enantiomers of dyes, amino acids, peptides, proteins, nucleotides, and drugs.
stationary phase 3b

- 104 008 S. WANGTHONG*, I. TONSIRIPAKDEE, T. MONHAPHOL, R. NONTHABENJAWAN, S. PATTANAARGSON WANICHWECHARUNGRUANG (*Department of Chemistry, Faculty of Science, Chulalongkorn University, Payatai, Bangkok 10330, Thailand): Post TLC developing technique for tyrosinase inhibitor detection. *Bio. Chromatogr.* 21(1), 94-100 (2008). The method for detection of tyrosinase inhibiting substances involves spraying of the TLC layer with tyrosinase and L-tyrosine solutions successively. Positive results are detected as white spots against a brownish-purple background. The method is suitable either as a quick screening method for tyrosinase inhibitor detection or as a guiding procedure for an isolation of tyrosinase inhibitors from mixtures or natural product extracts. The technique is sensitive enough for results in the presence of 6 ng/zone glabridin.
review, postchromatographic derivatization 3d

4. Special techniques

- 104 009 P. ABU-RABIE*, N. SPOONER (*PreClinical Development Drug Metabolism and Pharmacokinetics, GlaxoSmithKline Research and Development, Park Road, Ware, Hertfordshire, SG12

ODP, United Kingdom; Paul.2.Abu-Rabie@gsk.com): Direct quantitative bioanalysis of drugs in dried blood spot samples using a thin-layer chromatography mass spectrometer interface. *Anal. Chem.* 81, 10275-10284 (2009). Direct quantitative bioanalysis of drugs from dried blood spot samples using a TLC-MS interface with or without HPLC separation. The method gave acceptable sensitivity, linearity, accuracy, and precision data for bioanalytical validations. The direct elution technique was shown to increase assay sensitivity for a range of analytes. Investigations were performed to optimize extraction time, minimize sample-to-sample carry-over, and compare chromatographic performance. On the basis of this preliminary assessment, it has been demonstrated that this TLC-MS interface has the potential to be an effective tool for the direct analysis of drugs in dried blood samples at physiologically relevant concentrations.

clinical chemistry research

4e

- 104 010 S.-C. CHENG (Cheng Sy-Chyi), M.Z. HUANG (Huang Min-Zong), J. SHIEA* (Shiea Jentaie) (*National Sun Yat-Sen University-Kaohsiung Medical University Joint Research Center, Kaohsiung, 804, Taiwan; jetea@mail.nsysu.edu-tw): Thin-layer chromatography/laser-induced acoustic desorption/electrospray ionization mass spectrometry. *Anal. Chem.* 81, 9274-9281 (2009). TLC of dye standards (FD&C Green No. 3, FD&C Red No. 3, eriochromcyanin R), drug standards (3,4-methylene-dioxy-N-methamphetamine, lysergic acid diethylamide, flunitrazepam), and rosemary essential oil on 1) RP-18 with 65 % acetone containing 1.5 % formic acid and 2) on silica gel with chloroform - methanol 9:1. Rosemary essential oil was also separated on silica gel with toluene - ethyl acetate 9:1. Detection under UV 254 nm and in daylight. The combination of laser-induced acoustic desorption and electrospray ionization mass spectrometry (LIAD/ESI/MS) can be used to rapidly characterize chemical compounds separated on a TLC plate.

qualitative identification

4e

- 104 011 N. GOTO-INOUE*, T. HAYASAKA, T. TAKI, Tania VALDES GONZALEZ, M. SETOU (*Department of Molecular Anatomy, Hamamatsu University School of Medicine, Hamamatsu, Shizuoka 431-3192, Japan): A new lipidomics approach by thin-layer chromatography-blot-matrix-assisted laser desorption/ionization imaging mass spectrometry for analyzing detailed patterns of phospholipid molecular species. *J. Chromatogr. A* 1216 (42), 7096-7101 (2009). Presentation of a TLC-blot-MALDI-IMS method which combines TLC and IMS (imaging mass spectrometry) for use in lipidomics. In comparison with common staining methods the method allows highly sensitive detection of whole lipids and individual molecular species. Linearity for all lipids ranged approximately over one order of magnitude. Precision (% RSD) was <16 %. The TLC step allows precise separation of complex lipid mixtures into individual lipid classes before MS analysis is performed.

qualitative identification

4e

- 104 012 E. HARRY, J. REYNOLDS, A. BRISTOW, I. WILSON, C. CREASER* (*Centre for Analytical Science, Department of Chemistry, Loughborough University, Loughborough LE11 3TU, UK, c.s.creaser@lboro.ac.uk): Direct analysis of pharmaceutical formulations from non-bonded reverse-phase thin-layer chromatography plates by desorption electrospray ionisation ion mobility mass spectrometry. *Rapid Commun. Mass Spectrom.* 23, 2597-2604 (2009). RP-TLC of pharmaceutical formulations containing paracetamol, ephedrine, codeine, and caffeine on hydrocarbon-impregnated silica with methanol - water 1:1. Detection by desorption electrospray ionization (DESI) combined with ion mobility mass spectrometry (IM-MS). The limit of detection was 9, 16, 34, 239 and 225 µg/cm² for codeine, caffeine, ephedrine and paracetamol, respectively.

pharmaceutical research, quality control, qualitative identification

4e

- 104 013 L. KOMSTA (Department of Medicinal Chemistry, Medical University of Lublin, Jaczewskiego 4, 20-090 Lublin, Poland): A comparative study on several algorithms for denoising of thin-layer densitograms. *Anal. Chim. Acta* 641 (1-2), 52-58 (2009). Comparison of classical filtering techniques (Savitzky-Golay, Adaptive Degree Polynomial Filter, Fourier denoising, Butterworth and Chebyshev IIR filters) and wavelet shrinkage (31 mother wavelets, 3 thresholding techniques and

11 decomposition levels) with the original noisy signal and a reference signal which was denoised experimentally by averaging 64 measurements. The best similarity to the reference signal was obtained with filters, however the signal was slightly oversmoothed. The wavelet shrinkage method gave less denoised signals. There was a significant influence of the thresholding technique and decomposition level, and best conditions were at level 2 or 3 and soft thresholding), whereas changing of the mother wavelet almost did not change the result. The presented results can be used as general recommendations for denoising densitometric fingerprints before applying further chemometric algorithms. The best choices were: Savitzky-Golay filter of appropriate window width (optimized against autocorrelation) or wavelet shrinkage with Haar wavelet, soft thresholding and high decomposition level.

HPTLC, quantitative analysis, qualitative identification, densitometry 4c

- 104 014 F. SCHULTE, J. MAEDER, L. W. KROH, U. PANNE, Janina KNEIPP* (*Chemistry Department, Humboldt Universität zu Berlin, Brook-Taylor-Strasse 2, 12489 Berlin, Germany; janina.kneipp@chemie.hu-berlin.de): Characterization of pollen carotenoids with in situ and high-performance thin-layer chromatography supported resonant Raman spectroscopy. *Anal. Chem.* 81, 8426-8433 (2009). HPTLC of zeaxanthin, cryptoxanthin, beta-carotene, lutein and pollen extracts on silica gel with tetrahydrofuran - methylene chloride - *n*-hexane by automated multiple development. Quantitative determination by absorbance measurement at 425 nm, which has to be accomplished within 5 min after development due to the fast bleaching of the carotenoid color. The analysis of carotenoids in pollen extracts was confirmed by resonance Raman data measured directly on the HPTLC plates.

HPTLC, AMD, densitometry, quantitative analysis, biochemical research 4e, 30b

- 104 015 M. SHARIATGORJI, Z. SPACIL, G. MADDALO, L. CARDENAS, L. ILAG* (*Department of Analytical Chemistry, Stockholm University, 106 91 Stockholm, Sweden, Leopold.ilag@anchem.su.se): Matrix-free thin layer chromatography/laser desorption ionization mass spectrometry for facile separation and identification of medicinal alkaloids. *Rapid Commun. Mass Spectrom.* 23, 3655-3660 (2009). TLC of berberine and palmatine in roots of *Berberis barandana* on silica gel with butanol - acetic acid - water 14:3:4. Detection under UV 366 nm. Bands were cut out for further analysis by laser desorption ionization mass spectrometry. The hR_F value of berberine and palmatine was 56 and 46, respectively.

herbal, qualitative identification 4e

- 104 016 C. SIMOES-PIRES, B. HMICHA, A. MARSTON, K. HOSTETTMANN* (*Laboratory of Pharmacognosy and Phytochemistry, University of Geneva, 1211 Geneva 4, Switzerland, kurt.hostettmann@unige.ch): A TLC bioautographic method for the detection of alpha- and beta-glucosidase inhibitors in plant extracts. *Phytochem. Anal.* 20, 511-515 (2009). Bioautography of alpha-D-glucosidase (1) and beta-D-glucosidase (2) in buffer solution (sodium acetate 4.1 % in water pH=7.5) sprayed onto a silica gel plate. Incubation at room temperature for 60 min for (1) and at 37 °C for 20 min for (2). For detection of the active enzyme, solutions of 2-naphthyl-alpha-D-glucopyranoside or 2-naphthyl-beta-D-glucopyranoside and Fast Blue salt were mixed at a ratio of 1:1 for (1) or 1:4 for (2), and sprayed onto the plate to give a purple background coloration after 2-5 min. Methanol extracts of the aerial parts of *Tussilago farfara* and *Urtica dioica* were tested as enzyme inhibitors and visualized as white spots on the TLC plates.

pharmaceutical research, herbal, qualitative identification, AMD, bioautography 4e

- 104 017 G. STUBIGER, E. PITTENAUER, O. BELGACEM, P. REHULKA, K. WIDHALM, G. ALLMAIER* (*Institute of Chemical Technologies and Analytics, Vienna University of Technology, 1060 Vienna, Austria, guenter.allmaier@tuwien.ac.at): Analysis of human plasma lipids and soybean lecithin by means of high-performance thin-layer chromatography and matrix-assisted laser desorption/ionization mass spectrometry. *Rapid Commun. Mass Spectrom.* 23, 2711-2723 (2009). HPTLC in combination with matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS) was used for the analysis of complex lipid mixtures. For the separation of

lipids one-dimensional HPTLC on silica gel aluminum foil was used with a two-phase mobile phase. The combination with MALDI-MS allowed the identification of 70 distinct lipid species and the analysis of even minor lipid classes from only very small volumes of human plasma (50 µL).

pharmaceutical research, clinical chemistry research, food analysis, HPTLC, quantitative analysis

4e

5. Hydrocarbons and halogen derivatives

104 018 I. BELAI*, G. OROS, B. BORDAS (*Plant Protection Institute, Hungarian Academy of Sciences, 1525 Budapest, P.O. Box 102, Hungary; ibel@nki.hu): Quantitative structure-retention relationship and 3D molecular modeling studies in the unusual chromatographic behavior of triphenylmethane derivatives in RPTLC systems. *J. Planar Chromatogr.* 22, 255-263 (2009). TLC of 25 triphenylmethane derivatives using paraffin oil-coated silica gel and acetone-water mixtures, in which the acetone content varied between 40 and 70 % in increments of 10 %. Dual retention behavior is observed for triphenylmethane derivatives in reversed phase HPTLC when the composition of acetone-water mobile phase is varied. The physicochemical and molecular properties of triphenylmethane derivatives lead to an unusual retention behavior, which was investigated by traditional quantitative structure-retention relationship modeling and by 3D molecular modeling. Lipophilicity was found to be the most important molecular property governing the retention of triphenylmethane derivatives.

qualitative identification

5b

8. Substances containing heterocyclic oxygen

104 019 H.M. KURTBAY, I. KAYNAK, S. S. BOZKURT, M. MERDIVAN* (*Department of Chemistry, Dokuz Eylul University, Faculty of Science and Arts, 35160 Izmir, Turkey; melek.merdivan@deu.edu.tr): Densitometric HPTLC analysis of the 5-hydroxymethylfurfural content of Turkish fruit wines and vinegars. *J. Planar Chromatogr.* 22, 363-366 (2009). HPTLC of 5-hydroxymethylfurfural in seven Turkish fruit wines and three Turkish vinegars on silica gel with toluene - ethyl acetate - 90 % formic acid 6:3:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 286 nm. The limit of detection and quantification was 0.05 and 0.13 ng/mL, respectively.

food analysis, toxicology, HPTLC, densitometry, quantitative analysis

8b

9. Oxo compounds, ethers and epoxides

104 020 Petra JAZBEC*, A. SMIDOVNIK, M. PUKLAVEC, M. KRIZMAN, J. SRIBAR, L. MILIVOJEVIC, M. PROSEK (*Department of Food Chemistry, National Institute of Chemistry, Ljubljana, Slovenia; Petra.jazbec@ki.si): HPTLC and HPLC-MS quantification of coenzyme Q10 and cholesterol in fractionated chicken-breast tissue. *J. Planar Chromatogr.* 22, 395-398 (2009). HPTLC of coenzyme Q10, cholesterol and biological extracts on silica gel with petroleum ether - diethyl ether - acetic acid 85:15:1. Detection by dipping into 5 % phosphomolybdic acid in ethanol for 10 s followed by heating for 10 min at 110 °C. Quantitative evaluation in visible light.

food analysis, HPTLC, quantitative analysis, densitometry, biochemical research

9, 20

10. Carbohydrates

104 021 T. BERNARDI, Elena TAMBURINI* (*Department of Chemistry, University of Ferrara, Via L. Borsari 46, 44100 Ferrara, Italy; tme@unife.it): An HPTLC-AMD method for understanding the metabolic behavior of microorganisms in the presence of mixed carbon sources. The case of *Bifidobacterium adolescentis* MB 239. *J. Planar Chromatogr.* 22, 321-325 (2009). HPTLC of glucose, fructose, galactose, lactose, raffinose, 1-kestose, nystose, and fructosyl-nystose on diol phase by automated multiple development with acetonitrile - acetone - water and acetonitrile - water. Preliminary isocratic developments were performed at 22-25 °C at 65-75 % relative humidity in a twin trough chamber. Detection by vaporization with 37 % hydrochloric acid vapor for 30 min followed by dipping in 4-aminobenzoic acid. Quantitative determination by fluorescence measurement at 313 nm.

HPTLC, densitometry, quantitative analysis, AMD, biochemical research

10a

- 104 022 J. STROKA*, I. DONCHEVA, B. SPANGENBERG (*European Commission Joint Research Centre, Institute for Reference Materials and Measurements, Food Safety and Quality Unit, Reetieweg 111, 2440 Geel, Belgium; joerg.stroka@ec.europa.eu): Determination of sucralose in soft drinks by high-performance thin-layer chromatography: Interlaboratory study. *J. AOAC Int.* 92, 1153-1159 (2009). HPTLC of sucralose on amino phase with acetonitrile - water 4:1 in a horizontal or standard development chamber without chamber saturation. For detection the plate was heated at 190 °C for 20 min, either in a drying oven or on a temperature-controlled heating plate. Quantitative determination by absorbance and fluorescence measurement at 254 nm. The results of the interlaboratory comparison show good precision characteristics. The fluorescence measurements of the sucralose derivatives indicated better method performance, compared with absorbance measurements in the UV.

food analysis, quality control, HPTLC, quantitative analysis, densitometry

10a

11. Organic acids and lipids

- 104 023 Ritu ARORA*, B. SINGH, R. SINGH, C. KATIYAR (*Guru Nanak Dev University, Amritsar, Punjab, India): Validated HPTLC method for the determination of cinnamic acid in crude plant materials, herbal extracts and pharmaceutical dosage forms containing *Cinnamomum cassia*. 60th Indian Pharmaceutical Congress PA-214 (2008). HPTLC of cinnamic acid (in plant raw material, herbal extracts and pharmaceutical dosage forms) on silica gel with chloroform - methanol 4:1 in a saturated twin trough chamber at room temperature (25 °C). Quantitative determination by absorbance measurement at 277 nm. The hR_F value for cinnamic acid was 50. The method was linear in the range of 700-1400 ng/spot.

pharmaceutical research, herbal, HPTLC, densitometry, quantitative analysis

11a

- 104 024 F. HASAN*, R. KHAR, F. AHMAD, M. ALI, M. RAZA (*Dept. of Pharmaceutics, Jamia Hamdard, Hamdard Nagar, New Delhi, India): Development of novel high-performance thin-layer chromatography method for estimation of lipid in egg oil. Abstract No. F-282, 61st (2009). HPTLC of lipids on silica gel in a saturated twin trough chamber with carbon tetrachloride - methanol - acetic acid 270:30:11 at room temperature (25 °C). Quantitative determination by fluorescence measurement at 366 nm. The hR_F value of cholesterol was 35. The calibration curve showed good linear relationship with $r^2=0.999$ in the range of 100-600 ng/spot (via peak area).

pharmaceutical research, quality control, HPTLC, quantitative analysis

11c

- 104 025 P. HAZAM*, D. SARKAR, B. DEY, S. DAS (*Himalayan Pharmacy Institute, Dept. of Pharmacognosy, Majhotar, Sikkim, India: A high-performance thin-layer chromatographic (HPTLC) method for the estimation of rosmarinic acid from *Salvia officinalis* (sage). 60th Indian Pharmaceutical Congress PG-256 (2008). HPTLC of rosmarinic acid in *Salvia officinalis* on silica gel with toluene - ethyl acetate - formic acid 5:4:1. Quantitative determination by absorbance measurement at 328 nm. Linearity was 0.1-1.0 ng/mL, recovery was 99.4 %. The method was found suitable for routine quality control of the *Salvia officinalis* raw material.

herbal, environmental, densitometry, HPTLC, quantitative analysis

11a

- 104 026 Y. KAWAI*, M. MIYOSHI, J. MOON, J. TERAOKA (*Department of Food Science, Graduate School of Nutrition and Biosciences, The University of Tokushima, Tokushima 770-8503, Japan): Detection of cholesteryl ester hydroperoxide isomers using gas chromatography-mass spectrometry combined with thin-layer chromatography blotting. *Anal. Biochem.* 360 (1), 130-137 (2007). Determination of cholesteryl ester hydroperoxides, especially cholesteryl linoleate hydroperoxide isomers, by combination of TLC blotting with diphenyl-1-pyrenylphosphine fluorescent detection (DPPP-TLC blotting) and GC-electron ionization-MS (GC-EI-MS). After DPPP-TLC blotting of cholesteryl ester hydroperoxides fluorescent zones were obtained which were extracted and derivatized by hydrogenation, transmethylation, and trimethylsilylation. The resulting methyl ester/trimethylsilyloxy derivatives of hydroxyoctadecenoic acid were then analyzed by GC-EI-MS using selected ion monitoring of isomer-specific fragment ions originating from the alpha-cleavage of the trimethylsilyloxy group.

agricultural, clinical routine analysis, postchromatographic derivatization,
qualitative identification, quantitative analysis 11

- 104 027 H. LI (Li Haixing)*, T. QIU (Qiu Ting), Y. CAO (Cao Yusheng), J. YANG (Yang Jiyan), Z. HU-ANG (Huang Zhibing) (*Sino-German Joint Research Institute, Nanchang University, Nanchang 330047, China): Pre-staining paper chromatography method for quantification of gamma-aminobutyric acid. *J. Chromatogr. A* 1216 (25), 5057-5060 (2009). Paper chromatography of gamma-aminobutyric acid. The method consists of application, separation and detection and is clean, rapid, inexpensive and reproducible compared to the routine paper chromatography. The derivatization procedure with ninhydrin reagent was optimized regarding reagent concentration, derivatization temperature and time and Cu²⁺ concentration. Quantification of gamma-aminobutyric acid by combination of with vis spectrophotometry. The limit of detection was 0.05 mg/mL and the linear range was from 0.5 to 20.0 mg/mL. The determination coefficient was $r^2 = 0.998$. The method was accurate ($\%RSD < 2.6 \%$), and recoveries were 102.7-103.9 %.

quality control, traditional medicine, pharmaceutical research, quantitative analysis,
qualitative identification, paper chromatography 11a

- 104 028 N. MANJU*, A. BINDU, N. ALEYKUTTY, J. SAJAN (*Department of Pharmaceutical Sciences, Cheruvandoor Campus, Ettumanoor, Kottayam, Kerala, India): Development of HPTLC method for quantification of gallic acid from the fruits of *Phyllanthus emblica* L. 60th Indian Pharmaceutical Congress PG-265 (2008). HPTLC of gallic acid in the ethyl acetate fraction of fruits of *Phyllanthus emblica* on silica gel with toluene - ethyl acetate - formic acid - methanol 30:30:8:2. Detection and quantitative determination of gallic acid by absorbance measurement at 280 nm. The proposed HPTLC method provided a good resolution of gallic acid from other constituents present in the ethyl acetate fraction of fruits of *Phyllanthus emblica* and can be used for the quantification of gallic acid.

herbal, HPTLC, densitometry, quantitative analysis 11a

- 104 029 D. MUKHERJEE*, T. BARMAN, S. RAJA, P. MUKHERJEE (*School of Natural Product Studies, Jadavpur University, Kolkata 700032, India): Estimation of betulinic acid in *Nelumbo nucifera* (Nymphaeaceae) rhizome and seed extract by validated HPTLC method. Abstract No. 9192, IHCB (2009). HPTLC of betulinic acid in hydro alcoholic extracts of *Nelumbo nucifera* rhizome and seed, on silica gel with chloroform - methanol - formic acid 49:1:1. Detection by spraying with anisaldehyde reagent. Quantitative determination by absorbance measurement at 420 nm. The method was linear in the range of 2-10 ng/spot, recovery was 98.1-98.4 %.

pharmaceutical research, quality control, herbal, HPTLC, densitometry,
quantitative analysis, postchromatographic derivatization 11a

- 104 030 B. NIMAVAT*, D. MOVALIA, S. MISHRA, H. TANK (*S. J. Thakkar Pharmacy College, Sausrashtra University, Rajkot, Gujarat, India): Development of validated HPTLC method for quantitative estimation of oleanolic acid as marker in total methanolic extract of fruits of *Randia dumetorum* lam. 60th Indian Pharmaceutical Congress PA-217 (2008). HPTLC of oleanolic acid in total methanolic extract of fruits of *Randia dumetorum* lam. on silica gel with toluene - ethyl acetate - glacial acetic acid 70:30:1 in a twin trough chamber saturated for 10 min. Detection by treatment with 10 % sulphuric acid in methanol, followed by heating at 110 °C and immediate densitometric evaluation. Quantitative determination by absorbance measurement at 540 nm. The method was linear in the range of 50-500 ng/spot. Recovery was in the range of 99.4-100.8 %.

pharmaceutical research, traditional medicine, herbal, HPTLC, quantitative analysis 11a

- 104 031 P. PATEL*, R. SHAH, S. PATEL, Unnati SHAH (*Pharmanza Herbal Pvt. Ltd., Plot No. 214, Kania 388435, Gujarat, India; and Lachoo Memorial College of Science & Technology, Jodhpur, India): Method development and validation for the estimation of (-)hydroxy citric acid in fruits of *Garcinia gummigutta* D. by HPTLC. Abstract No. 9191, IHCB (2009). HPTLC of (-)hydroxy

citric acid (the main constituent of *Garcinia gummigutta* fruits) on silica gel with *n*-propanol - water - glacial acetic acid 50:50:1. Quantitative determination by absorbance measurement at 210 nm. The hR_F value was 46. The method was linear in the range of 100-1000 ng/spot, recovery was 99.8-100.9 %.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis

11a

- 104 032 B. RAJ*, Salma KHANAM, L. JOSEPHINE (*Dept. of Pharmacognosy, Dayanand Sagar College of Pharmacy, Bangalore 560078, India, bincyraj@yahoo.com): HPTLC method for estimation of ursolic acid in *Ocimum sanctum* extract. *Asian J. of Chem.* 21(9), 6999-7004 (2009). HPTLC of ursolic acid in methanolic and aqueous extracts of *Ocimum sanctum* (Tulsi) on silica gel with toluene - ethyl acetate - acetic acid 55:45:2 with chamber saturation. Detection by treatment with Liebermann Burchard's reagent. Quantitative determination by absorbance measurement at 550 nm. The method was linear in the range of 100-400 ng/band. The amount of ursolic acid in aqueous and methanolic extracts was in the range of 0.2 and 0.4 %. Samples collected from different geographical regions were compared, samples from Kerala contained the highest levels of ursolic acid.

pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 11a

- 104 033 B. RAJ*, Salma KHANAM, S. JOHN, J. JENITA, Ayesha SIDDIQUA (*Dept. of Pharmacognosy, Dayananda Sagar College of Pharmacy, Kumaraswamy Layout, Bangalore 560078, India): Comparative HPTLC analysis of oleanolic acid in *Ocimum sanctum* collected from different geographical sources. Abstract No. 9226, IHC (2009). HPTLC of oleanolic acid (in extracts of *Ocimum sanctum* collected from different geographical sources) on silica gel with toluene - ethyl acetate - glacial acetic acid 55:45:2. Detection by spraying with Liebermann-Burchard's reagent. Quantitative determination by absorbance measurement at 600 nm. Linearity was in the range of 1-4 µg, recovery was 100.4-102.1 %. Hydro alcoholic extracts of the plant contained high amounts of oleanolic acid, maximum contents were found in plants collected in the Kerala region.

pharmaceutical research, quality control, herbal, densitometry, HPTLC, quantitative analysis

11a

- 104 034 M. SAJEWICZ, D. KRONENBACH, M. GONTARSKA, M. WRÓBEL, R. PIETKA, Teresa KOWALSKA* (*Institute of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland; kowalska@us.edu.pl): TLC in a search for structural limitations of spontaneous oscillatory in-vitro chiral conversion. Alpha-hydroxybutyric and mandelic acids. *J. Planar Chromatogr.* 22, 241-248 (2009). TLC of alpha-hydroxybutyric acids and mandelic acid on silica gel, prewashed with methanol - water 9:1 and impregnated by dipping for 2 s in 0.05 g/L aqueous copper acetate solution, with dioxane - water 9:1 at 22 +/- 2°C. Quantitative determination by absorbance measurement during 16 days at 326 nm. Spontaneous oscillatory in-vitro chiral conversion was observed for alpha-substituted propionic acids as well as chiral carboxylic acids with two and four carbon atoms.

qualitative identification, densitometry

11a

13. Steroids

- 104 035 R. XIA (Xia Rui)*, SH. DONG (Dong Shuying), B. CHE (Che Baoquan) (*Beijing Municip. Inst. Drug Cont., Beijing 100035, China): (Rapid identification of glucocorticoid in Chinese traditional medicinal formulations by thin-layer chromatography) (Chinese). *Chinese J. Pharm. Anal.* 28 (3), 470-471 (2008). TLC of glucocorticoid in TCM formulations extracted with chloroform - methanol 9:1 on silica gel with dichloromethane - diethyl ether - methanol - water 385:60:15:2. Detection by spraying with a solution of tetrazolium blue chloride. Successful separation and identification of five glucocorticoids.

doping, pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification

13

- 104 036 B. YUAN (Yuan Baoqiang)*, L. Yang (Yang Lahu) (*Fuzhou Municip. Inst. Drug Cont., Fuzhou, Fujian 350001, China): (Analysis of dexamethasone acetate and the related compounds in the tablets) (Chinese). Chinese J. Pharm. Anal. 27 (3), 412-413 (2007). TLC of dexamethasone acetate on silica gel with dichloromethane - methanol 9:1. Detection under UV 254 nm.

pharmaceutical research, quality control, qualitative identification, quantitative analysis

13

15. Terpenes and other volatile plant ingredients

- 104 037 B. CHENGAIHAH*, B. PRATAP, M. ALAGUSUNDARAM, M. RUTHU, V. SAROVAR REDDY (*Annamacharya College of Pharmacy, Rajampet, Kadapa, A.P., India): Identification of terpenes in stem of *Ocimum basilicum* Linn by HPTLC technique. 60th Indian Pharmaceutical Congress PA-221 (2008). HPTLC of terpenes in stem of *Ocimum basilicum* on silica gel with toluene - ethyl acetate 19:1. Evaluation of 5 spots under UV 254 nm and under visible light after treatment with anisaldehyde reagent followed by heating at 100 °C for 5 min: borneol (yellow, hR_F 24), menthol (red, hR_F 27), eugenol (blue, hR_F 50), thymol (violet, hR_F 57), safrole (blue, hR_F 93).

traditional medicine, herbal, HPTLC, qualitative identification

15a

17. Amines, amides and related nitrogen compounds

- 104 038 A. ALPMANN, Gertrud MORLOCK* (*University of Hohenheim, Institute of Food Chemistry, Garbenstrasse 28, 70599 Stuttgart, Germany; gmorlock@uni-hohenheim.de): Rapid and cost-effective determination of acrylamide in coffee by planar chromatography and fluorescence detection after derivatization with dansulfinic acid. J. AOAC Int. 92, 725-729 (2009). HPTLC of acrylamide extracted from coffee samples on silica gel with ethyl acetate - tert. butyl methyl ether 4:1 in a twin trough chamber or automatic developing chamber. Pre-chromatographic in situ derivatization of the extracts (applied as area) by overspraying with dansulfinic acid produced the fluorescent dansylpropanamide band. Quantitative determination by fluorescence measurement at 254/>400 nm. The limit of quantification was 48 µg/kg. The linearity over the whole procedure showed determination coefficients between 0.9995 and 0.9825 ($n = 6$). The within-run precision (% RSD, $n = 6$) of the chromatographic method was 3%. Commercial coffee samples analyzed showed acrylamide contents between 52 and 191 µg/kg, which was in correlation with amounts reported in publications.

food analysis, toxicology, quality control, HPTLC, quantitative analysis, densitometry

17c

18. Amino acids and peptides, chemical structure of proteins

- 104 039 J.D. VASTA, M. CICCHI, J. SHERMA*, B. FRIED (*Lafayette College, Department of Chemistry, Easton PA 18042-1782 USA): Evaluation of thin-layer chromatography systems for analysis of amino acids in complex mixtures. Acta Chromatographica 21 (1), 29-38 (2009). Evaluation of different TLC systems for analysis of 21 amino acids in biological tissues and fluids. Detection by derivatization with ninhydrin reagent, and determination of R_F values by slit-scanning densitometry. The five most suitable systems were cellulose and silica gel plates developed with either 2-butanol - pyridine - acetic acid - water 39:34:10:26 or 2-butanol - pyridine - 25 % ammonia - water 39:34:10:26, and ion exchange plates developed with citrate buffer of pH 3.3. Detection with ninhydrin allowed the identification of all amino acids except for leucine and isoleucine in complex mixtures. Quantification is possible as well if the amino acid of interest is well separated from adjacent components of the mixture. The method is illustrated with example chromatograms on cellulose HPTLC layer showing the identification and separation of amino acids in snail tissue samples.

clinical chemistry research, HPTLC, quantitative analysis, qualitative identification, densitometry

18a

- 104 040 M. VEGA*, M. ARANDA (*University of Concepcion, Faculty of Pharmacy, Department of Food Science, Nutrition and Dietetic, Barrio Universitario s/n Casilla 237, PO 403-0249 Concepcion, Chile; mveha@udec.cl): Determination of available lysine by planar chromatography: A useful tool for protein quality evaluation in fish feed. J. AOAC Int. 92, 699-702 (2009). HPTLC of a dinitrophenyl-lysine derivative (produced by incubation with 1-fluoro-2,4-dinitrobenzene and hydrolyzation with hydrochloric acid) on silica gel (prewashed with methanol) with n -pro-

panol - 25 % ammonia 7:3 in a twin trough chamber. Quantitative determination by absorbance measurement at 360 nm. The method was linear ($r^2 = 0.9991$) in the range from 12.5 to 125.0 ng/band. Repeatability (%RSD) and intermediate precision (%RSD) in matrix were 0.8 % and 3.6 %, respectively. Recoveries of spiked samples at two levels ranged from 72 to 85 % with RSD from 3 to 8%. This method is a reliable, high throughput and low cost analytical method for the salmon feed industry.

food analysis, quality control, HPTLC, densitometry, quantitative analysis 18a

20. Enzymes

104 020 Petra JAZBEC et al., see section 9

22. Alkaloids

104 041 K. DHALWAL*, V. SHINDE, K. MAHADIK (* Dept. of Pharmacognosy and Phytochemistry, Poona College of Pharmacy, Bharathi Vidyapeeth University, Erandwane, Pune 411038, India): Effective and sensitive methods for quantitative analysis of alkaloids in sida species by using HPLC and HPTLC. Abstract No. 9402, IHC (2009). HPLC and HPTLC methods were developed for the simultaneous estimation of vasicine and vasicinone in *Sida cordifolia* and *Sida acuta* roots. HPTLC of vasicine and vasicinone on silica gel with ethyl acetate - methanol - ammonia 40:10:1. Quantitative determination by absorbance measurement at 300 nm. Linearity was 320-960 ng/spot (vasicine) and 80-400 ng/spot (vasicinone). Linearity by HPLC was 4-20 µg/mL. The HPTLC method was more suitable because of high throughput and low analysis time.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, comparison of methods 22

104 042 Danuta RAJ*, A. KOKOTKIEWICZ, M. LUCZKIEWICZ (*Department of Pharmacognosy, Wrocław Medical University, pl. Nankiera 1, 50-140 Wrocław, Poland; dankaraj@wp.pl): Densitometric HPTLC analysis of indolizidine alkaloids in the herb and in-vitro culture of *Securinega suffruticosa*. *J. Planar Chromatogr.* 22, 371-376 (2009). HPTLC of indolizidine alkaloids and extracts on silica gel and RP-18 (both prewashed with chloroform-methanol 1:1) with 11 mobile phases in a horizontal chamber without chamber saturation. The best resolution was achieved on silica gel with chloroform - methanol 20:1. Detection under UV 254 nm and after spraying with Dragendorff reagent. Quantitative determination of securinine and allosecurinine by absorbance measurement at 254 nm. The limit of detection and quantification was 11 and 36 ng/zone for securinine and 12 and 41 ng/zone for allosecurinine, respectively.

herbal, quality control, traditional medicine, HPTLC, densitometry, quantitative analysis 22

104 043 H. WIEDENFELD*, G. HOESCH, E. ROEDER, TH. DINGERMANN (*Pharmazeutisches Institut der Universität, An der Immenburg 4, 53121 Bonn, Germany; wiedenfeld@uni-bonn.de): Lycopsamine and cumambrin B from *Eupatorium maculatum*. *Pharmazie* 64, 415-416 (2009). TLC of the pyrrolizidine alkaloid lycopsamine and the guaianolide cumambrin B on silica gel with dichloromethane - methanol - 25 % ammonia 85:14:1. Detection see A. R. Mattocks, Detection of pyrrolizidine alkaloids on thin-layer chromatograms, *J. Chromatogr.* 27, 505-508 (1967).

pharmaceutical research, herbal, qualitative identification 22

104 005 M. WAKSMUNDZKA-HAJNOS et al., see section 2

104 044 L. ZHANG (Zhang Lu)*, H. LI (Li Hongyu), Y. WEI (Wei Yuhui) (*State Key Lab of Minist. Educ. of Drought & Grassplot Zoology, Coll. Life Sci., Lanzhou Univ., Lanzhou 730000, China): (Analysis of the alkaloids in the seeds of *Sophora moorcroftiana* (Benth.) Baker by TLC and HPLC) (Chinese). *Chinese J. Pharm. Anal.* 28 (7), 1071-1074 (2008). TLC of the alkaloids matrine, oxymatrine, sophocarpine, sophoridine, cytosinethe in seed extracts of *Sophora moorcroftiana* on silica gel with chloroform - methanol - ammonia 50:6:1. Detection under UV 254 nm.

Comparison with reference standards showed that extracts contained matrine, oxymatrine, sophocarpine, sophoridine, and cytosine.

pharmaceutical research, traditional medicine, quality control, herbal, densitometry, quantitative analysis, qualitative identification

22

27. Vitamins and various growth regulators

- 104 045 A. MOHAMMAD*, A. ZEHRA (*Aligarh Muslim University, Analytical Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh, India): Specific separation of thiamine from hydrophilic vitamins with aqueous dioxane on precoated silica TLC plates. *Acta Chromatographica* 20 (4), 637-642 (2008). TLC of thiamine hydrochloride from riboflavin, nicotinic acid, calcium D-pantothenate, pyridoxine hydrochloride, cyanocobalamin, and ascorbic acid on silica gel with dioxane - water 1:1. Detection under UV light. Examination of the effect of impurities (metal cations and inorganic anions) on the chromatography of thiamine hydrochloride. The detection limit for thiamine hydrochloride was 90 ng/spot and %RSD of thiamine hydrochloride was 14.9 % (n = 5).

pharmaceutical research, quality control, food analysis, quantitative analysis, qualitative identification

27

- 104 046 A. MOHAMMAD*, A. ZEHRA (*Analytical Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh Muslim University, Aligarh 202002, India; mohammadali4u@rediffmail.com): Simultaneous separation and identification of cyanocobalamin, thiamine, and ascorbic acid on polyoxyethylene sorbitan monooleate-impregnated silica layers with water as mobile phase. *J. Planar Chromatogr.* 22, 429-433 (2009). TLC of the water-soluble vitamins cyanocobalamin, thiamine, and ascorbic acid on silica gel (impregnated with a 2 % solution of the non-ionic surfactant Tween 80) with double distilled water in glass jars saturated for 30 min at 30 +/- 2° C. Detection of all substances except folic acid and cyanocobalamin by exposure to iodine vapor and evaluation in daylight.

quality control, qualitative identification

27

- 104 047 M. SUE-CHU, S. KRISTENSEN, H.H. TONNESEN* (*University of Oslo, School of Pharmacy, Department of Pharmaceutics, P. O. Box 1068, Blindern, 0316 Oslo, Norway; h.h.tonnesen@farmasi.nio.no): Photoinduced color changes in two different qualities of riboflavin in the solid state and various tablet formulations. Photoreactivity of biologically active compounds. *Pharmazie* 64, 428-435 (2009). TLC of riboflavin and lumichrome (7,8-dimethylbenzo[g]pteridine-2,4-(1H,3H)-dione) on silica gel with acetic acid - acetone - methanol - benzene 1:1:4:14 Detection under visible light and UV 254 and 366 nm.

pharmaceutical research, food analysis, qualitative identification

27

28. Antibiotics, Mycotoxins

- 104 242 R. BHUSHAN et al., see section 38

- 104 048 U. HUBICKA, J. KRZEK*, H. WOLTYNSKA, B. STACHAZ (*Collegium Medicum of Jagiellonian University, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Krakow, Poland; jankrzek@cm-uj.krakow.pl): Simultaneous identification and quantitative determination of selected aminoglycoside antibiotics by thin-layer chromatography and densitometry. *J. AOAC Int.* 92, 1068-1075 (2009). TLC and HPTLC of six antibiotics (amikacin, gentamicin, kanamycin, neomycin, netilmicin, and tobramycin) in pharmaceutical preparations on silica gel with methanol - 25 % ammonia - chloroform 3:2:1. Detection by derivatization with 0.2 % ethanolic ninhydrin reagent. Quantitative determination by absorbance measurement at 500 nm. The limit of detection and quantification was 250 and 500 ng/zone for amikacin, 500 and 1000 ng/zone for kanamycin, 480 and 800 ng/zone for neomycin, 380 and 630 ng/zone for netilmicin, 480 and 800 ng/zone for tobramycin and 1000 and 1650 ng/zone for gentamicin. Precision was good with %RSD of 0.3-0.6 %.

quality control, pharmaceutical research, HPTLC, densitometry, quantitative analysis

28a

- 104 049 S. JOSHI*, A. SHARMA, M. S. M. RAWAT, C. DHIMAN (*Department of Chemistry, K. L. D. A. V. (P. G.) College, Roorkee-247667. India; shajoshi@yahoo.com): Development of conditions for rapid thin-layer chromatography of beta-lactam antibiotics. *J. Planar Chromatogr.* 22, 435-437 (2009). TLC of penicillins (benzylpenicillin, ampicillin, and amoxicillin) and cephalosporins (cephalexin, cefoperazone, ceftriaxone, cefixime, and cefadroxil) in powder extracts on silica gel (impregnated with 0.2 % ammonium chloride) with propanol - acetic acid 4:1 and butanol - acetic acid - water 4:1:2. Detection under UV light at 365 nm.
quality control, pharmaceutical research, qualitative identification 28a
- 104 001 Tara MCGLINCHEY et al., see section 1
- 104 050 P.N. RANJANE*, S.V. GANDHI, S.S. KADUKAR, K.G. BOTHARA, (*Department of Pharmaceutical Analysis, A.I.S.S.M.S. College of Pharmacy, Kennedy Road, Near R.T.O., Pune 411 001, India): HPTLC determination of cefuroxime axetil and ornidazole in combined tablet dosage form. *J. Chromatogr. Sci.* 48 (1), 26-28 (2010). HPTLC of cefuroxime axetil and ornidazole in combined tablet dosage form on silica gel with toluene - *n*-butanol - triethylamine 17:4:1. The hR_F value of ornidazole was 51 and of cefuroxime axetil 67. Quantification by densitometry at 285 nm. Linearity was between 100 and 500 ng/band for both cefuroxime axetil and ornidazole. The method was used for the assay of the compounds in pharmaceutical formulations. The content of cefuroxime axetil was 102.4 % and of ornidazole 101.0 %.
pharmaceutical research, quality control, HPTLC, autoradiography, densitometry, quantitative analysis, qualitative identification 28a
- 104 051 A.R. ROTE*, S.P. PINGLE* (*Department of Pharmaceutical Chemistry, M. G. V.'s Pharmacy College, Panchavati (Pune University) Mumbai, Agra Road, Nashik 422003, Maharashtra, India): Reverse phase-HPLC and HPTLC methods for determination of gemifloxacin mesylate in human plasma. *J. Chromatogr. B* 877 (29), 3719-3723 (2009). HPTLC of gemifloxacin mesylate in human plasma, extracted with chloroform - acetic acid 59:1, on silica gel with ethyl acetate - methanol - ammonia 8:4:3. The hR_F value of gemifloxacin mesylate was 33. Quantification by densitometry at 254 nm. The calibration curve was established in the range of 50 to 600 ng/spot. Recovery of gemifloxacin mesylate was between 80.0 and 86.2 %. The stability of gemifloxacin mesylate in plasma was confirmed with samples submitted to three cycles of freeze-thawing at <20 °C, and with samples stored on the bench for 12 h.
clinical chemistry, research, HPTLC, densitometry, quantitative analysis, qualitative identification, comparison of methods 28a
- 104 052 D.H. SHEWIYO*, E. KAALE, P.G. RISHA, B. DEJAEGHER, J. SMEYERS-VERBEKE, Y. VANDER HEYDEN (*Directorate of Laboratory Services, Tanzania Food and Drugs Authority, P.O. Box 77150, Dar es Salaam, Tanzania): Development and validation of a normal-phase high-performance thin layer chromatographic method for the analysis of sulfamethoxazole and trimethoprim in co-trimoxazole tablets. *J. Chromatogr. A* 1216 (42), 7102-7107 (2009). HPTLC of co-trimoxazole tablets (combination of sulfamethoxazole and trimethoprim) on silica gel with toluene - ethyl acetate - methanol 100:57:43. Detection under UV 254 nm. The hR_F values were 30 and 61 for trimethoprim and sulfamethoxazole, respectively. Quantification by densitometry at UV 254 nm. Cochran's criterion test indicated homoscedasticity of variances for the calibration data. The F-tests for lack-of-fit indicated that straight lines were adequate to describe the relationship between spot areas and concentrations for each compound. Repeatability and precision (%RSD) was 1.0 and 0.8 % for sulfamethoxazole as well as 1.3 % and 1.6 % for trimethoprim, respectively. Recovery was 99.0 % and 99.7 % for sulfamethoxazole and trimethoprim, respectively.
pharmaceutical research, quality control, HPTLC, qualitative identification, quantitative analysis, densitometry 28a
- 104 053 U. VINCENT, G. GIZZI, C. VON HOLST*, J. DE JONG, J. MICHARD (*European Commission, Joint Research Centre, Insitute for Reference Materials and Measurements, Food Safety and

Quality Unit, Retieseweg, 2440 Geel, Belgium, christoph.von-holst@ec.europa.eu): Validation of an analytical method for the determination of spiramycin, virginiamycin, and tylosin in feeding-stuffs by thin-layer chromatography and bio-autobiography. *Food Addit. Contam.* 24, 351-359 (2007). Inter-laboratory validation of the analytical method (published in the SIMBAG-FEED report 4.6) based on TLC coupled to bio-autography for the detection of tylosin, spiramycin and virginiamycin in feeding-stuffs for poultry, pig, cattle and calf. The detection limit of spiramycin was 2 mg/kg and the method has a target concentration of 1 mg/kg for tylosin and virginiamycin. The method showed high specificity and offers the possibility for screening before LC/MS analysis.

quality control, quantitative analysis

28a

- 104 054 L. YANG (Yang Lihong)*, CH. HU (Hu Changqin), W. LIU (Liu Wenying) (*China Pharm. Univ., Nanjing 210009, China): (Determination of gentamycin and the related compounds by thin-layer chromatography) (Chinese). *Chinese J. Pharm. Anal.* 26 (2), 221-224 (2006). HPTLC of gentamycin and related compounds on silica gel with chloroform - methanol - 25 % ammonia 5:7:6. The main compound is well separated from the impurities. Quantification by densitometry at 485 nm. Linearity was between 4.0 - 40 ng/spot ($r^2 = 0.99$) and the limit of detection was at the low ng level.

quality control, pharmaceutical research, HPTLC, densitometry, quantitative analysis, qualitative identification

28a

29. Pesticides and other agrochemicals

- 104 055 V.R. CHANDEGAONKAR, D.B. SHINDE, D.V. MANE* (*Department of Chemistry, Chhatrapati Shivaji College, Omerga, (MS) 413606 India; manedv.2007@rediffmail.com): Thin-layer chromatographic detection and identification of the insecticide imidacloprid in biological materials. *J. Planar Chromatogr.* 22, 459-460 (2009). TLC of imidacloprid (1-[(6-chloro-3-pyridinylmethyl)]-4,5-dihydro-N-nitro-1H-imidazol-2-amine) and biological extracts on silica gel with chloroform - acetone 7:3 or hexane - acetone - ethanol 8:1:1 with chamber saturation. Detection by spraying with 5 % dimethylaminobenzaldehyde in hydrochloric acid, followed by heating at 100 °C for 10 min. Imidacloprid was detected as a pink zone under visible light.

agricultural, qualitative identification, postchromatographic derivatization

29a

- 104 056 V.R. CHANDEGAONKAR, J.N. SANGSHETTI, D.B. SHINDE, D.V. MANE* (*Department of Chemistry, Chhatrapati Shivaji College, Omerga (MS) 431606, India; manedv.2007@rediffmail.com): A new chromogenic spray reagent for detection and identification of monocrotophos. *J. Planar Chromatogr.* 22, 457-458 (2009). TLC of monocrotophos and biological extracts, dime-thoate, endosulfan, carbaryl, and cypermethrin on silica gel with chloroform - acetone 7:3 with chamber saturation. Detection by spraying with 5 % sodium hydroxide solution followed by 5 % benzil reagent (5 g benzil in 100 mL acetone) and heating at 100 °C for 10 min. Monocrotophos was detected as a pink zone in daylight.

toxicology, agricultural, qualitative identification

29b

- 104 057 W. SCHWACK*, T. ZEISLER, C. STIEFEL (*University of Hohenheim, Institute of Food Chemistry, Garbenstrasse 28, 70599 Stuttgart, Germany; wschwack@uni-hohenheim.de): Determination of dialkyl phosphates as breakdown products of organophosphorus insecticides in fruit juices by HPTLC with fluorescence detection. *J. AOAC Int.* 92, 691-697 (2009). HPTLC of dialkyl phosphate standards (dimethyl phosphate, diethyl phosphate, dimethyl thiophosphate, diethyl thiophosphate and dibutyl phosphate as internal standard) on amino phase, prewashed with methanol, with dichloromethane in a twin trough chamber. Quantitative determination by fluorescence measurement at 366/>400 nm. The limit of quantification was between 0.8 and 1.4 ng/zone. Fluorescence enhancement was achieved by dipping the plate into a 50 % solution of paraffin oil in *n*-hexane, increasing the sensitivity and resulting in an LOQ of 0.5-0.6 ng/zone.

agricultural, toxicology, quality control, food analysis, HPTLC, densitometry, quantitative analysis

29b

- 104 058 T. TOSTI, G. RAKIC, M. NATIC, D. MILOJKOVIC-OPSENICA, S. HUSINEC, V. SAVIC, Z. TESIC* (*Faculty of Chemistry, University of Belgrade, P. O. Box 51, 11158 Belgrade, Serbia; ztesic@chem.bg.ac.rs): TLC retention behavior of brodifacoum, bromadiolone, and coumatetralyl and their impurities on different adsorbents. *J. Planar Chromatogr.* 22, 333-343 (2009). TLC of brodifacoum, bromadiolone, coumatetralyl and impurities on silica gel, alumina, cellulose, and RP-18 with 46 different mobile phases in a twin trough chamber saturated for 30 min. The most selective phases on silica gel were chloroform - ethyl acetate - *n*-hexane in various ratios, ethyl methyl ketone- toluene 1:9, and chloroform - toluene 7:3; on alumina the best mobile phase was dichloromethane - *n*-hexane 3:2. Detection under UV light at 254 nm.

toxicology, qualitative identification

29f

- 104 059 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Medical University of Lublin, 4 Staszica Street, 20-081 Lublin, Poland, tomasz.tuzimski@umlub.pl): Application of SPE-HPLC-DAD and SPE-HPTLC-DAD to the analysis of pesticides in lake water. *J. Planar Chromatogr.* 22, 235-240 (2009). HPTLC of pesticides (clofentezine, neburon, chlorfenvinphos, lenacyl, trifluralin, thiram, procymidone, flufenoxuron, tralkoxydim, propaquizafop, dinoseb) and water samples (sample preparation by solid phase extraction) on silica gel with ethyl acetate - *n*-heptane 1:4 and 3:7 in a horizontal chamber. Quantitative determination by diode array densitometry in the range of 200 to 600 nm. The limit of detection was between 40 and 650 ng/spot and the limit of quantification was between 120 and 1920 ng/spot.

environmental, toxicology, HPTLC, quantitative analysis

29a

30. Synthetic and natural dyes

- 104 060 Gertrud MORLOCK*, C. OELLIG (*University of Hohenheim, Garbenstrasse 28, 70599 Stuttgart, Germany; gmorlock@uni-hohenheim.de): Rapid planar chromatographic analysis of 25 water-soluble dyes used as food additives. *J. AOAC Int.* 92, 745-756 (2009). HPTLC of 25 dyes (brilliant black BN, tartrazine, ponceau 6 R, resorcin yellow, fast yellow AB, orcein, allura red, green S, amaranth, quinoline yellow, acid blue, erythrosine, sunset yellow FCF, indigo carmine, ponceau 4R, azorubine, brilliant blue FCF, carmine, scarlet GN, copper chlorophyll, encyanine, chlorophyllin trisodium copper salt, curcumin, riboflavin-5-phosphate, riboflavin) on silica gel in a horizontal developing chamber with ethyl acetate - methanol - water - acetic acid 65:23:11:1 for 40 samples in 12 min. Relative humidity was 21 +/- 3 % at a temperature of 20 +/- 3 °C. Alternatively, a twin trough chamber or automatic developing chamber can be used. Quantitative determination by absorbance measurement at 11 different wavelengths. Repeatabilities (%RSD, n = 4) near the limit of quantification showed precisions of mostly <2.7 %, ranging between 0.2 and 5.2 %. Correlation coefficients (R >0.9987) and RSD values (<4.2 %) of the calibration curves were highly satisfactory using classical quantification. However, digital evaluation of the plate image was also used for quantification, which resulted in RSD values of the calibration curves of mostly <3.0%, except for two <6.0%. The method was applied for the analysis of some energy drinks and bakery ink formulations, directly applied after dilution. By recording of absorbance spectra in the visible range, the identities of the dyes found in the samples were ascertained by comparison with the respective standard bands (correlation coefficients >0.9996). If necessary for confirmation, online mass spectra were recorded within a minute.

food analysis, quality control, HPTLC, densitometry, quantitative analysis

30a

- 104 014 F. SCHULTE et al., see section 4e

- 104 061 ZH. WANG (Wang Zhiyong)*, B. WANG (Wang Bingquan), H. ZOU (Zou Hong) (*Dep. Chem., Capital. Norm. Univ., Beijing 10037, China): (Identification of the category of black gel pen ink by thin-layer chromatography) (Chinese). *Physical Testing and Chem. Anal., Part B: Chem. Anal.* 45 (1), 14-18 (2009). TLC of ink extracts on silica gel with *n*-butanone - ethanol - water - acetic acid 14:4:6:1. Detection in visible light and identification by comparison of spot colors and hR_F values. The method was used for the identification of 37 different black ink samples from gel pens from different sources, which could then be classified into 13 categories.

quality control, qualitative identification

30a

32. Pharmaceutical and biomedical applications

- 104 062 S. AGARWAL*, A. AIL, Y. DU, S. ABUJA (*Dept. of Pharmaceutics, Faculty of Pharmacy, Jamie Hamdard University, New Delhi 110062, India, agarwal_sp@yahoo.com): Determination of artemisinin in bulk and pharmaceutical dosage forms using HPTLC. Ind. J. Pharma. Sci. 71(1), 98-100 (2009). HPTLC of artemisinin on silica gel with toluene - ethyl acetate with chamber saturation for 30 min. Quantitative determination by absorbance measurement at 520 nm. The method was linear over the range of 100-600 ng/band, recovery was in the range of 98.8-100.5 %.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 063 S. AHMAD* , M. SINGH, M. KAUR, A. AHMED (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Quantitative estimation of glycyrrhizic acid in the tablets of Yashtimadhu by HPTLC. 60th Indian Pharmaceutical Congress PG-260 (2008). HPTLC of glycyrrhizic acid on silica gel with chloroform - glacial acetic acid - methanol - water 15:8:3:2. Quantitative determination by absorbance measurement at 254 nm. The hR_F value of glycyrrhizic acid was 28. The method was linear in the range of 50-500 ng/spot. The sample analyzed by the proposed method contained 87.8 μg glycyrrhizic acid per tablet, equivalent to 0.015 % w/w of the tablet formulation.
- quality control, herbal, HPTLC, densitometry, quantitative analysis 32e
- 104 064 S. AHMAD*, A. KAMAL, R. PARVEEN, F. AHMAD, K. SALEEM (*JMI, Dept. of Biosciences, New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of strychnine and brucine in seeds of Strychnos Nux Vomica. 60th Indian Pharmaceutical congress PA-235 (2008). HPTLC of strychnine and brucine (in seeds of Strychnos Nux Vomica) on silica gel with chloroform - methanol - ammonia 38:2:1. Quantitative determination by absorbance measurement at 258 nm (strychnine, hR_F value 29) and 271 nm (brucine, hR_F value 21). The method was linear in the range of 100-1000 ng/spot (both compounds). The method was suitable for standardization of several herbal formulations containing nux vomica as an ingredient.
- herbal, HPTLC, densitometry, quantitative analysis 32e
- 104 065 S. AHMAD*, M. SINGH, R. PARVEEN, Y. KAMAL (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Quantitative estimation of withaferin a in the tablets of Ashwagandha by HPTLC. 60th Indian Pharmaceutical Congress PG-262 (2008). HPTLC of withaferin A in Ashwagandha on silica gel aluminum layer with toluene - ethyl acetate - formic acid 5:5:1. Quantitative determination by absorbance measurement at 214 nm. The chromatograms of the tablet formulation showed a zone corresponding to standard withaferin A (hR_F value 37) indicating the presence of the same in the herbal formulation. Linearity was in the range 50 to 500 ng/spot. The concentration of withaferin A was 8.07 μg /tablet and 0.00134 % w/w in the tablet formulation.
- quality control, herbal, HPTLC, densitometry, quantitative analysis 32e
- 104 066 M. ALAVALA (KLE University's College of Pharmacy, Belgaum, Karnataka, India): New stability indicating high-performance liquid chromatography and high-performance thin-layer chromatography method for the estimation of olopatadine hydrochloride. Abstract No. F-309, 61st IPC (2009). HPTLC of olopatadine HCl on silica gel with methanol - chloroform - 25 % ammonia 80:20:1. The hR_F value was 46. Quantitative determination by absorbance measurement at 301 nm. The method was linear in the range of 100-900 ng/band.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 067 B. ARUN*, A. SUGANTHI, A. FATHIMUNNISA, T. RAVI (*College of Pharmacy, Sri Ramakrishna Institute of Paramedical Science, Coimbatore, Tamil Nadu, India): Development of validated HPTLC method for the estimation of buclizine hydrochloride in tablet dosage form. Abstract No. F-276, 61st IPC (2009). HPTLC of buclizine hydrochloride on silica gel with methanol - chloroform - ammonia 8:1:1 %. Quantitative determination by absorbance measurement at 234 nm.

The calibration curve was linear in the range of 100-700 ng/spot. The limit of detection and limit of quantification were 20 and 100 ng/spot, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 068 J. BAGYALAKSHMI*, S. VIJAYARAJ, Sajna JOHN, T. RAVI (*Sri Ramkrishna Institute of Paramedical Science, College of Pharmacy, Coimbatore, Tamilnadu, India): Development and validation of simultaneous estimation of paracetamol, aceclofenac and rabeprazole in combined tablet dosage formulation by HPTLC method. 60th Indian Pharmaceutical Congress PA-222 (2008). HPTLC of paracetamol, aceclofenac and rabeprazole on silica gel with ethyl acetate - methanol - glacial acetic acid 90:10:1. Quantitative determination by absorbance measurement at 275 nm. Linearity was in the range of 100-500 µg/mL, 20-100 µg/mL and 2-10 µg/mL for paracetamol, aceclofenac and rabeprazole respectively. Recovery was in the range of 98.9-100.1 % for all three compounds.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 069 N. BAIRWA*, A. TRIVEDI, S. MISHRA (*The M. S. University of Baroda, Pharmacy Dept., Vadodara, Gujarat, India): Simultaneous estimation of markers in a haematinic herbomineral formulation using high-performance thin-layer chromatography. 60th Indian Pharmaceutical Congress PG-251 (2008). HPTLC of glycyrrhetic acid and piperine in haematinic herbomineral capsule formulation on silica gel with toluene - ethyl acetate - glacial acetic acid 25:15:1. Quantitative determination by absorbance measurement at UV 254 nm. The method was linear in the range of 0.8-2.4 ng/spot (glycyrrhetic acid) and 10-50 ng/spot (piperine). Recovery was 96.3-98.6 %.

herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 070 V. BALI*, M. ALI, J. ALI (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): A novel and rapid HPTLC method for the analysis of rosuvastatin calcium. 60th Indian Pharmaceutical Congress PA-219 (2008). HPTLC of rosuvastatin calcium on silica gel with toluene - ethyl acetate - formic acid 9:7:1. Quantitative determination by absorbance measurement at 254 nm. The hR_F value was 32. The method was linear in the range of 100-100 ng/spot, recovery was 99.7 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 071 V. BHAVNANI*, V. DIWALE, P. DESHMUKH, A. DHIWARE (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): Validated method development for estimation of famotidine and domperidone in combined tablet dosage form. Abstract No. F-335, 61st IPC (2009). HPTLC of famotidine and domperidone on silica gel with toluene - methanol - triethyl amine 12:6:1. The hR_F value was 23 and 67 for famotidine and domperidone, respectively. Quantitative determination by absorbance measurement at 290 nm. The method was linear in the range of 100-500 ng/band. Recovery was 98.6-98.9 % for both drugs.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 072 D. BHAWSAR*, P. RATHI, M. PURANIK, P. YEOLE (*Institute of Pharmaceutical Education and Research, Wardha, Maharashtra, India): Development and validation of HPTLC technique for simultaneous estimation of gatifloxacin and ornidazole in solid dosage forms. Abstract No. F-347, 61st IPC (2009). HPTLC of gatifloxacin and ornidazole on silica gel with dichloromethane - methanol - 25 % ammonia 95:10:3. The hR_F value was 16 and 60 for gatifloxacin and ornidazole, respectively. Quantitative determination by absorbance measurement at 302 nm. The method was linear in the range of 20-200 ng/band for gatifloxacin and 50-500 ng/band for ornidazole. Recovery was between 100.4 and 101.9 for both drugs.

pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 32a

- 104 073 R. BIDAWAI*, N. RAUT, D. WANKHEDE, N. GAIKWAD (*University Dept. of Pharmaceutical Science, RTM Nagpur University, Nagpur, Maharashtra, India): Validated stability indicating

HPTLC method for the estimation of olmesartan medoxomil in bulk and pharmaceutical dosage form. 60th Indian Pharmaceutical Congress PA-226 (2008). HPTLC of olmesartan medoxomil (an angiotensin-II antagonist) on silica gel with toluene - acetonitrile - methanol - ethyl acetate - acetic acid (mobile phase ratio not specified by the authors). The hR_F value was 56. Quantitative determination by absorbance measurement at 262 nm. The linearity was between 300-800 ng/spot. The method was suitable for separation of olmesartan medoxomil from degradation products obtained by forced stress conditions (acid, alkali, peroxide, light, heat).

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 074 A. CHABUKSWAR*, S. JAGDALE, S. KUMBHAR, R. WAVHALE (*Bombay College of Pharmacy, Mumbai, Maharashtra, India): Simultaneous HPTLC estimation of certain antihypertensive drugs in tablet dosage form. 60th Indian Pharmaceutical Congress PA-230 (2008). HPTLC of amlodipine besylate (AB) and telmisartan (TMS) on silica gel with ethyl acetate - 1,4-dioxane - methanol - 25 % ammonia 30:3:6:3. The hR_F value was 16 for TMS and 33 for AB. Quantitative determination by absorbance measurement at 323 nm. The method was linear in the range of 100-500 $\mu\text{g/mL}$ (TMS) and 200-1000 $\mu\text{g/mL}$ (AB), average recovery was 100.2-100.4 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 075 Mrinalini DAMLE*, K. BOTHARA, Kirti TOPAGI (*Dept. of Chem. A.I.S.S.M.S. College of Pharma, Kennedy Road, R.T.O. Pune 411044, India., mcdamle@rediffmail.com): Stability-indicating HPTLC method for determination of nebivolol hydrochloride and valsartan. Ind. J. Pharma. Sci. 8(4), 198-201(2009). HPTLC of nebivolol hydrochloride and valsartan on silica gel with ethyl acetate - methanol - acetic acid 12:2:1 in a twin trough chamber saturated for 15 min. Quantitative determination by absorbance measurement at 240 nm for valsartan and 280 nm for nebivolol hydrochloride. The method was found to be linear in the range of 600-1400 ng/band for valsartan 1200-2800 ng/band for nebivolol. The sample were subjected to different stress conditions (acid, alkali, oxidation, photolysis, thermal) and all degradation products were well separated from the main compounds.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 076 R.S. DAREKAR, A.B. KHETRE, S.M. SINGH, M.C. DAMLE* (*Department of Pharmaceutical Chemistry, AISSMS College of Pharmacy, Near R. T. O., Kennedy Road, Pune 411 001, India, mcdamle@rediffmail.com): HPTLC quantitation of 2-hydroxy-4-methoxybenzaldehyde in Hemidesmus indicus R. Br. root powder and extract. J. Planar Chromatogr. 22, 453-456 (2009). HPTLC of 2-hydroxy-4-methoxybenzaldehyde and biological extracts on silica gel with toluene - ethyl acetate - acetic acid 7:2:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 277 nm.

herbal, traditional medicine, quality control, HPTLC, densitometry, quantitative analysis 32e

104 077 P. DESHPANDE*, G. SHRIDHARAN, L. ANANDI, D. JADHAV, M. DAMLE, S. GANDHI (*Dept. of Pharmaceutical Analysis, AISSMS College of Pharmacy, Kennedy Road, Pune, India, santoshvgandhi@rediffmail.com): Validated method development for estimation of atorvastatin calcium and fenofibrate in fixed dose combination by HPTLC. The Pharma Review 7(39), 151-153 (2009). HPTLC of atorvastatin calcium and fenofibrate on silica gel (pre-washed with methanol) with chloroform - methanol 4:1 over 20 mm with chamber saturation. The hR_F value of atorvastatin calcium was 29 and of fenofibrate 77. The method was linear in the range of 200-1000 ng/band for atorvastatin calcium and 320-1600 ng/band for fenofibrate.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 078 S.R. DHANESHWAR*, V.K. BUSARI, M.V. MAHADIK (*Bharati Vidyapeeth University, Poona College of Pharmacy, Department of Pharmaceutical Chemistry, Pune, Maharashtra, India 411038; sunil.dhaneshwar@gmail.com): Application of a stability-indicating thin-layer chromatographic method to the determination of tenatoprazole in pharmaceutical dosage forms. J.

AOAC Int. 92, 387-393 (2009). TLC of tenatoprazole - before and after acid and alkali hydrolysis, oxidation and photodegradation - on silica gel, prewashed with methanol and dried at 110 °C for 5 min, with toluene - ethyl acetate - methanol 6:4:1 in a twin trough chamber saturated with the mobile phase for 30 min at 25 °C. Quantitative determination by absorbance measurement at 306 nm. The limit of detection and limit of quantitation were 50 and 100 ng/spot, respectively.

quality control, pharmaceutical, research, densitometry, quantitative analysis 32a

104 079 V.V. DIGHE, G.A. CHAREGAONKAR* (*S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai 400 019, India; gauricharegaonkar@gmail.com): HPTLC analysis of myristicin and safrole in seed powder of *Myristica fragrans* Houtt. J. Planar Chromatogr. 22, 445-448 (2009). HPTLC of myristicin, safrole and extract of seeds on silica gel, prewashed with methanol, with toluene in an automatic developing chamber saturated for 20 min. Quantitative determination by absorbance measurement at 210 nm for myristicin and at 290 nm for safrole.

pharmaceutical research, quality control, herbal, traditional medicine, HPTLC, densitometry, quantitative analysis 32e

104 080 Avani DODIYA, Shweta PAWAR, C. PATEL (*School of Pharmacy and Technology Management, NMIMS University, Mumbai, Maharashtra, India): Simultaneous determination of nebivolol hydrochloride and hydrochlorothiazide in tablets by high-performance thin-layer chromatography. Abstract No. F-280, 61st IPC (2009). HPTLC of nebivolol HCl and hydrochlorothiazide on silica gel with toluene - ethyl acetate - methanol - 25 % ammonia 30:27:17:2. The hR_F value was 38 and 68 for hydrochlorothiazide and nebivolol, respectively. Quantitative determination by absorbance measurement at 281 nm. The method was linear in the range of 500-3000 ng/band for nebivolol and 1000-6000 ng/band for hydrochlorothiazide. Recovery was 100 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 081 N. DUBEY*, N. DUBEY, R. MEHTA, A. K. SALUJA (*Devi Ahilya Vishwa Vidyalaya, School of Pharmacy, Indore, India; nidhidubeypharm@yahoo.com) : Determination of psoralen and plumbagin from its polyherbal oil formulations by an HPTLC densitometric method. J. AOAC Int. 92, 779-784 (2009). HPTLC of psoralen and plumbagin and extracts of ayurvedic polyherbal oil formulations on silica gel at 22 °C and 55 % humidity with toluene - ethyl acetate 3:1 in a twin trough chamber with chamber saturation. UV spectra were recorded from 200 to 600 nm; densitometric measurements were performed at 302 nm (for psoralen) and 275 nm (for plumbagin).

traditional medicine, herbal, quality control, HPTLC, densitometry, quantitative analysis 32e

104 082 N. DUBEY*, N. DUBEY, R. MEHTA, A. SALUJA (*Devi Ahilya Vishwavidyalaya (DAVV), School of Pharmacy, Indore, Madhya Pradesh, India; nidhidubeypharm@yahoo.com): Estimation of catechin in Ayurvedic oil formulations containing *Acacia catechu*. J. AOAC Int. 92, 1021-1026 (2009). HPTLC of catechin and extracts from polyherbal oil formulations on silica gel using chloroform - acetone - 0.1 % formic acid 77:15:8 % in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 296 nm. The limit of detection and quantification was 6 and 20 ng/spot, respectively.

traditional medicine, herbal, quality control, HPTLC, quantitative analysis, densitometry 32e

104 083 Nidhi DUBEY*, N. DUBEY, R. MEHTA, A. SALUJA (*Devi Ahilya Vishwavidyalaya, School of Pharmacy, Indore, M.P., India): Rapid densitometric determination of *Allium sativum* in polyherbal oil formulations. 60th Indian Pharmaceutical Congress PA-202 (2008). HPTLC of allyl disulphide (an active ingredient of *Allium sativum*, garlic) on silica gel with *n*-hexane. The hR_F value was 52. Quantitative determination by absorbance measurement at 298 nm. The linearity range was 200-1200 ng/spot. Several polyherbal formulations containing garlic were analyzed with the proposed method using allyl disulphide as marker.

traditional medicine, herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 085 R.R. DURÓN, L. CENICEROS ALMAGUER, N.C. CAVAZOS ROCHA, P.G.S. FLORES, Noemi WAKSMAN DE TORRES* (*Universidad Autónoma de Nueve León, Departamento de Química Analítica, Facultad de Medicina, PO Box 2316, Sucursal Tecnológico, 64841, Monterrey Nuevo León, Mexico; nwaksman@fm.uanl.mx): Comparison of high-performance liquid chromatographic and thin-layer chromatographic methods for determination of aloin in herbal products containing Aloe vera. *J. AOAC Int.* 91, 1265-1270 (2008). TLC of aloin A and extracts of dried herbal products applied bandwise on silica gel with ethyl acetate - methanol - water 100:17:10 at 25 °C with chamber saturation. Detection by spraying with 1 % methanolic diphenylboric acid 2-aminoethylester (natural products reagent), followed by 5 % ethanolic polyethylene glycol 400 and visualization under UV light at 365 nm. The hR_F value of aloin (as a mixture of aloin A and B stereoisomers) was 38. Linearity was between 0.5 and 10 mg/L. Precision (%RSD) of the hR_F value of the aloin band was 0.88. The limit of detection was 1 µg/band. Recovery (by standard addition) was 87.6 % for aloin.
quality control, herbal, pharmaceutical research, qualitative identification 32e
- 104 086 Halina EKIERT*, A. SZEWCZYK, A. KUS (*Jagiellonian University, Collegium Medicum, Faculty of Pharmacy, Chair and Department of Pharmaceutical Botany, 9 Medyczna Street, 30-688 Kraków, Poland; mfekiert@cyf-kr.edu.pl): Free phenolic acids in *Ruta graveolens* L. in vitro culture. *Pharmazie* 64, 694-696 (2009). Preparative TLC and HPTLC of protocatechuic acid, vanillic acid, syringic acid, and p-coumaric acid and methanolic extracts on silica gel. Detection under UV light at 254 nm.
pharmaceutical research, HPTLC, herbal, qualitative, identification, preparative TLC 32e
- 104 087 Izabella FECKA (Wroclaw Medical University, Department of Pharmacognosy, Pl. Nankierea 1, 50-140 Wroclaw, Poland; izabela@farmgn.am.wroc.pl): Development of chromatographic methods for determination of agrimoniin and related polyphenols in pharmaceutical products. *J. AOAC Int.* 92, 410-418 (2009). HPTLC of agrimoniin, pedunculagin, ellagic acid, gallic acid and catechin and plant extracts on silica gel, RP-18 and amino phase in a horizontal chamber. The best resolution and selectivity were achieved with diisopropyl ether - acetone - formic acid - water 4:3:2:1, tetrahydrofuran - acetonitrile - water 3:1:6, and acetone - formic acid 3:2. Polyphenols were detected under UV light at 254 nm and in visible light after spraying with 1 % methanolic iron(III) chloride or bis-diazotized sulfanilamide and after treatment with a vanillin-hydrochloric acid reagent.
herbal, pharmaceutical research, quality control, HPTLC, qualitative identification 32e
- 104 088 J. FISCHEDICK*, R. GLAS, A. HAZEKAMP, R. VERPOORTE (*Division of Pharmacognosy, Leiden University, Gorlaeus Laboratories, 2333 CC Leiden, The Netherlands, jtfische@gmail.com): A qualitative and quantitative HPTLC densitometry method for the analysis of cannabinoids in *Cannabis sativa*. *Phytochem. Anal.* 20, 421-426 (2009). HPTLC of delta-9-tetrahydrocannabinol in the flowertops of *Cannabis sativa* on silica gel with chloroform with chamber saturation for 20 min. Quantitative determination by absorbance measurement at 206 nm. Derivatization by dipping in Fast Blue B solution for 5 s. The hR_F value of delta-9-tetrahydrocannabinol was 47 and selectivity regarding matrix was given. Linearity was given between 50 and 500 ng/zone. The limit of quantification and detection was 50 and 10 ng/zone, respectively. The intra- and inter-day repeatability (%RSD, n = 9) were not higher than 5.0 %. Recovery was 85.8 % for delta-9-tetrahydrocannabinol in decarboxylated *Cannabis* samples. The method was shown to be comparable within a small degree of error (0.5 %) to results from a validated HPLC method.
toxicology, quality control,
herbal, HPTLC, densitometry, quantitative analysis, qualitative identification 32e
- 104 089 M. GAME*, M. JADHAO, V. WANKHADE, G. GHENGE (*Vidyabharti College of Pharmacy, Amravati, Maharashtra, India): Estimation of andrographolide in herbal powder and polyherbal asava by HPTLC. Abstract No. F-299, 61st IPC (2009). HPTLC of herbal powder and polyherbal formulation containing *Andrographis paniculata* on silica gel with benzene - ethyl acetate 1:1.

Quantitative determination by absorbance measurement at 222 nm. The calibration curve for andrographolide was linear in the range of 360-660 ng/band.

quality control, herbal, HPTLC, densitometry 32e

- 104 090 D. GANDHI*, N. PATEL, P. MEHTA (*Institute of Pharmacy, Nirma University, Ahmedabad, Gujarat, India): Development and validation of HPTLC method for simultaneous determination of levodopa and carbidopa in their combined dosage form. Abstract No. F-246, 61st IPC (2009). HPTLC of levodopa and carbidopa on silica gel with acetone - chloroform - *n*-butanol - acetic acid - water 50:45:42:35:25. Quantitative determination by absorbance measurement at 283 nm. The method was linear in the range of 200-700 ng/band for both compounds, with a recovery of 98.7-99.9 %.

pharmaceutical research, quality control, HPTLC, densitometry, comparison of methods 32a

- 104 091 S.V. GANDHI*, S.I. KHAN, R.T. JADHAV, S.S. JADHAV, G.A. JADHAV (*AISSMS College of Pharmacy, Department of Pharmaceutical Analysis, Pune, India; santoshvgandhi@rediffmail.com): High-performance thin-layer chromatographic determination of rabeprazole sodium and domperidone in combined dosage form. J. AOAC Int. 92, 1064-1067 (2009). HPTLC of rabeprazole sodium and domperidone in tablets on silica gel (prewashed with methanol and dried at 110 °C for 5 min) with toluene - acetone - methanol 9:9:1 in a twin trough chamber saturated with the mobile phase for 5 min. Quantitative determination by absorbance measurement at 285 nm. The hR_F value of rabeprazole sodium and domperidone was 53 and 32, respectively. Linearity was between 50 and 800 ng/band for both substances.

quality control, pharmaceutical, research, HPTLC, quantitative analysis, densitometry 32a

- 104 092 M. GANESH*, S. SWANT, R. JAMBHALE, A. KASABE (*Arvind Gawali College of Pharmacy, Satara, Maharashtra, India): Extraction and estimation of theobromine in marketed tea by HPTLC and UV method. Abstract No. F-277, 61st IPC (2009). HPTLC of theobromine in different extracts of tea (*Camelia sinensis*) on silica gel with ethyl acetate - methanol 27:3. Quantitative determination by absorbance measurement at 274 nm. The maximum content of theobromine in tea samples was 2.3 %. Linearity was in the range of 3-15 µg/zone. The limit of detection and quantification was 30 and 140 ng/spot, respectively.

pharmaceutical research, herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 093 A. GANTAIT* , K. MUKHERJEE, S. PONNUSANKAR, P. MUKHERJEE (*Jadavpur University, School of Natural Product Studies, Kolkata, India): A validated method for standardization of *Centella asiatica* extract. 60th Indian Pharmaceutical Congress PG-244 (2008). HPTLC of *Centella asiatica* extract with asiaticoside as marker on silica gel with chloroform - glacial acetic acid - methanol - water 15:8:3:2 with chamber saturation. Detection by spraying with anisaldehyde reagent, followed by heating in oven and immediate quantitative determination by absorbance measurement at 607 nm. The hR_F value of asiaticoside was 81. Linearity was in the range of 0.96-3.36 µg/spot. Hydroalcoholic extracts contained approx. 3.2 % of asiaticoside.

herbal, HPTLC, densitometry, quantitative analysis, postchromatographic derivatization 32e

- 104 094 A GANTAIT*, N. NEMA, A. SAHU, S. PANDIT, S. BHADRA, P. MUKHERJEE (*School of Natural Product Studies, Jadavpur University, Kolkata 700032, India): A validated method for quantification of glycyrrhizin in *Glycyrrhiza glabra* extract by HPTLC. Abstract No. 9162, IHC (2009). HPTLC of glycyrrhizin in methanolic (70 %) extracts of *Glycyrrhiza glabra* on silica gel with chloroform - methanol - water 130:72:15. Quantitative determination by absorbance measurement at 254 nm and at 420 nm after spraying with anisaldehyde reagent. The method was linear in the range of 0.8-3.8 µg/spot. The limit of detection and quantification was 0.16 and 0.52 µg/spot, respectively. Glycyrrhizin was used as bioactive marker for quality control.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, postchromatographic derivatization 32e

- 104 095 A. GANTAIT*, P. ROY, S. PANNUSANKAR, P. MUKHERJEE, B. SAHA (*School of Natural Product Studies, Jadavpur, Kolkata 700032, India): Standardization of *Tinospora cordifolia* extract through HPTLC densitometry. Abstract No. 91334, IHCB (2009). Standardization of *Tinospora cordifolia* extract by HPTLC of syringic acid on silica gel with chloroform - methanol 8:1. The hR_F value of syringic acid was 53. Quantitative determination by absorbance measurement at 254 nm.
pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32e
- 104 096 Vidya GAWANDE*, Manisha PURANIK, A. CHANDEWAR (*Pataldhamal Wadhvani College of Pharmacy, Yavatmal, Maharashtra, India): Development of validated HPTLC method for simultaneous estimation of domperidone in combination with esomeprazole magnesium in solid dosage form. 60th Indian Pharmaceutical Congress PA-220 (2008). HPTLC of domperidone and esomeprazole on silica gel with chloroform - methanol 9:1 with chamber saturation for 30 min. The hR_F value was 25 for domperidone and 46 for esomeprazole. Quantitative determination by absorbance measurement at 295 nm. The method was linear in the range of 60-300 ng/spot for domperidone and 80-400 ng/spot for esomeprazole.
pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 097 S. GHODKE*, A. RATHORE, L. SATHIYANARAYANAN, K. MAHADIK (*Bharati Vidya-peeth University, Poona College of Pharmacy, Pune, Maharashtra, India): Validated HPTLC method for simultaneous estimation of isotretinoin and erythromycin in bulk drug and topical gel form. Abstract No. F-243, 61st IPC (2009). HPTLC of isotretinoin and erythromycin on silica gel with toluene - dimethyl sulfoxide - methanol 65:2:25. Quantitative determination by absorbance measurement at 340 nm before derivatization for isotretinoin and at 410 nm for erythromycin after derivatization with 10 % sulfuric acid followed by heating at 100 °C for 15 min. The hR_F value was 38 and 55 for isotretinoin and erythromycin, respectively. The linearity was in the range of 30-150 ng/band for isotretinoin and 1200-6000ng/band for erythromycin. Recovery was between 96.9 and 99.7 for isotretinoin and between 97.2 and 102.6 % for erythromycin. Intra-day and inter-day relative standard deviations for both components were <2.0 %.
pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 098 S. GOHIL*, S. PATEL, N. PATEL, D. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganapat University, Mehsana, India): Estimation of atomoxetine hydrochloride by HPTLC method in pharmaceutical formulations. 60th Indian Pharmaceutical Congress PA-212 (2008). HPTLC of atomoxetine HCl on silica gel with acetone - methanol - triethylamine 30:15:2. Quantitative determination by absorbance measurement at 275 nm. The method was linear in the range of 300-2100 ng/spot and was suitable for routine quality control of pharmaceutical formulations.
pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 099 P. HAMRAPURKAR*, N. LONE, K. KAMAT, T. SAMBARE (*Principal K. M. Kundnani College of Pharmacy, Mumbai, Maharashtra, India): Quantitative determination of aloe-emodin in *Rheum emodi* using HPTLC. 60th Indian Pharmaceutical Congress PA-224 (2008). HPTLC of aloe-emodin in extract of *Rheum Emodi* (prepared by supercritical fluid extraction) on silica gel with toluene - acetone - formic acid 80:20:1. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 100-400 ng/spot. Compared with other extraction techniques supercritical fluid extraction was more efficient and less time consuming.
herbal, HPTLC, quantitative analysis, qualitative identification 32e
- 104 100 T. HONG, M.L. JEONG, M. ZAHN, B.A. FAY, K. LEE, H. HWANGBO, E. PARK, M. KIM, W. MA* (*Unigen Inc., Quality Control/Quality Assurance Department, 2660 Willamette Dr, NE, Lacey, WA 98516, USA; WenwenM@unigen.net): Detection of the potential adulterant *Teuc-*

rium chamaedrys in *Scutellaria baicalensis* raw material and extract by high-performance thin-layer chromatography. *J. AOAC Int.* 92, 785-788 (2009). HPTLC of plant extracts and herbal preparations applied bandwise on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:25 after preconditioning for 5 min. Detection by immersion in natural products reagent (diphenylboric acid 2-aminoethylester) followed by polyethylene glycol 400 reagent for 2 s. After air-drying the plates were evaluated under UV 366 nm.

herbal, traditional medicine, quality control, HPTLC, qualitative identification 32e

- 104 101 F. HOU (Hou Feng)*, F. LIU (Liu Fang), Q. MO (Mo Qiwu) (*Guangzhou Meichen Pharm. Co. Ltd., Guangzhou 510075, China): (Study of the quality standard for Conghuang Bushen capsules) (Chinese). *J. Chinese Trad. & Herb. Drugs* 40 (8), 1249-1252 (2009). TLC of extracts of the TCM drug on silica gel with 1) methanol - acetic acid - water 18:1:4; 2) petroleum ether (60-90 °C) - ethyl acetate 1:1; 3) toluene - ethyl acetate - methanol 5:5:3; 4) petroleum ether (60-90 °C) - ethyl acetate - formic acid 15:5:1. Detection 1) by spraying with potassium iodobismuthate reagent; 2) under UV 254 nm; 3) by spraying with 5 % AlCl₃ in ethanol and evaluation under UV 365 nm.
- quality control, pharmaceutical research, traditional medicine, quantitative analysis, qualitative identification 32e

- 104 102 X. HOU (Hou Xiaotao)*, L. MU (Mu Liquan), L. HUANG (Huang Lifen), J. ZHOU (Zhou Jianguyu) (*Guangxi Inst. TCM, Nanning, Guangxi 530001, China): (Study on the quality standard for Yixuean Pills) (Chinese). *Chinese J. Hospit. Pharm.* 29 (8), 686-688 (2009). TLC of the extracts of Yixuean pills on silica gel with 1) chloroform - ethyl acetate - acetone - formic acid 60:25:25:4; 2) *n*-hexane - chloroform - methanol 15:5:2; 3) ethyl acetate - formic acid - acetic acid - water 15:1:1:2. Detection 1) under UV 254 nm; 2) by exposure to iodine vapor and under UV 254 nm; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration evaluation under visible light and UV 254 nm.
- cosmetics, pharmaceutical research, traditional medicine, quality control, qualitative identification, quantitative analysis, review, densitometry 32c

- 104 103 J. HUANG (Huang Jiefen)*, H. LI (Li Huixia), H. DENG (Deng Huimin) (*Guangzhou Zhongyi Pharm. Co. Ltd., Guangzhou 510530, China): (Study of the quality standard for Xinyi Biyan pills) (Chinese). *J. Chinese Trad. & Herb. Drugs* 40 (8), 245-247 (2009). TLC of the TCM drug extracts on silica gel with 1) petroleum ether (90-120 °C) - toluene - formic acid 20:40:1; 2) chloroform - ethyl acetate - methanol - water - formic acid 3:10:2:2:2; 3) ethyl acetate - formic acid - water 12:2:3. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C; 2) by exposure to iodine vapor; 3) under UV 254 nm.
- pharmaceutical research, quality control, traditional medicine, herbal, qualitative identification, quantitative analysis 32e

- 104 104 X. HUANG (Huang Xiaoyu)*, N. HUANG (Huang Nojia) (*Wannianqing Pharm. Co., Guangdong Prov., Shantou, Guangdong 515031, China): (Simultaneous identification of *Fructus Schisandrae sphenantherae*, *Rhizoma Acori tatarinowii*, *Rhizoma Chuanxiong* and vitamin E in Naolibao pills by thin-layer chromatography) (Chinese). *J. Chinese Pharm. Standard* 10 (4), 284-286 (2009). TLC of Naolibao pill extracts on silica with *n*-hexane - ethyl acetate 5:1. Qualitative identification by detection under UV 254 nm and 365 nm. The method is simple, rapid, reliable, and suitable for the quality control of the TCM formulation.
- pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification 32c

- 104 105 Demiana I. NESSEEM*, C.G. MICHEL, A.A. SLEEM, T.S. EL-ALFY (*Pharmaceutics Department, National Organization for Drug Control & Research (NODCAR), 6 Abou Hazem St. Pyramids Ave, Cairo, Egypt; demianaessem@yahoo.com): Formulation and evaluation of antihyperglycemic leaf extracts of *Zizyphus spina-christi* (L.) WILLD. *Pharmazie* 64, 104-109

(2009). TLC of christinin-A as marker and leaf extracts on silica gel with chloroform - methanol - water 13:8:2 and butanol - acetic acid - water 4:1:5 (upper phase). Detection by spraying with p-anisaldehyde reagent.

pharmaceutical research, herbal, qualitative identification 32e

104 106 P.S. JAIN (R.C. Patel College of Pharmacy, Karwand Naka, Shirpur Dist. Dhule 425 405 (M.S.) India): Stability-indicating HPTLC determination of ambroxol hydrochloride in bulk drug and pharmaceutical dosage form. J. Chromatogr. Sci. 48 (1), 45-48 (2010). HPTLC of ambroxol hydrochloride on silica gel with methanol - triethylamine 2:3. The hR_F value of ambroxol was 53. Quantification by absorbance measurement at 254 nm. Linearity was given in the range of 100-1000 ng/spot with $r^2=0.9966$ (via peak area). The limits of detection and quantitation were 10 and 30 ng/spot, respectively. Ambroxol hydrochloride was susceptible to degradation under oxidation and thermal stress conditions. The method is suitable for purity testing of the drug as it detects the related impurities.

pharmaceutical research, quality control, HPTLC, qualitative identification, quantitative analysis, densitometry 32c

104 107 V. JAITAK*, A. GUPTA, V. KAUL, P. AHUJA (*Natural Plant Products Div. Institute of Bio-resource Technology, Palampur 176061, H.P., India, vkaul2002@yahoo.co.in): Validated high-performance thin-layer chromatography method for steviol glycosides in Stevia rebaudiana. J. Pharm. Biomed. Anal. 47, 790-794 (2008). HPTLC of the steviolbioside, stevioside and rebaudioside A in Stevia rebaudiana leaves on silica gel with ethyl acetate - ethanol - water 20:5:3. Detection by spraying with acetic anhydride - sulphuric acid - ethanol 1:1:10 reagent. Quantitative determination by absorbance measurement at 510 nm. Linearity was in the range of 160-960 ng/spot for steviolbioside, 1-6 µg/spot for stevioside and 0.5-3 µg/spot for rebaudioside A with good correlation coefficients (0.998-0.999). The method was used for the assay of steviol glycosides in S. rebaudiana leaves collected from ten different locations.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32e

104 108 P. JHA*, S. KHAN, R. PARVEEN, S. AHMAD (*Jamia Hamdard, Faculty of Pharmacy (Hamdard University), New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of omeprazole in pharmaceutical dosage form. 60th Indian Pharmaceutical congress PA-234 (2008). HPTLC of omeprazole on silica gel with chloroform - methanol 9:1. The hR_F value was 39. Quantitative determination by absorbance measurement at 302 nm. The method was linear in the range of 5-3000 ng/spot, recovery was 99.5 %. The method was suitable for routine quality control of formulations.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 109 Maithilee JOSHI*, A. NIKALJE, M. SHAHED, M. DEHGAN (*Y. B. Chavan College of Pharmacy, Dr. Rafiq Zakaria Campus, Rauza Bagh, Aurangabad 431001, India, ana@k.st): HPTLC method for the simultaneous estimation of emtricitabine and tenofovir in tablet dosage form. Ind. J. Pharma. Sci. 71(1), 95-97 (2009). HPTLC of emtricitabine and tenofovir on silica gel with chloroform - methanol 9:1. Quantitative determination by absorbance measurement at 265 nm. The calibration curve was linear between 200 and 1000 ng with a regression coefficient of 0.9995.

pharmaceutical research, densitometry, HPTLC, quantitative analysis 32a

104 110 S.S. KADUKAR, S.V. GANDHI*, P.N. RANJANE, S.S. RANHER (*Department of Pharmaceutical Analysis, AISSMS College of Pharmacy, Kennedy Road, Near R. T. O., Pune 411 001, Maharashtra, India; santoshvgandhi@rediffmail.com): HPTLC analysis of olmesartan medoxomil and hydrochlorothiazide in combination tablet dosage forms. J. Planar Chromatogr. 22, 425-428

(2009). HPTLC of olmesartan medoxomil and hydrochlorothiazide on silica gel, prewashed with methanol, with chloroform - methanol - toluene 6:4:5 in a twin trough chamber saturated for 15 min. Quantitative determination by absorbance measurement at 258 nm.

pharmaceutical research, quality control, HPTLC

32a

104 111 R. KAKDE*, D. SATONE, N. BAWANE (*Department of Pharmaceutical Sciences, RTM Nagpur University, Nagpur-440 033, Maharashtra, India; drkakde@yahoo.com): HPTLC method for simultaneous analysis of escitalopram oxalate and clonazepam in pharmaceutical preparations. J. Planar Chromatogr. 22, 417-420 (2009). HPTLC of escitalopram oxalate and clonazepam on silica gel, prewashed with methanol, in a twin trough chamber saturated for 20 min at 25 °C with methanol - toluene - triethylamine 10:35:1. Quantitative determination by absorbance measurement at 253 nm.

pharmaceutical research, quality control, HPTLC, quantitative analysis, densitometry 32a

104 112 P. KAKULTE*, M. DESHPANDE, S. CHAUDHRI, V. KASTURE (*Amrutvahini College of Pharmacy, Ahmednagar, Maharashtra, India): High-performance thin-layer chromatographic determination of ambroxol in human plasma by liquid-liquid extraction and its use in stability study. Abstract No. F-286, 61st IPC (2009). HPTLC of ambroxol (extracted from plasma with diethylether, after centrifugation the organic layer was evaporated and the residue was taken up in 1 mL of methanol) on silica gel with acetonitrile - methanol - triethylamine 41:5:4. Quantitative determination by absorbance measurement at 254 nm.

pharmaceutical research, clinical routine analysis, HPTLC, quantitative analysis 32b

104 113 R. KANT*, M. GUPTA (*Delhi Institute of Pharmaceutical Science and Research New Delhi, India): HPTLC method development and its validation for determination of ranolazine in pharmaceutical formulations. Abstract No. F-259, 61st IPC (2009). HPTLC of ranolazine on silica gel with methanol - toluene 9:11 in a twin trough chamber with chamber saturation for 30 min. The hR_F value was 64. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 2-14 µg/band. Recovery was 98.3-101.4 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

104 114 A. KAUR*, V. RAVICHANDRAN, P. JAIN, R. AGRAWAL (*Pharmaceutical Chemistry Research Lab. Dept. of Pharmaceutical Sciences, Dr. Hari Singh Gour Univ. Sagar, MP 470003, India, dragrawal2001@yahoo.co.in): High-performance thin-layer chromatography method for estimation of conessine in herbal extract and pharmaceutical dosage formulations. J. Pharm. Biomed. Anal. 46, 391-394 (2008). TLC of conessine on silica gel with toluene - ethyl acetate - diethyl amine 13:5:2 in a twin trough chamber saturated at 25 °C. Detection by treatment with modified Dragendorff's reagent. Quantitative determination by absorbance measurement at 520 nm. The hR_F value of conessine was 82. Linearity was in the range of 1-10 µg/zone with a correlation coefficient of 0.9998 via peak area.

pharmaceutical research, herbal, densitometry, quantitative analysis 32e

104 115 H. KHAN*, M. ALI, A. AHUJA, S. AHMAD, J. Ali (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Stability indicating TLC method for simultaneous estimation of aceclofenac and paracetamol in bulk drugs and in their fixed dose combinations. 60th Indian Pharmaceutical Congress PA-218 (2008). TLC of aceclofenac and paracetamol on silica gel with toluene - isopropylalcohol - ammonia 8:7:1. The hR_F value of aceclofenac was 24 and of paracetamol 68. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 25-2000 ng/band with correlation coefficients of 0.9998 for aceclofenac and 0.9996 for paracetamol. The limits of detection and quantification were 25 and 150 ng/band for aceclofenac and 50 and 200 ng/band for paracetamol. Both drugs were subjected to acid and alkali hydrolysis, oxidative degradation, and photodegradation. The degradation products were well resolved from the pure drug.

pharmaceutical research, quality, control, densitometry, quantitative analysis 32a

- 104 116 P. KHATWANI*, S. KULKARNI (*Bombay College of Pharmacy, Mumbai, Maharashtra, India): A sensitive high-performance thin-layer chromatography method for estimation of wedelolactone from *Eclipta alba* by different methods of extraction. Abstract No. F-272, 61st IPC (2009). HPTLC of wedelolactone on silica gel with toluene - ethyl acetate 9:1. Quantitative determination by fluorescence measurement at 366 nm. The method was linear in the range of 500-8000 ng/mL. Recovery was in the range of 100.2-101.0 %. The plant material was extracted using percolation, maceration, hot solvent extraction, supercritical fluid extraction, microwave assisted extraction, ultra sonication, and an orbital shaker. Quantification of wedelolactone in the extracts showed highest levels for Soxhlet extraction and lowest levels for supercritical fluid extraction.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32a

- 104 117 A. KHODKE*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Pune, Maharashtra, India): A validated stability indicating HPTLC method for simultaneous estimation of irbesartan and hydrochlorothiazide. Abstract No. F-269, 61st IPC (2009). HPTLC of hydrochlorothiazide and irbesartan on silica gel with acetonitrile - chloroform 5:6. The hR_F value was 27 and 45 for irbesartan and hydrochlorothiazide, respectively. Quantitative determination by absorbance measurement at 270 nm. The sample was exposed to different stress conditions (acid, alkali, oxidative, photodegradation, thermal). Neither of the compounds showed degradation under thermal and photodegradation conditions, but both compounds showed significant degradation under acid, alkali and hydrolytic conditions. Degraded products were well resolved from the parent compounds.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 118 E. KILINC*, F. AYDIN (*Department of Chemistry, Faculty of Art and Science, University of Dicle, Diyarbakir, 21280, Turkey; ekilinc@dicle.edu.tr): Stability-indicating HPTLC analysis of flurbiprofen in pharmaceutical dosage forms. *J. Planar Chromatogr.* 22, 349-354 (2009). HPTLC of flurbiprofen (2-(3-fluoro-4-phenyl)phenylpropanoic acid) and degradation products on silica gel, prewashed with methanol, with chloroform - acetone - xylene 5:2:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement (the authors report no wavelength). Linearity was between 50 and 600 ng/band. The limit of detection and quantification was 10 and 32 ng/band, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 119 S. KOTHARI*, D. PATEL, N. SHAH, B. SUHAGIA (*Shri B. M. Shah College of Pharmacy, Education and Research, Modasa, Gujarat, India): HPTLC method for simultaneous estimation of telmisartan and hydrochlorothiazide from their combination drug product. Abstract No. F-378, 61st IPC (2009). HPTLC of telmisartan and hydrochlorothiazide on silica gel with chloroform - toluene - methanol 2:5:5. The hR_F value was 53 and 75 for telmisartan and hydrochlorothiazide respectively. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 240-640 ng/band for telmisartan and 200-700 ng/band for hydrochlorothiazide.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 120 Miroslawa KRAUZE-BARANOWSKA*, I. MALINOWSKA, D. GLOD, M. MAJDAN, A. WILCZANSKA (*Department of Pharmacognosy, Medical University of Gdansk, Gen. J. Hallera 107, 80-416, Poland, Krauze@amg.gda.pl): UTLC of flavonols in *Sambucus nigra* flowers. *J. Planar Chromatogr.* 22, 385-387 (2009). Ultrathin-layer chromatography of quercetin, rutin, and quercetin-3-O-glucoside on monolithic silica gel (size 30 mm x 18 mm) with binary and tertiary mobile phases in a cylindrical glass chamber previously saturated for 1 min. The migration distance was 20 mm and development time was 2 min. The investigated mobile phases were ethyl

acetate - *n*-hexane 1:4 and 3:7, tetrahydrofurane - hexane 2:3 and 3:2, tetrahydrofurane - methanol - hexane 3:3:4, and hexane - acetone - methyl ethyl ketone 3:3:4. The best separation was achieved with acetone - methyl ethyl ketone - hexane 3:4:3. Densitometric evaluation at 366 nm.

herbal, quality control, densitometry, HPTLC, quantitative analysis 32e

- 104 121 M. KRISHNA*, V. MURAGAN, P. MUSMADE, S. VENKATARAM (*Dayananda Sagar College of Pharmacy, Bangalore, Karnataka, India): Stability indicating high-performance thin-layer chromatography method for determination of triamcinalone acetonide in bulk drug and pharmaceutical dosage forms. 60th Indian Pharmaceutical Congress PA-211 (2008). HPTLC of triamcinalone acetonide on silica gel aluminum foil with toluene - ethyl acetate - ammonia 33:67:1 %. The hR_F value of triamcinalone acetonide was 38. Quantitative determination by absorbance measurement at 240 nm. The method was linear in the range of 100-2000 ng/spot; recovery was 99.5 %. The stability indicating method has been successfully applied to forced degradation studies of triamcinalone acetonide (acid, alkali, hydrogen peroxide, photo degradation thermal and neutral hydrolysis) and resolved degradation products and excipients from triamcinalone acetonide.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 122 M. KUMAR*, B. SRINIVASAN (*Delhi Institute of Pharmaceutical Sciences and Research (DIPASR), New Delhi, India): Stability indicating HPTLC method for the determination of cinitapride hydrogen tartrate in bulk drug and pharmaceutical formulations. Abstract No. F-240, 61st IPC (2009). HPTLC of cinitapride hydrogen tartrate on silica gel with methanol - toluene 17:3. The hR_F value was 71. Quantitative determination by absorbance measurement at 265 nm. The linearity was in the range of 90-450 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidative, photodegradation, dry and wet heat) and degradation products were well separated from the main component.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 123 R. KUNDU*, S. DHOLE, M. CHARDE, A. KASTURE (*J. L. Chaturvedi College of Pharmacy, Nagpur, Maharashtra, India): High-performance thin-layer chromatographic method for simultaneous estimation of benzhexol hydrochloride and trifluperazine hydrochloride in pharmaceutical preparations. Abstract No. F-248 61st IPC (2009). HPTLC of benzhexol HCl and trifluperazine HCl on silica gel with methanol - acetone - toluene - 25 % ammonia 10:10:70:1. The hR_F value was 37 and 82 for trifluperazine and benzhexol respectively. Quantitative determination by absorbance measurement at 210 nm. The method was linear in the range of 40-800 ng/band for benzhexol and 100-2000 ng/band for trifluperazine. Recovery was 99.7-99.9 % for both compounds.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 124 N. LADKAT*, M. ANRUTKAR, J. JAGADE, A. KALE, S. PAWAR, A. BHOSALE (*Poona Dist. Education Asso. Seth Govind Raghunath Sable College of Pharmacy, Pune, Maharashtra, India): HPTLC estimation of cefixime and cloxacillin in tablet dosage form. Abstract No. F-260, 61st IPC (2009). HPTLC of cefixime and cloxacillin on silica gel, prewashed with methanol, with *n*-butanol - methanol - water - formic acid 80:60:40:3. The hR_F values were 28 and 45 for cefixime and cloxacillin, respectively. Quantitative determination by absorbance measurement at 293 nm for cefixime and 343 nm for cloxacillin. The linearity range was 150-600 ng/band for both compounds.

pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 32a

- 104 125 Q. LI (Li Qongya)*, J. WANG (Wang Jiabin), Z. MA (Ma Zuo), SH. CHEN (Chen Shuhe), Y. LIU (Liu Yanwen), (*Joint State Key Lab of Minist. Educ. of Hubei Coll TCM & Head Off. of TCM Compound, Wuhan, Hubei 430061, China): (Qualitative and quantitative study of *Alternanthera philoxeroides* (Mart.) Griseb) (Chinese). Chinese J. Pharm. Anal. 28 (5), 732-734 (2008). TLC of *Alternanthera philoxeroides* extracts on silica gel with chloroform - methanol 40:1. The method is suitable for the quality control of the TCM drug and its formulations.

quality control, pharmaceutical research, traditional medicine, herbal, densitometry, quantitative analysis, qualitative identification 32e

- 104 126 H. LI* (Li Hui), J. HU (Hu Jiangyu), H. OUYANG (Ouyang Hui), Y. LI (Li Yanan), H. SHI (Shi Hui), C. MA (Ma Chengjin), Y. ZHANG (Zhang Yongkang) (*Jishou University, Hunan Provinc Key Laboratory of Forest Products and Chemical Industry Engineering, Hunan Zhangjiajie, 427000, People's Republic of China, and Jishou University, College of Chemistry and Chemical Engineering, Hunan Jishou 416000, People's Republic of China; lihuijdx@163.com): Extraction of aucubin from seeds of *Eucommia ulmoides* Oliv. using supercritical carbon dioxide. *J. AOAC Int.* 92, 103-110 (2009). Analytical and preparative TLC of aucubin and herbal extracts after extracton with supercritical carbon dioxide on silica gel with methanol - chloroform - petroleum ether - ethyl acetate 1:3:3:1. Visualization by spraying with 30 % sulfuric acid.

herbal, traditional medicine, pharmaceutical research, quality control, qualitative identification, preparative TLC 32e

- 104 127 M. LIU (Liu Ming)*, G. LI (Li Gengsheng), H. WANG (Wang Huisheng) (*Henan Provin. Acad. Chinese Trad. Med. & Pharm., Zhengzhou, Henan 450004, China): (Study of the quality standard of *Rehmannia glutinosa* (Gdertn) Libosch, a Chinese traditional herbal drug) (Chinese). *Chinese J. Phram. Anal.* 27 (9), 1311-1313 (2007). TLC of drug extracts on silica gel with trichloromethane - methanol - water 12:8:1. Detection by spraying with 5 % vanillin - sulfuric acid reagent and evaluation in daylight and under UV light.

pharmaceutical research, traditional medicine, quality control, herbal, quantitative analysis, qualitative identification 32c

- 104 128 H. LU (Lu Hui)*, H. XIE (Xie Hongping), SH. YANG (Yang Shilin), B. GU (Gu Bing) (*Pharm. Coll., Suzhou Univ., Suzhou, Jiangsu 215123, China): (Identification of leaf of *Vitex negundo* L. var. *cannabifolia* (Sieb. et Zucc.) Hand. -Mazz. and *Oleum Viticis Negundo* by thin-layer chromatography) (Chinese). *J. Chinese Trad. Med. & Pharm. (Shizhen Guoyi Guoyao)* 20 (11), 2799-2800 (2009). TLC of TCM drug extracts on silica gel with petroleum ether (60-90 °C) - ethyl acetate 100:3. Detection by spraying with a solution of 5 % vanillin - 10 % sulfuric acid in ethanol 1:10 followed by heating at 105 °C until coloration, evaluation in visible and UV light.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, quantitative analysis 32e

- 104 129 K. LUNIYA*, R. MANTRI, S. DUBEY, S. BHARANI (*A.I.S.S.M.S. College of Pharmacy, Pune Maharashtra, India): Validated method development for estimation of naproxen sodium as bulk drug and in tablet dosage form by HPTLC. Abstract No. F-283, 61st (2009). HPTLC of naproxen sodium on silica gel with toluene - ethyl acetate - acetic acid 15:3:2. The hR_F value was 63. Quantitative determination by absorbance measurement at 230 nm. The method was linear in the range of 100-500 ng/band.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 130 ROSIDAH, M. YAM*, A. SADIKUN, M. AHMAD, G. AKYIREM, M. ZAINI (*Department of Human Anatomy, Faculty of Medicine and Health Sciences, University Putra Malaysia, Serdang 43400, Selangor, Malaysia, yammunfei@yahoo.com) : Toxicology evaluation of standardized methanol extract of *Gynura procumbens*. *J. Ethnopharmacol.* 123, 244-249 (2009). HPTLC of kaempferol-3-O-rutinoside and astragaloside in leaves of *Gynura procumbens* on silica gel with acetic acid - methanol - dichloromethane 1:3:7. Quantitative determination by absorbance measurement at 366 nm. The hR_F values of kaempferol-3-O-rutinoside and astragaloside were 43 and 72, respectively, and selectivity regarding matrix was given. Linearity was given between 16 and 1000 µg/mL and the correlation coefficients were >0.987.

toxicology, herbal, HPTLC, quantitative analysis, densitometry 32e

- 104 131 A. MADAN*, B. PATEL (*K. B. Institute of Pharmaceutical Education and Research Gandhinagar, Gujarat, India): HPTLC method for simultaneous determination of rabeprazole and itopride in capsules and its validation. Abstract No. F-244, 61st IPC (2009). HPTLC of rabeprazole and itopride on silica gel with ethyl acetate - methanol - benzene - chloroform 2:4:3:1. The hR_F value was 42 and 61 for rabeprazole and itopride, respectively. Quantitative determination by absorbance measurement at 276 nm. The method was linear in the range of 200-300 ng/band for rabeprazole and 1500-2250 ng/band for itopride.
pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 132 V. MADHAVAN*, S. YOGANARSIMHAN, R. TIJARE (*M. S. Ramaiah College of Pharmacy, Bangalore, India): Pharmacognostical and phytochemical studies of roots tubers of *Asparagus gonocladus* Baker. Abstract No. 9966, IHCB (2009). HPTLC of root tuber extracts of *Asparagus gonocladus* Baker on silica gel with ethyl acetate - methanol - water 15:3:2. Detection by spraying with anisaldehyde reagent. Quantitative determination by absorbance measurement at 425 nm. Shatavarin IV was used as marker.
pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis, postchromatographic derivatization 32e
- 104 133 U. MALLAVADHANI*, G. SAHU (*Herbal Drugs and Bio-Remedies, Institute of Minerals and Material Technology (CSIR), Bhubaneswar-751 013, Orissa, India; uvmavadani@yahoo.com): A rapid HPTLC method for standardization of *Ficus bengalensis* Linn. J. Planar Chromatogr. 22, 377-380 (2009). HPTLC of stigmast-5-en-3beta-O-D-glucoside and bark extracts on silica gel with chloroform - methanol - water 33:7:4 in a saturated twin trough chamber. Detection by derivatization with anisaldehyde reagent followed by heating at 105 °C for 5 min. Quantitative determination by absorbance measurement at 515 nm.
herbal, traditional medicine, quality control, HPTLC, quantitative analysis, densitometry 32e
- 104 135 K. MANGUKIA*, T. VAJA, Hasumati RAJ, Sadhana RAJPUT (*N. R. Vekaria Institute of Pharmacy & Research Centre Junagadh, Gujarat, India): Development and validation of stability-indicating HPLC and HPTLC methods for analysis of ezetimibe in pure form and in pharmaceutical formulation. Abstract No. F-320, 62st IPC (2009). HPTLC of ezetimibe on silica gel with ethyl acetate - toluene - methanol - formic acid 10:10:1:1. Quantitative determination by absorbance measurement at 231 nm. The method was linear in the range of 301-3610 ng/band. Recovery was 99.3-100.4 % The drug was exposed to different stress conditions (acid, base, oxidative, thermal) and all degradation products were well resolved from the main compound.
pharmaceutical research, quality control, HPTLC, densitometry, comparison of methods, quantitative analysis 32a
- 104 136 T. MANI*, S. BADAMI, N. MAHADEVAN, S. MANIMARAN, B. SURESH (*Bharathi College of Pharmacy, Bharathi Nagara, Karnataka, India): Determination of harmalin in *Passiflora edulis* leaves by HPTLC. Abstract No. 9274, IHCB (2009). HPTLC of harmalin from ethanolic leaf extracts of *Passiflora edulis* on silica gel with ethyl acetate - acetic acid - formic acid - water 100:11:11:27. The hR_F value of harmalin was 39. The method was linear in the range of 200-1600 ng/spot, recovery was 98.4-99.2 %.
pharmaceutical research, quality control, herbal, densitometry, HPTLC, quantitative analysis 32e
- 104 137 S. MANIMARAN*, M. CHAITANYA, T. PRAVEEN, S. DHANABAL (*JSS College of Pharmacy, Ootacamund, Tamil Nadu, India): Method validation and estimation of isovitexin content in *Passiflora incarnata* Linn by HPTLC technique. 60th Indian Pharmaceutical Congress PG-258 (2008). HPTLC of isovitexin in *Passiflora incarnata* raw material and extract on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:26. The plant was found to contain 0.087 % w/w of isovitexin.
herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 138 R. MANTRI*, M. SENGAR, U. PATIL, S. GANDHI (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): High-performance thin-layer chromatographic determination of diclofenac sodium and thicolchicoside in fixed dose combination. Abstract No. F-284, 61st IPC (2009). HPTLC of diclofenac sodium and thicolchicoside on silica gel with toluene - ethyl acetate - methanol 5:3:2. The hR_F value was 17 and 53 for thicolchicoside and diclofenac sodium, respectively. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 50-300 ng/band for both drugs. Recovery was 100.5-101.1 %.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 139 A. MASLANKA, J. KRZEK*, M. STOLARCZYK (*Department of Inorganic and Analytical Chemistry, Jagiellonian University, Collegium Medicum, 9 Medyczna Street, 30-688 Cracow, Poland, jankrzek@cm-uj.krakow.pl): Simultaneous analysis of hydrochlorothiazide, triamterene, furosemide, and spironolactone by densitometric TLC. J. Planar Chromatogr. 22, 405-410 (2009). TLC of hydrochlorothiazide, triamterene, furosemide, and spironolactone on silica gel with hexane - ethyl acetate - methanol - water - acetic acid 42:40:15:2:1 with chamber saturation. Quantitative determination by absorbance measurement at 264 nm. The limit of detection for the different compounds was between 22 and 150 ng/band, and the limit of quantification was between 68 and 450 ng/band.
- pharmaceutical research, quality control, densitometry, quantitative analysis 32a
- 104 140 Annie MATHEW*, R. RAVINDRA (*C. U. Shah College of Pharmacy, S.N.D.T. Women's University, Juhu Road, Santacruz (W) Mumbai, India): Quantitative HPTLC analysis of diallyl disulfide in garlic oil macerate. Abstract No. 9286, IHCB (2009). HPTLC of diallyl disulfide in garlic oil macerate on silica gel with *n*-hexane - isopropyl alcohol - formic acid 196:4:3. Quantitative determination by absorbance measurement at 210 nm. The method was linear in the range of 16-48 µg/spot.
- pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32e
- 104 141 S. MATHUR*, D. SHARMA, P. SAINI, R. SINGH (*R & D Div., Indian Pharmacopoeia Commission, Ministry of Health & Family Welfare, Govt. of India, Ghaziabad, U.P., India): Simultaneous estimation of domperidone and paracetamol in bulk and its tablets dosage forms by HPTLC method. Abstract No. F-395, 61st IPC (2009). HPTLC of domperidone and paracetamol on silica gel with acetone - toluene - methanol 2:2:1 with chamber saturation at room temperature. The hR_F value was 52 and 74 for domperidone and paracetamol, respectively. Quantitative determination by absorbance measurement at 248 nm (paracetamol) and 285 nm (domperidone). The method was linear in the range of 16-48 ng/band. Recovery was 99.5-101.2 %.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 142 Dipali MEHETRE*, A. CHABUKSWAR, B. KUCHEKAR, A. KATEGAONKAR (*MAEER's Maharashtra Institute of Pharmacy, Pune, Maharashtra, India): Validation of HPTLC method for simultaneous quantitation of olmesartan medoximal and amlodipine besylate in bulk drug and formulation. Abstract No. F-258, 61st IPC (2009). HPTLC of olmesartan medoximal and amlodipine besylate on silica gel with chloroform - methanol - toluene - acetic acid 80:10:1:1. Quantitative determination by absorbance measurement at 254 nm. The linearity range was 800-5000 ng/band for olmesartan and 200-1400 ng/band for amlodipine. Recovery was in the range of 98-102 % for both drugs.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 143 A. MISHRA*, R. BHOMIA, S. VASANTHARAJU, A. KARTHIK, S. SAYED, K. BHAT (*L. M. College of Pharmacy, Ahmedabad, Gujarat, India): Simultaneous estimation of salbutamol silphate and guaiphenesin in their combined liquid dosage form by HPTLC method. Abstract No. F-238, 61st IPC (2009). HPTLC of salbutamol silphate and guaiphenesin, used as pharmaceutical

syrup against cough, on silica gel with ethyl acetate - methanol - 25 % ammonia 15:3:2. The hR_F value was 47 and 65 for salbutamol and guaiphenesin, respectively. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 200-1000 ng/band for salbutamol and 10-15 µg/band for guaiphenesin.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 144 A. MISHRA*, R. BHOMIA, S. VASANTHARAJU, A. KARTHIK, S. SYED, K. BHAT (*Manipal College of Pharmaceutical Sciences, Manipal Univeristy, Manipal, Karnataka, India): Stability-indicating HPTLC method for the estimation of tolterodine in bulk drug. Abstract No. F-237, 61st IPC (2009). HPTLC of tolterodine on silica gel with toluene - methanol - 25 % ammonia 250:250:1. The hR_F value was 40. Quantitative determination by absorbance measurement at 284 nm. The method was linear in the range of 200-1000 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidation, thermal) and degradation under alkaline conditions was observed. The degradation products were well separated from the main component.

pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 32a

- 104 145 S. MISHRA*, Sunita CHAUDHARY, K. GADHVI (*Saraswati Institute of Pharmaceutical Sciences, Ahmedabad, Gujarat, India): Estimation of glycyrrhizic acid and withanolide A in polyherbal formulation by HPTLC. 60th Indian Pharmaceutical Congress PA-233 (2008). HPTLC of glycyrrhizic acid on silica gel with toluene - ethyl acetate - glacial acetic acid 25:15:1. The hR_F value of glycyrrhizic acid was 52, linearity was in the range of 500-1000 ng/mL and recovery was 99.2 %. Withanolide A was separated with toluene - ethyl acetate - glacial acetic acid 20:20:1. The hR_F value of withanolide A was 44, linearity was in the range of 400-1000 ng/mL and recovery was 99.4 %.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis

32e

- 104 146 K. MODT*, N. PATEL, R. GOYAL (*Dept. of Pharmacology, Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa, Gujarat, India): A sensitive HPTLC method for the estimation of L-dopa from *Muccuna pruriens* Linn and a formulation containing *M. pruriens*. Abstract No. 9425, IHCB (2009). HPTLC of L-dopa in *Mucuna pruriens* seed extract and formulations on silica gel with *n*-butanol - acetic acid - water 4:1:1. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 100-1200 ng/spot with an average recovery of 100.3 %.

pharmaceutical research, quality control, herbal, densitometry, HPTLC, quantitative analysis

32e

- 104 147 P. MUCAJI*, M. NAGY, T. LIPTAJ, N. PRÓNAYOVÁ, E. SVAJDLENKA (*Department of Pharmacognosy and Botany, Faculty of Pharmacy, Comenius University, Odbojárov 10, 832 32 Bratislava, Slovak Republic, mucaji@fpharm.uniba.sk): Separation of a mixture of luteolin-7-rutinoside and luteolin-7-neohesperidoside isolated from *Ligustrum vulgare* L. J. Planar Chromatogr. 22, 301-304 (2009). Analytical and preparative TLC of luteolin-7-rutinoside and luteolin-7-neohesperidoside on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:23, on cellulose with 30 % acetic acid, and on polyamide with chloroform - methanol - 2-butanone - acetyl acetone (= pentane-2,4-dione) 9:4:3:1. Detection by spraying with natural products reagent followed by treatment with PEG 4000, or by aniline phthalate.

herbal, quality control, traditional medicine, preparative TLC, qualitative identification 32e

- 104 148 A. MUJTABA*, S. BABOOTA, J. ALI, K. KOHLI (*Dept. of Pharmaceutics, Faculty of Pharmacy, Hamdard University, New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of ondansetron HCl in bulk and pharmaceutical dosage form. Abstract No. F-274, 61st IPC (2009). HPTLC of ondansetron HCl on silica gel with chloroform - ethyl acetate - methanol - 25 % ammonia 90:50:40:1. The hR_F value was 52. Quantitative de-

termination by absorbance measurement at 254 nm. The method was linear in the range of 100-1400 ng/band. Recovery was 99.3 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 149 B. NARWATE*, P. GHULE, P. MOHITE, R. UGALE (*Dept. of Pharmaceutical Chem., MES College of Pharma., Sonai, Tal.-Newasa, Dist.-Ahmednagar 414105 (M.S.) India., balaji_narwate@rediffmail.com): A high-performance thin-layer chromatographic determination of clopidogrel bisulphate in tablets. *Ind. J. Pharma. Sci.* 8(4), 211-212 (2009). TLC of clopidogrel bisulphate on silica gel with carbon tetrachloride - ethyl acetate - ammonia 50:3:2. Quantitative determination by absorbance measurement at 230 nm. Linearity was between 300 and 1500 ng. In comparison with the labeled claim the amount of clopidogrel in tablets was 99.2 %. The recovery was 99.2 % (via peak area).

pharmaceutical research, quality control, densitometry, quantitative analysis 32a

- 104 150 J. NIRMAL*, S. MAHESHWARI, H. RAJ, S. RAJPUT (*N.R. Vekaria Institute of Pharmacy & Research Centre, Junagadh, Gujarat, India): Development and validation of stability-indicating HPLC and HPTLC methods for analysis of pravastatin in pure form and application of the methods for estimation of pharmaceutical formulation. Abstract No. F-315, 61st IPC (2009). HPTLC for pravastatin on silica gel with ethyl acetate - toluene - acetonitrile - formic acid 60:35:5:2. Quantitative determination by absorbance measurement at 237 nm. The method was linear in the range of 318-3816 ng/band. Recovery was 99.9-101.2 %. Stability tests showed that degradation products resulting under acid stress conditions were well resolved from the main component.

quality control, HPTLC, densitometry, comparison of methods, quantitative analysis 32a

- 104 151 K. NISHAMOL*, A. BINDU, N. ALEYKUTTY, S. JOSE (*M. G. Univeristy, Dept. of Pharmaceutical Sciences, Ettumanoor, Kottayam, Kerala, India): Development of a high-performance thin-layer chromatography method for the quantitative estimation of rutin in the fresh leaves of *Moringa pterygosperma* Gaertn. 60th Indian Pharmaceutical Congress PG-263 (2008). HPTLC of rutin in methanolic extracts of fresh leaves of *Moringa pterygosperma* on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:26. Quantitative determination by absorbance measurement at 254 nm. The method provided good resolution of rutin from other constituents of the plant.

herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 152 D.N. OLENNIKOV (Laboratory of Medical and Biological Research, Department of Biologically Active Substances, Institute of General and Experimental Biology, Siberian Division, Russian Academy of Sciences, Sakh'yanovoy St 6, 670047, Ulan-Ude, Russia; oldaniil@rambler.ru): Densitometric HPTLC analysis of aloenin in aloe pharmaceuticals. *J. Planar Chromatogr.* 22, 359-362 (2009). HPTLC of aloenin in aloe juice, tablets, and liquid extracts on silica gel with ethyl acetate - 95 % ethanol - water 20:3:1 at room temperature in a saturated chamber. Detection by immersion for 1 s in freshly prepared 5 % sodium hydroxide solution in 95 % ethanol, followed by heating at 100 °C for 5 min. Quantitative determination by absorbance measurement at 365 nm.

herbal, quality control, HPTLC, densitometry, quantitative analysis 32e

- 104 153 A. OSMAN*, M. OSMAN (*National Organization for Drug Control and Research, 6 Abu Hazem St Pyramids, PO Box 29, Cairo, Egypt; afaf_osmanelteti@yahoo.com): Spectrofluorometry, thin-layer chromatography, and column high-performance liquid chromatography determination of rabeprazole sodium in the presence of its acidic and oxidized degradation products. *J. AOAC Int.* 92, 1373-1381 (2009). TLC of rabeprazole sodium and its degradation products on silica gel with isopropanol - 30 % ammonia 40:1 with chamber saturation. Quantitative determination by absorbance measurement at 284 nm.

pharmaceutical research, quality control, quantitative analysis, densitometry 32a

- 104 154 M. PAI*, Rajashree GUDE, Swati KENY (*Goa College of Pharmacy, Panaji, Goa, India): Development and validation of a new sensitive method for the quantitative analysis of ranitidine hydrochloride and domperidone in antiulcer combination by using HPTLC. 60th Indian Pharmaceutical Congress PA-215 (2008). HPTLC of ranitidine HCl and domperidone in combined dosage form on silica gel with ethyl acetate - methanol - ammonia 100:10:1 in a twin trough chamber saturated for 10 min. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 100-500 ng/ μ L for both compounds with a recovery of 102.5-100.8 %.
- pharmaceutical research, quality control, HPTLC, densitometry, comparison of methods, quantitative analysis 32a
- 104 155 H.J. PANCHAL*, B.N. SUHAGIA, N.J. PATEL (*Patel College of Pharmaceutical Education and Research, Ganpat Vidyannagar, Kherva, Mehsana 382711, Gujarat, India; hiral.panchal@ganpatuniversity.ac.in): Simultaneous HPTLC analysis of atorvastatin calcium, ramipril, and aspirin in a capsule dosage form. J. Planar Chromatogr. 22, 265-271 (2009). HPTLC of atorvastatin calcium, ramipril, and aspirin and extracts of pharmaceutical formulations on silica gel, prewashed with methanol, with methanol - benzene - ethyl acetate - glacial acetic acid 9:140:100:1 in a twin trough chamber, saturated with mobile phase for 30 min at room temperature. Quantitative determination by absorbance measurement at 210 nm. The limit of detection was 5 ng/zone for atorvastatin calcium, 3 ng/zone for ramipril, and 19 ng/zone for aspirin.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 156 M. PANDE*, Shweta GONDKAR, J. RAO, S. YADAV (*Poona College of Pharmacy, Bharati Vidyapeeth University, Pune, Maharashtra, India): Simultaneous determination of tenofovir and emtricitabine in the bulk drug and tablet dosage form by HPTLC method. Abstract No. F-253, 61st IPC (2009). HPTLC of tenofovir and emtricitabine on silica gel with toluene - methanol - ethyl acetate - acetic acid 40:20:50:1. Quantitative determination by absorbance measurement at 273 nm. The hR_F value was 52 for tenofovir and 40 for emtricitabine. Linearity of tenofovir and emtricitabine was in the range of 120-600 ng/spot and 80-560 ng/spot, respectively. The recovery was 99.9 and 99.5 % for tenofovir and emtricitabine, respectively.
- quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 157 P. PARMAR*, Ankita MEHTA (L. M. College of Pharmacy, Navrangpura, Gujarat 380009, India, parul1383@gmail.com): Development and validation of HPTLC method for the estimation of clotrimazole in bulk drug and tablet formulation. Ind. J. Pharma. Sci. 71(4), 451-454 (2009). HPTLC of clotrimazole in bulk drug and tablet dosage form on silica gel with cyclohexane - toluene - methanol - triethyl amine 80:20:5:2. Quantitative determination by absorbance measurement at 262 nm. The calibration curve was linear between 200 to 1000 ng/spot for clotrimazole. The limit of detection and limit of quantification for clotrimazole were 50 ng/spot and 200 ng/spot, respectively.
- pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 32a
- 104 160 A. PATEL*, B. DHANYA, A. SEN, A. SETH (*Dept. of Pharma., Sumandeep Vidyapeeth University, Vadodara, Gujarat, India): Development and validation of a HPTLC method for estimation of doxazosin mesylate in tablet dosage form. Abstract No. F-251, 61st IPC (2009). HPTLC of doxazosin mesylate on silica gel with acetone - toluene - 25 % ammonia 60:40:1. The hR_F value was 65. Quantitative determination by absorbance measurement at 251 nm. Linearity was in the range of 20-100 ng/band. Recovery was 103.3 %.
- pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a
- 104 159 B. PATEL*, A. MODH, P. MEHTA, H. BHATT (*Institute of Pharmacy, Nirma University Science and Technology, Ahmedabad, Gujarat, India): Development and validation of spectrophotometric and HPTLC method for simultaneous estimation of levocetirizine dihydrochloride and

montelukast sodium in their combined dosage form. Abstract No. F-311, 61st IPC (2009). HPTLC of levocetirizine dihydrochloride and montelukast sodium on silica gel with chloroform - methanol 93:7. The hR_F value was 21 and 65 for levocetirizine and montelukast, respectively. Quantitative determination by absorbance measurement at 345 nm. The method was linear in the range of 100-350 ng/band for levocetirizine and 600-1100 ng/band for montelukast.

pharmaceutical research, quality control, HPTLC, densitometry,
comparison of methods, quantitative analysis

32a

- 104 161 C. PATEL*, B. PATEL, P. PATEL, C. PATEL (*Shri Sarvajanic Pharmacy College, Hemchandracharya North Gujarat University, Mehsana, Gujarat, India): Development and validation of a simultaneous HPTLC method for the estimation of atorvastatin calcium and amlodipine besilate in tablet dosage form. 60th Indian Pharmaceutical Congress PA-213 (2008). HPTLC of atorvastatin calcium and amlodipine besilate on silica gel with chloroform - toluene - methanol - water 55:10:20:2. Quantitative determination by absorbance measurement at 242 nm. The calibration curve was found to be linear between 400 and 1200 ng/spot for both atorvastatin calcium and amlodipine besilate. The limit of detection and the limit of quantification for atorvastatin calcium were 100 and 400 ng/spot, and for amlodipine besilate 200 and 400 ng/spot, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 173 D. PATEL*, S. HEMALATHA, SURESH B., S. DHANABAL (*Dept. of Pharmacognosy. J.S.S. College of Pharmacy, Oacamund 643001, Tamil Nadu, India): Phytochemical standardization and fingerprinting analysis of Berberis aristata extract by HPTLC. Abstract No. 9185, IHCB (2009). HPTLC of hydro alcoholic extracts of Berberis aristata on silica gel with benzene - ethyl acetate - diethyl amine 6:3:1. Detection by spraying with $AlCl_3$ reagent (for estimation of flavonoids) or with Folin Ciocalteu reagent (for total phenolic content). The fingerprint profile was optimized using two different mobile phases: *n*-butanol - acetic acid - water 14:1:5 and *n*-propanol - formic acid - water 90:1:9. The extract showed 6 different spots and was found to contain 13.5 % w/w of berberin.

pharmaceutical research, quality control, herbal, densitometry, HPTLC,
quantitative analysis, postchromatographic derivatization

32e

- 104 162 D. PATEL*, B. SHAH, B. PATEL (*K. B. Institute of Pharmaceutical Education and Research Gandhinagar, Gujarat, India): Simultaneous estimation of atorvastatin calcium, ramipril and aspirin in capsule dosage form by HPTLC. Abstract No. F-245, 61st IPC (2009). HPTLC of atorvastatin (AT) calcium, ramipril (RA) and aspirin (AS) on silica gel with benzene - ethyl acetate - toluene - methanol - acetic acid 40:45:10:5:1. Quantitative determination by absorbance measurement at 220 nm. The hR_F values were 45, 28 and 72 for AT, RA and AS, respectively. The linearity ranges were 0.5-2.5 $\mu\text{g}/\text{band}$ ($r^2=0.998$) for AT, 0.5-2.5 $\mu\text{g}/\text{band}$ ($r^2=0.9978$) for RA and 0.75-3.75 $\mu\text{g}/\text{band}$ ($r^2=0.9946$) for AS with mean recoveries of 100.3, 99.1 and 98.9 for AT, RA and AS, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 164 F. PATEL*, J. DONGA, N. PATEL, V. GANDHI (*Dharmaj Degree Pharmacy College, Dharmaj, Gujarat, India): Determination of camylofin dihydrochloride in bulk and tablet by liquid chromatography and HPTLC. Abstract No. F-267, 61st IPC (2009). HPTLC of camylofin dihydrochloride on silica gel with chloroform - ethyl acetate - methanol - 25 % ammonia 50:30:20:1. Quantitative determination by absorbance measurement at 215 nm. Linearity was in the range of 1500-7500 ng/band. The proposed method is suitable for routine quality control of bulk drug and tablets.

pharmaceutical research, HPTLC, densitometry, quantitative analysis

32a

- 104 165 J. PATEL*, K. BHAT, F. SHAIKH, S. PANDYA (*Babaria Institute of Pharmacy, Varnama, Vadodara, Gujarat, India): Simultaneous determination of strychnine and piperine in their combined

herbal dosage form by HPTLC. 60th Indian Pharmaceutical Congress PA-192 (2008). HPTLC of strychnine and piperine in herbal extracts and herbal formulations on silica gel with toluene - ethyl acetate - diethyl amine 7:2:1. Quantitative determination by absorbance measurement at 283 nm. Linearity was 400-2000 ng/spot, recovery was in the range of 99.5-101.0 both for strychnine and piperine.

herbal, HPTLC, densitometry, quantitative analysis

32e

- 104 158 J.B. PATEL, S.K. LAHIRI, M.B. SHAH* (*Department of Pharmacognosy, L. M. College of Pharmacy, Navarangpura, Ahmedabad (Gujarat), 380009, India; mbshah2007@rediffmail-com): Development of a new method for identification of *Withania somnifera* root, and a method for quantitative analysis of withaferin A in young and old roots. *J. Planar Chromatogr.* 22, 283-286 (2009). HPTLC of withaferin and extracts of the powdered root on silica gel, prewashed with methanol, with toluene - ethyl acetate - acetone 2:3:3 in a twin trough chamber saturated with mobile phase for 30 min. Detection by spraying with anisaldehyde reagent followed by heating for 15 min at 105 °C; characteristic orange fluorescence was observed for withaferin. Quantitative determination by absorbance measurement at 214 nm. The limit of detection and quantification for withaferin A was 258 and 782 ng/zone, respectively.

traditional medicine, herbal, quality control, HPTLC, densitometry, quantitative analysis 32e

- 104 170 M. PATEL *, P. MANDLEKAR, S. MULGUND, K. JAIN (*Sinhgad College of Pharmacy, Pune, Maharashtra, India): Simultaneous HPTLC determination of ramipril hydrochlorothiazide and telmisartan in combined tablets. Abstract No. F-239, 61st IPC (2009). HPTLC of hydrochlorothiazide, ramipril and telmisartan on silica gel with ethyl acetate - chloroform - methanol 6:3:1. Quantitative determination by absorbance measurement at 215 nm. The method was suitable for routine quality control of combined formulations.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 163 N. PATEL*, G. PATEL, H. BHATT, C. SHASTRY (*Shree Dhanvantry Pharmacy College, Surat, Gujarat, India): HPTLC method for simultaneous determination of aspirin and atorvastatin in pharmaceutical formulation. Abstract No. F-293, IPC (2009). HPTLC of aspirin and atorvastatin on silica gel with *n*-hexane - acetone - butyl acetate - formic acid 60:30:12. Quantitative determination by absorbance measurement at 242 nm. For both drugs, the method was linear in the range of 3-7 µg/band and the recovery was between 99.3 and 101.0 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 171 P. PATEL*, R. MASHRU (*Babaria Institute of Pharmacy, Vadodara, Gujarat, India): Two dimensional thin layer chromatography (2D-TLC) for resolution of isomers of (±) bupropion HCl. Abstract No. F-254, 61st IPC (2009). HPTLC of bupropion HCl on silica gel with quinine sulphate - methanol - water 13:20:12 in the first direction. Quinine sulphate was used as a chiral selector at a concentration of 3.5 mM. The two separated bands were detected under UV 366 nm. The hR_F values of I (-) and d (+) isomers of bupropion HCl were 90 and 84, respectively. After the second run in the second dimension with methanol - water 80:13 they were better resolved with hR_F values of 88 and 76.

HPTLC, qualitative identification

32a

- 104 172 P. PATEL*, R. MASHRU, T. PATEL (*Babaria Institute of Pharmacy, Varnama, Vadodara, Gujarat, India): Development and validation of a direct HPTLC method for separation of isomers of (±) bupropion HCl using quinine sulphate as a chiral selector in mobile phase. 60th Indian Pharmaceutical Congress PA-216 (2008). HPTLC of isomers of bupropion HCl on silica gel with quinine sulphate - methanol - water 13:20:12 (quinine sulphate served as a chiral selector). Evaluation under UV 366 nm. Linearity was in the range of 10-100 µg/spot for d(+)- and l(-)-isomers of bupropion. The isomer ratio was 80 % d(+)-bupropion and 20 % l(-)-bupropion.

pharmaceutical research, quality control, HPTLC, densitometry,
quantitative analysis

32a

- 104 169 P. PATEL*, N. PATEL, R. GOYAL (*Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa, Gujarat, India): Quality control of polyherbal formulations used in diabetes mellitus. 60th Indian Pharmaceutical Congress PG-246 (2008). HPTLC of biomarkers such as curcumin, charantin, and swertiamarin in some polyherbal formulations on silica gel with benzene - methanol 4:1 (for charantin), chloroform - methanol - formic acid 74:4:1 (for curcumin), and ethyl acetate - methanol - water 77:15:5 (for swertiamarin). Quantitative determination by absorbance measurement at 536 nm for charantin (hR_F value 33), 425 nm for curcumin (hR_F value 89), and 238 nm for swertiamarin (hR_F value 54).

traditional medicine, quality control, HPTLC, densitometry, quantitative analysis

32e

- 104 167 R. PATEL*, Mrunali PATEL, K. BHATT, B. PATEL (*A. R. College of Pharmacy and G. H. Patel Institute of Pharmacy, Vallabh Vidyanagar, Gujarat, India): Development and validation of HPTLC method: its application in qualification of olanzapine in mucoadhesive microemulsion formulations and invitro study. Abstract No. F-235, 61st IPC (2009). HPTLC of olanzapine on silica gel with methanol - ethyl acetate 4:1. The hR_F value was 35. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 100-600 ng/band. The method was suitable for analysis of formulations and in-house prepared mucoadhesive microemulsions.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis

32a

- 104 166 R.B. PATEL*, M.R. PATEL, M.B. SHANKAR, K.K. BHATT (*Sardar Patel University, A. R. College of Pharmacy & G. H. Patel Institute of Pharmacy, Vallabh Vidyanagar 388120, Gujarat, India; rashmru@gmail.com): Simultaneous determination of alprazolam and fluoxetine hydrochloride in tablet formulations by high-performance column liquid chromatography and high-performance thin-layer chromatography. J. AOAC Int. 92, 1082-1087 (2009). HPTLC of alprazolam and fluoxetine hydrochloride in pure powder and formulations on silica gel with acetone - toluene - ammonia 12:7:1 in a twin trough chamber saturated for 30 min. Quantitative determination by absorbance measurement at 230 nm. There was no significant difference in the determined content of alprazolam and fluoxetine by HPTLC and HPLC methods (assay results compared by applying the paired t-test).

pharmaceutical research, quality control, HPTLC, densitometry,
quantitative analysis, comparison of methods

32a

- 104 168 S. PATEL*, N. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganapat University, Mehsana, Gujarat, India): HPTLC estimation of amitriptyline HCl, trifluoperazine HCl, risperidone and alprazolam in pharmaceutical products using single mobile phase. 60th Indian Pharmaceutical Congress PA-210 (2008). HPTLC of amitriptyline HCl, trifluoperazine HCl, risperidone and alprazolam on silica gel with carbon tetrachloride - acetone - triethylamine 80:20:3. Quantitative determination by absorbance measurement at 250 nm in the range of 50-1200 ng/spot for amitriptyline HCl, 50-1200 ng/spot for trifluoperazine HCl, 100-2400 ng/spot for risperidone, and 25-600 ng/spot for alprazolam. The limit of quantification for amitriptyline HCl and trifluoperazine HCl was 50 ng/spot, for risperidone 100 ng/spot, and for alprazolam 25 ng/spot.

pharmaceutical research, quality control, densitometry, quantitative analysis, HPTLC

32a

- 104 174 P. PATIDAR *, S. MANIMARAN, N. SONI, S. DHANABAL (*J S S College of Pharmacy, Ooty, Tamil Nadu, India): Simultaneous HPTLC estimation of quercetin and rutin from *Tylophora indica* and *Tephrosia purpurea*. 60th Indian Pharmaceutical Congress PG-261 (2008). HPTLC of quercetin and rutin in ethanolic extracts of aerial parts of *Tylophora indica* and *Tephrosia purpurea* on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:26. Quantitative determination by fluorescence measurement at 366-400 nm. The extract of *Tephrosia purpurea*

contained 1.56 % of quercetin and 1.40 % of rutin, whereas *Tylophra indica* contained 4.30 % of quercetin.

herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 175 N.G. PATRE, L. SATHIYANARAYANAN, M.V. MAHADIK, S.R. DHANESHWAR* (*Department of Pharmaceutical Chemistry, Bharati Vidyapeeth University, Poona College of Pharmacy, Center for Advanced Pharmaceutical Research, Erandwane, Pune 411038, Maharashtra State, India; Sunil.dhaneshwar@gmail.com): A validated, stability-indicating HPTLC method for analysis of doxofylline. *J. Planar Chromatogr.* 22, 245-348 (2009). HPTLC of doxofylline (7-(1,3-dioxalan-2-ylmethyl)theophylline) in bulk drug and in formulations on silica gel, prewashed with methanol, with toluene - methanol 4:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 275 nm.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 176 M. PENG (Peng Minjie)*, D. LI (Li Duowei), Y. WANG (Wang Yichao), SH. JIA (Jia Shaoliang) (*Inst. Life Sci., North-West University, Xi'an 710069, China): (Determination of scalreol in its formulations by thin-layer chromatography) (Chinese). *Chinese J. Pharm. Anal.* 28 (9), 1554-1556 (2008). TLC of scalreol on silica gel with *n*-hexane - ethyl acetate - formic acid 32:16:1. Detection by spraying with vanillin - sulfuric acid - ethanol 1:1:18. Quantification by densitometry at 520 nm. Linearity was between 10 and 50 µg/zone with a determination coefficient of 0.9994. Recovery was 100.1 % (n = 6, RSD = 0.9 %). Repeatability (%RSD, n = 6) was 2.1 % within plate and 2.3 % plate-to-plate.

pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis, qualitative identification 32c

- 104 177 CH. PIAO (Piao Chunmei)*, X. QU (Qu Xiangling), X. ZHOU (Zhou Xunrong) (*Affiliated Hosp. No.2, Guizhou Inst. TCM, Guiyang, Guizhou 550003, China): (Identification procedure for Tongmaitang Yanming capsules) (Chinese). *Chinese J. Hosp. Pharm.* 29 (4), 1246-1247 (2009). TLC of TCM drug extracts on silica gel with 1) chloroform - methanol - acetone 10:1:1; 2) toluene - chloroform - acetone - methanol - formic acid 4:6:8:1:4; 3) chloroform - methanol 4:1. Detection 1) after exposure to ammonia vapor under UV 254 nm; 2) under UV 254 nm; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration.

pharmaceutical research, quality control, traditional medicine, qualitative identification 32e

- 104 178 L. POTALE*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Kenned Raod, Pune, Maharashtra, India): A validated stability indicating HPTLC method for simultaneous estimation of telmisartan and rampril. Abstract No. F-271, 61st IPC (2009). HPTLC of telmisartan and rampril on silica gel with methanol - chloroform 1:6. The hR_F value was 38 and 68 for rampril and telmisartan, respectively. Quantitative determination by absorbance measurement at 210 nm. The sample was exposed to different stress conditions (acid, alkali, oxidative, photo degradation and thermal). Both drugs did not degrade under acidic and photolytic conditions, but showed significant degradation under alkaline and thermal conditions. Both compounds were well separated from different degradation products under experimental conditions.

pharmaceutical research, quality control, densitometry, HPTLC, quantitative analysis 32a

- 104 179 Kirti PRABHU*, R. LOBO, Richa AGRAWAL, A. SHIRWAIKAR, A. SHIRWAIKAR, Mamatha BALLAL (*Dept. of Pharmacognosy, Manipal College of Pharmaceutical Science, Manipal University, Manipal, India): Application of a stability-indicating HPTLC method for the quantitative determination of hesperidin in pharmaceutical dosage form. Abstract No. 9324, IHC (2009). HPTLC of hesperidin in orange peel extract and formulation on silica gel with ethyl acetate - methanol - water 100:17:13. Quantitative determination by absorbance measurement at 287 nm. The method was linear in the range of 10-1000 ng/spot. Hesperidin was subjected to degradation studies (acid, alkali, hydrolysis, oxidation, and thermal stress) and was found susceptible to diffe-

rent stress condition. The method was suitable for determination of hesperidin and its degradation products in bulk drug as well as formulations.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis

32e

- 104 180 S.L. PRABU*, T. SINGH, C.D. KUMAR, A. JOSEPH, K.K. SRINAVASAN (*Manipal College of Pharmaceutical Sciences, Manipal 576104, India; slaxmanvel@gmail.com): High-performance thin-layer chromatographic method for analysis of racecadotril in the bulk drug. *J. Planar Chromatogr.* 22, 277-281 (2009). HPTLC of racecadotril (2-{2(acetylsulfanylmethyl)-3-phenylpropanoyl}amino acetic acid benzyl ester) and its degradation products in the bulk drug and in a pharmaceutical formulation on silica gel with *n*-hexane - ethyl acetate 7:3 in a twin trough chamber saturated for 30 min. Quantitative determination by absorbance measurement at 230 nm. The limit of detection and quantification was 50 and 100 ng/band, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 181 N. PRAJAPATI*, V. YADAV, S. PANCHOLI (*S. K. Patel College of Pharmaceutical Education and Research, Mehsana, Gujarat, India): Development and validation of HPTLC method for determination of repaglinide and rosiglitazone maleate in combined dosage form. Abstract No. F-263, 61st IPC (2009). HPTLC of repaglinide (REPA) and rosiglitazone (ROSI) on silica gel with benzene - methanol - acetone - acetic acid 80:10:9:1. Quantitative determination by absorbance measurement at 266 nm. Linearity was in the range of 800-2800 ng/band and 400-2400 ng/band for REPA and ROSI, respectively. Recovery was 101.7 and 99.5 % for REPA and ROSI, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 182 P. PUSHPALATHA*, R. K. SARIN, M. A. RAO, T. R. R. BAGGI (*Central Forensic Science Laboratory, Directorate of Forensic Science, Ministry of Home Affairs, Government of India, Ramanthapur, Hyderabad 500 013, India; sarinrk@yahoo.com): A new thin-layer chromatographic method for analysis of zolpidem and zopiclone. *J. Planar Chromatogr.* 22, 449-451 (2009). TLC of zolpidem and zopiclone on silica gel with methanol - triethylamine 39:1, acetonitrile - triethylamine 39:1, chloroform - methanol - triethylamine 38:2:1, and acetonitrile - methanol - triethylamine 34:4:1 with chamber saturation. Detection by spraying with chloranilic acid reagent (0.5 chloranilic acid in dioxane) and evaluation of colored zones in daylight.

toxicology, qualitative identification 32a

- 104 183 Y. QI (Qi Yanfei)*, Q. GONG (Gong Qing), M. LU (Lu Min) (*Zhejiang Provin. Inst. Food & Med., Hangzhou, Zhejiang 310004, China): (Study of the quality standard for Qingshen Jianfei pills) (Chinese). *J. Chinese Trad. & Herb. Med.* 39 (12), 1818-1829 (2008). TLC of the extracts of the TCM drug on silica gel with 1) chloroform - methanol - acetone - ammonia 2:1:4:4; 2) chloroform - methanol - acetone - formic acid 32:8:4:7; 3) ethyl acetate - butanone - formic acid - water 10:1:1:1; 4) chloroform - methanol - water 13:7:2. Detection by evaluation under UV 365 nm and after spraying with 1) potassium iodobismuthate reagent; 2) a 1:1 mixture of 1 % potassium ferricyanide and 1 % FeCl₃; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration.

pharmaceutical research, traditional medicine, quality control, qualitative identification 32c

- 104 184 T. QU (Qu Tingli), Y. DENG (Deng Yaning), L. HAU (Hau Lihong), ZH. ZHAO (Zhao Zhengbao)* (*Pharm. Coll., Shanxi Univ. Med., Taiyuan Shanxi 030001, China): (Identification of Shenlingbaizhu pills by thin-layer chromatography) (Chinese). *J. Chinese Trad. Patent Med.* 30 (12), Supl. 4-6 (2008). TLC of the TCM drug extracts on silica gel with 1) dichloromethane - ethyl acetate - methanol - water 15:40:22:10; 2) petroleum ether (60-90 °C) - diethyl ether 3:2; 3) petroleum ether (60-90 °C) - ethyl acetate 25:2; 4) *n*-butanol - acetic acid - water 4:1:2; 5) chloroform - diethyl ether 1:1. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed

by heating at 105 °C until coloration; 2) under UV 365 nm; 3) under UV 254 nm.

pharmaceutical research, traditional medicine, quality control, qualitative identification 32c

- 104 185 P. RAJA* , K. BHATT, V. JOSHI, K. AMIN (*A. R. College of Pharmacy and G. H. Patel Institute of Pharmacy, Anand, Gujarat, India): Development and validation of HPTLC method for simultaneous estimation of olmesartan medoxomil and hydrochlorothiazide in their combined tablet dosage form. 60th Indian Pharmaceutical Congress PA-232 (2008). HPTLC of olmesartan medoxomil and hydrochlorothiazide on silica gel with methanol - toluene - ethyl acetate 5:11:4 with chamber saturation for 30 min. The hR_F value of olmesartan medoxomil was 27 and of hydrochlorothiazide 44. The method was linear in the range of 300-1800 ng/spot for both drugs.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 186 K.P. RANDAU, S. SPROLL, H. LERCHE, F. BRACHER* (*Department Pharmazie - Zentrum für Pharmaforschung, Ludwig-Maximilians-Universität, Butenandtstr. 5-13, 81377 München, Germany; Franz.Bracher@cup.uni-muenchen.de): Pernambucone, a new tropone derivative from *Croton argyroglossum*. Pharmazie 64, 350-351 (2009). Preparative TLC of pernambucone (3,8-dimethyl-5-isopropyl-2,3-dihydro-1H-azulene-1,6-dione), orobanone and extracts of stem bark on silica gel with hexane - ethyl acetate 4:1 and dichloromethane - methanol 39:1. Detection in visible light.

pharmaceutical research, herbal, preparative TLC 32e

- 104 187 P. RAO*, R. KUMAR, G. REDDY, R. BABOO (*A. M. Reddy Memorial College of Pharmacy, Guntur, A.P., India): Method development and validation of HPTLC method for estimation of quetiapine in bulk drugs and in tablet dosage form. Abstract No. F-261, 61st IPC (2009). HPTLC of quetiapine on silica gel with methanol - toluene 4:3. The hR_F value was 41. Quantitative determination by absorbance measurement at 235 nm. The method was linear in the range of 100-500 ng/band. Recovery was 98.9 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 188 J. RAO*, S. YADAV, M. APNDE, S. GONDKAR (*Bharati Vidyapeeth University, Poona college of Pharmacy, Pune, Maharashtra, India): Stability indicating HPTLC method for tenofovir in the bulk drug and tablet dosage form. Abstract No. F-252, 61st IPC (2009). HPTLC of tenofovir on silica gel with *n*-butanol - acetic acid - water 4:1:1. The hR_F value was 58. Quantitative determination by absorbance measurement at 260 nm. Linearity was in the range of 120-600 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidation, photodegradation and thermal) and degradations products were well resolved from the main component.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 189 K. REMYA*, A. BINDU, N. ALEYKUTTY, J. SAJAN (*Department of Pharmaceutical Science, Cheruvandoor Campus, Ettumanoor, Kottayam, Keral, India): High-performance thin-layer chromatographic method for quantitative determination of quercetin in tender leaves of *Psidium guajava*. 60th Indian Pharmaceutical Congress PG-264 (2008). HPTLC of quercetin in acetone extracts of *Psidium guajava* leaves on silica gel with toluene - acetone - formic acid 38:10:5. Quantitative determination of quercetin by absorbance measurement at 364 nm. The correlation coefficient was 0.9847. There was a good correlation between peak area and corresponding concentration of quercetin. The proposed HPTLC method provided a good resolution of quercetin from other constituents present in acetone extract of tender leaves of *Psidium guajava* and can be used for the quantification of quercetin.

herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 190 A. REN (Ren Ainu)*, Y. LI (Li Yun), M. JU (Ju Mingqiao) (*Jiangsu Provin. Acad. Med. & Pharm., Nanjing, 210028, China): (Study of the quality standard for Baozhi pills, a Chinese

traditional patent medicine) (Chinese). Chinese J. Pharm. Anal. 28 (1), 20-23 (2008). TLC of TCM drug extracts on silica gel with toluene - ethyl acetate - methanol - isopropanol - ammonia 12:6:3:3:1. Detection under UV light. The method is suitable for quality control of Baozhi pills.

pharmaceutical research, traditional medicine, quality control, herbal,
quantitative analysis, qualitative identification

32c

104 191 A. RICHA*, S. KIRTI, L. RICHARD, S. ANNIE (*Dept. of Pharmacognosy, Manipal College of Pharmaceutical Science, Manipal, Karnataka 576104, India): Pharmacognostical, phytochemical and HPTLC fingerprinting evaluation of *Dendrophthoe falcata* leaf. Abstract No. 9712, IHC B (2009). HPTLC of extracts from leaves of *Dendrophthoe falcata* on silica gel with methanol - formic acid - water 40:3:57. Quantitative determination by absorbance measurement at 280 nm using quercetin as marker.

pharmaceutical research, quality control, herbal, HPTLC, densitometry,
quantitative analysis

32e

104 192 K.K. ROUT*, O.P. ROUT, S.K. MISHRA (*Pharmacognosy and Phytochemistry Division, University Department of Pharmaceutical Sciences, Utkal University, Vani Vihar, Bhubaneswar 751004, Orissa, India; kd_rout@yahoo.co.in): Standardization of Ayurvedic formulations containing *Aloe vera* by quantification of a marker compound. J. Planar Chromatogr. 22, 381-384 (2009). TLC of aloin in commercial Ayurvedic preparations on silica gel (prewashed with methanol) with ethyl acetate - methanol - water 50:7:2 in a twin trough chamber with chamber saturation for 5-7 min at 30 +/- 4 °C and a relative humidity of 57 +/- 3 %. Quantitative determination by absorbance measurement at 360 nm. The limit of detection and quantification was 10 and 20 ng/band, respectively.

traditional medicine, herbal, quality control, quantitative analysis, densitometry

32e

104 193 P. SAINI*, C. JAIN, R. SINGH, S. MATHUR, G. SINGH, M. NASLAM (*Research & Development Div., Indian Pharmacopoeia Commission, Govt. of India, Ministry of Health & Family Welfare, Sector-23, Rajnagar, Ghaziabad 201002, India, ipclab@vsnl.net): A simple and sensitive HPTLC method for simultaneous determination of abacavir sulphate and lamivudine in tablet dosage form. J. Pharma. Research 8(4), 187-191 (2009). HPTLC of abacavir sulphate and lamivudine on silica gel with methanol - acetone - *n*-butyl acetate 1:1:2. Quantitative determination by absorbance measurement at 284 nm. The hR_F value of abacavir sulphate was 58 and of lamivudine 35. Linearity of abacavir sulphate and lamivudine was in the range of 240-1200 ng/spot and 120-600 ng/spot, respectively. The limit of detection and quantification of abacavir sulphate was 0.7 and 2 ng/spot, respectively, and of lamivudine 1 and 3 ng/spot, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis

32e

104 194 A. SARASWATHY*, D. RAMASAMY, S. ARIMASAMY, D. NANDINI (*CSM Drug Research Inst. for Ayurveda and Siddha, Anna Hospital Campus, Arumbakkam, Chennai 600106, saraswathy20042000@yahoo.co.in): Development of HPTLC profile and heavy metal analysis of stem bark of three *Ficus* species. Indian Drugs 46(6), 493-496 (2009). HPTLC of chloroform extracts of the bark of *Ficus racemosa*, *F. bengalensis*, and *F. religiosa* on silica gel with toluene - ethyl acetate - formic acid 90:10:1. Detection under UV 254 nm and visible light after treatment with vanillin sulfuric acid reagent, followed by heating at 105 °C until coloration.

herbal, HPTLC, qualitative identification

32e

104 195 N. SARATHI*, M. GANDHIMATHI, R. SAKTHI, T. RAVI (*Sri Ramakrishna Institute of Paramedical Science, Coimbatore, Tamil Nadu, India): A rapid HPTLC analysis of oxcarbazepine in human plasma. 60th Indian Pharmaceutical Congress PA-227 (2008). HPTLC of oxcarbazepine (in acetonitrile extracts of human plasma) on silica gel with ethyl acetate - toluene - methanol 7:2:1. The hR_F values of oxcarbazepine and the internal standard chlorzoxazone were 54 and 86, respectively. The linearity range was 10-300 ng/mL, recovery from plasma was 75.2 %.

quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 196 S. SATHE*, S. BARI (*Department of Pharmaceutical Chemistry, R. C. Patel College of Pharmacy, Karwand Naka, Shirpur Dhule, Maharashtra 425405, India, sbbari@rediffmail.com, shitalathe@rediffmail.com): Quantitative analysis of losartan potassium and atenolol by high-performance thin-layer chromatography. Indian Drugs 46(1), 78-81 (2009). HPTLC of atenolol and losartan potassium in tablets on silica gel with toluene - methanol - triethylamine 12:8:1 with chamber saturation for 45 min. Quantitative determination by absorbance measurement at 230 nm. The hR_F value of atenolol was 45 and of losartan potassium 67. The method was linear in the range of 1000-4000 ng/spot for both compounds. The recovery was 98.8-98.9 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 197 Sunita SEERAPU*, B. SRINIVASAN (*Delhi Institute of Pharmaceutical Sciences and Research (DIPSAR), New Delhi, India): Development and validation of analytical method on HPTLC for the determination of ivabradine hydrochloride as bulk drug and in pharmaceutical formulations. Abstract No. F-275, 61st IPC (2009). HPTLC of ivabradine HCl on silica gel with methanol - chloroform 1:1. The hR_F value was 59. Quantitative determination by absorbance measurement at 285 nm. Linearity was in the range of 100-800 ng/spot with $r^2=0.9989$ (via peak area).

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 198 M. SHAH*, A. PRAJAPATI, S. PATEL, N. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganpat University, Ganpat Vidyanagar, Mehsana, Gujarat, India): Estimation of voriconazole in powder by HPTLC method. 60th Indian Pharmaceutical Congress PA-231 (2008). HPTLC of voriconazole on silica gel with toluene - ethyl acetate 1:3. Quantitative determination by absorbance measurement at 255 nm. The linearity range was 10-1200 ng/spot, recovery was 99.5 %. The method was suitable for routine quality control of formulations.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 199 N. SHAH*, B. SUHAGIA, R. SHAH, N. PATEL (*Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa 383315): HPTLC method for the simultaneous estimation of valsartan and hydrochlorothiazide in tablet dosage form. Ind. J. Pharma. Sci. 71(1), 72-74 (2009). HPTLC of valsartan and hydrochlorothiazide on silica gel with chloroform - methanol - toluene - acetic acid 60:20:10:1. Quantitative determination by absorbance measurement at 260 nm. The calibration curve was linear between 300 to 800 ng/spot for valsartan and 100 to 600 ng/spot for hydrochlorothiazide. The limit of detection and the limit of quantification for valsartan were 100 and 300 ng/spot, respectively, and for hydrochlorothiazide 30 and 100 ng/spot, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 200 R. SHAH*, H. BHATT, G. PATEL, C. SHARASTRY (*Shree Dhanvantary Pharmacy College, Surat, Maharashtra, India): Simultaneous estimation of mosapride citrate and pantoprazole in solid dosage form by HPTLC method. Abstract No. F-236, 61st IPC (2009). HPTLC of pantoprazole and mosapride citrate on silica gel (pre-washed with methanol) with ethyl acetate - benzene - methanol - 25 % ammonia 48:35:15:2 at room temperature. Quantitative determination by absorbance measurement at 250 nm or 276 nm. The method was linear in the range of 3-7 $\mu\text{g}/\text{band}$ for both drugs. Recovery was between 99.0 and 103.5 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 201 R. SHAH*, H. PANCHAL, B. SUHAGLA, N. PATEL (*S. K. Patel College of Pharma. Education & Research, Mehsana, Gujarat, India): Simultaneous determination of atorvastatin calcium, ramipril and aspirin in capsule dosage form by HPTLC. 60th Indian Pharmaceutical Congress PA-209 (2008). HPTLC of atorvastatin calcium, ramipril and aspirin on silica gel with methanol - benzene - ethyl acetate - glacial acetic acid 9:140:100:1. Quantitative determination by absor-

bance measurement at 210 nm. The hR_F value of ramipril was 6, of atorvastatin 38, and of aspirin 86. Linearity was 100-600 ng/band (atorvastatin), 50-300 ng/band (ramipril) and 500-3000 ng/band (aspirin). Recovery was 99.9-100.0 % for all three compounds. Salicylic acid, an impurity of aspirin, was observed in capsule dosage form with an hR_F value of 72. The method was suitable for analysis of combined dosage form.

pharmaceutical research, quality control, densitometry, quantitative analysis, HPTLC 32a

- 104 202 K. SHAH*, I. SOJITRA, R. SHAH, U. VACHHANI (*Rofel Shri G. M. Bilakhia College of Pharmacy, Vpi, Gujarat, India): Development and validation of HPTLC analytical method for determination of L-dopa in *Mucuna pruriens* powdered extract and polyherbal formulations. Abstract No. F-262, 61st IPC (2009). HPTLC of L-dopa on silica gel with *n*-butanol - water - acetic acid 4:1:1. The hR_F was 37. Detection by spraying with 0.5 % ethanolic ninhydrin solution, followed by heating at 120 °C for 2 min. Quantitative determination by absorbance measurement at 520 nm. The method was linear in the range of 600-1400 ng/band.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32e

- 104 203 N. SHARMA, U. SHARMA, A. GUPTA, DEVLAA, A. SINHA*, B. LAL, P. AHUJA (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology (CSIR), Palampur, Himachal Pradesh, India, aksinha08@rediffmail.com) : Simultaneous densitometric determination of shikonin, acetylshikonin, and beta-acetoxyisovalerylshikonin in ultrasonic-assisted extracts of four *Arnebia* species using reversed-phase thin layer chromatography. J. Sep. Sci. 32, 3239-3245 (2009). HPTLC of shikonin (1), acetylshikonin (2), and beta-acetoxyisovalerylshikonin (3) in four species of *Arnebia* on RP-18 with acetonitrile - methanol - 5 % formic acid 20:1:4. Quantitative determination by absorbance measurement at 520 nm. Linearity was in the range of 100-600 ng/zone for (1) and (2) and 100-1800 ng/zone for (3). The limits of detection for (1), (2) and (3) were 18, 15 and 12 ng/zone, respectively, while the limits of quantification were 60, 45 and 40 ng/zone, respectively.

herbal, traditional medicine, HPTLC, quantitative analysis, densitometry 32e

- 104 204 A.A. SHIRKHEDKAR*, P.M. BUGDANE, S. SURANA (*R.C. Patel College of Pharmacy, Shirpur Dist: Dhule, (M.S.) 425 405 India): Stability-indicating TLC-densitometric determination of neбиволol hydrochloride in bulk and pharmaceutical dosage form. J. Chromatogr. Sci. 48 (2), 109-113 (2010). HPTLC of neбиволol hydrochloride on silica gel with toluene - methanol - triethylamine 19:6:1. The hR_F value of neбиволol hydrochloride was 33. Quantification by densitometry in the absorbance mode at 281 nm. Linearity was between 500 and 3000 ng/spot with $r^2=0.9994$. The limit of detection and quantification was 63 and 191 ng/spot, respectively. Neбиволol hydrochloride was subjected to acid and alkali hydrolysis, oxidation, thermal degradation, and photodegradation. The degradation products were well-resolved from the main component.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis, qualitative identification 32c

- 104 205 A.A. SHIRKHEDKAR*, S. J. SURANA (*Department of Pharmaceutical Chemistry, R. C. Patel Institute of Pharmaceutical Education and Research, Shirpur Dist: Dhule (M.S.) India 425 405; atulshirkhedkar@rediffmail.com; sjsurana@yahoo.com): Simultaneous densitometric TLC analysis of atorvastatin calcium and fenofibrate in the bulk drug and in pharmaceutical formulations. J. Planar Chromatogr. 22, 355-358 (2009). TLC of atorvastatin calcium and fenofibrate on silica gel, prewashed with methanol, with toluene - methanol - triethylamine 35:15:1 in a twin trough chamber saturated for 25 min at room temperature and relative humidity of 60 +/- 5 %. Quantitative determination by absorbance measurement at 258 nm. The limit of detection and quantification for atorvastatin calcium was 25 and 77 ng/zone, respectively and for fenofibrate 292 and 886 ng/zone, respectively.

pharmaceutical research, quality control, densitometry, quantitative analysis 32a

- 104 206 A.R. SHRIVASTAVA, C.R. BARHATE, C.J. KAPADIA* (*Department of Pharmaceutics, Bombay College of Pharmacy, Kalina, Santacruz, Mumbai 400 098, India; drcjkapadia@gmail.com): Stress degradation studies in valsartam using validated stability-indicating high-performance thin-layer chromatography. *J. Planar Chromatogr.* 22, 411-416 (2009). HPTLC of valsartan in bulk drug and in formulations on silica gel (prewashed with methanol) with toluene - ethyl acetate - methanol - formic acid 60:20:20:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 250 nm. The limit of detection and quantification was 25 and 150 ng/band, respectively.
pharmaceutical research, quality control, HPTLC 32a
- 104 207 N. SINGH, S. KHATOON*, N. SRIVASTAVA, A.K. SINGH RAWAT, S. MEHROTA (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Rana Pratp Marg, Lucknow 226001, India; neha_somvanshi@yahoo.com, sayyadak@yahoo.com): Qualitative and quantitative standardization of *Myrica esculenta* Buch.-Ham stem bark by use of HPTLC. *J. Planar Chromatogr.* 22, 287-291 (2009). HPTLC of the biomarkers gallic acid, lupeol, oleanolic acid, and stigmaterol and plant extracts on silica gel with toluene - ethyl acetate - formic acid 5:5:1 for gallic acid and with toluene - ethyl acetate 4:1 for lupeol, oleanolic acid, and stigmaterol in a saturated twin trough chamber. Quantitative determination by absorbance measurement at 272 nm. Detection of oleanolic acid, lupeol, and stigmaterol by dipping in anisaldehyde reagent followed by heating at 110 °C for 5 min. Densitometric evaluation at 652 nm.
traditional medicine, herbal, quality control, HPTLC, quantitative analysis, densitometry 32e
- 104 209 B. SINGH*, S. ANANDJIWALA, M. NIVSARKAR (*National Institute of Pharmaceutical Education and Research, Ahmedabad, Gujarat, India): TLC densitometric analysis of glycyrrhizin, glycyrrhetic acid, apigenin, kaempferol and quercetin from *Glycyrrhiza glabra* using HPTLC. 60th Indian Pharmaceutical Congress PG-323 (2008). HPTLC of glycyrrhizin, apigenin and kaempferol in methanolic extracts and glycyrrhetic acid and quercetin in hydrolyzed extracts of *Glycyrrhiza glabra* on silica gel with ethyl acetate - methanol - acetic acid - water 40:5:5:10 (for glycyrrhizin), toluene - ethyl acetate - methanol - formic acid 30:15:1:2 (for kaempferol and quercetin), ethyl acetate - ethanol - water - ammonia 65:20:4:1 (for glycyrrhetic acid). Quantitative determination by absorbance measurement at 254 nm (glycyrrhetic acid and apigenin), 258 nm (glycyrrhizin) and 280 nm (kaempferol and quercetin). The plant was found to contain 1.07 % glycyrrhizin, 0.64 % glycyrrhetic acid, 0.007 % apigenin, 0.03 % kaempferol and 0.24 % quercetin.
HPTLC, densitometry, quantitative analysis 32e
- 104 208 K. SINGH*, S. AGRAWAL, M. GUPTA (*Delhi Institute of Pharmaceutical Sciences and Research, New Delhi, India): Development and validation of improved HPTLC method for simultaneous determination of curcumin, demethoxycurcumin and bis-demethoxycurcumin. 60th Indian Pharmaceutical Congress PA-223 (2008). HPTLC of curcumin, demethoxycurcumin and bis-demethoxycurcumin on silica gel with chloroform - methanol 19:1. The hR_F values were 25, 38, and 61 for bis-demethoxycurcumin, demethoxycurcumin, and curcumin respectively. Quantitative determination by absorbance measurement at 420 nm. The method was linear in the range of 50-400 ng/spot (curcumin), 10-150 ng/spot (demethoxycurcumin), and 5-40 ng/spot (bis-demethoxycurcumin). Recovery was in the range of 99.2-100.5 % for all three compounds.
traditional medicine, quality control, herbal, HPTLC, densitometry, preparative TLC, quantitative analysis 32e
- 104 210 P. SINHA*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Pune, Maharashtra, India): A validated stability indicating method for determination of aspirin and clopidogrel bisulphate. 60th Indian Pharmaceutical Congress PA-208 (2008). TLC of aspirin and clopidogrel bisulphate on silica gel with carbon tetrachloride - acetone 5:2. The hR_F value of aspirin was 15 and of clopidogrel bisulphate 80. Quantitative determination by absorbance measurement at 220 nm. The method was linear in the range of 2-6 µg/spot for aspirin and 3-6 µg/spot for clopidogrel.

pharmaceutical research, quality control, densitometry, quantitative analysis 32a

- 104 211 Krystyna SKALICKA-WOZNIAK*, M. L. HAJNOS, K. GLOWNIAK (* Department of Pharmacognosy with Medicinal Plant Laboratory, Medical University of Lublin, Chodzki 1, 20-093 Lublin, Poland; kskalicka@pharmacognosy.org): High-performance thin-layer chromatography combined with densitometry for quantitative analysis of chlorogenic acid in fruits of *Peucedanum alsaticum* L. *J. Planar Chromatogr.* 22, 297-300 (2009). HPTLC of chlorogenic acid and of plant extracts on silica gel with ethyl acetate - formic acid - water 10:2:3 and ethyl acetate - formic acid - acetic acid - water 100:11:11:21 in a saturated horizontal chamber. Quantitative determination by absorbance measurement at 320 nm. Qualitative detection by derivatization with natural products reagent (1 % in methanol) followed by treatment with 5 % PEG 400 in ethanol.

herbal, quality control, HPTLC, densitometry, quantitative analysis 32e

- 104 212 Danuta SOBOLEWSKA*, Z. JANECZKO, I. PODOLAK, A. SZERLOMSKA (*Department of Pharmacognosy, Jagiellonian University, Collegium Medicum, Medyczna 9, 30-688 Cracow, Poland; dsobolew@cm-uj.krakow.pl): Densitometric analysis of diosgenin in methanolic extracts of *Allium ursinum* collected at different times during plant development. *J. Planar Chromatogr.* 22, 305-307 (2009). TLC of diosgenin in methanolic extracts of fresh leaves and bulbs on silica gel with *n*-hexane - acetone 4:1. Detection by spraying with 25 % sulfuric acid in methanol and heating at 100 °C for 2 min. Quantitative determination by absorbance measurement at 540 nm.

herbal, quality control, traditional medicine, densitometry, quantitative analysis 32e

- 104 213 P. SONAWANE*, M. DHOKA, V. GAWANDE, P. VAIDYA (*All India Shri Shivaji Memorial Society's College of Pharmacy, Pune, Maharashtra, India): Simultaneous estimation of cefixime trihydrate and erdosteine in pharmaceutical dosage form by HPTLC method. Abstract No. F-256, 61st IPC (2009). HPTLC of cefixime trihydrate and erdosteine in combined capsule dosage form on silica gel with ethyl acetate - acetone - methanol - water 15:5:5:3. Quantitative determination by absorbance measurement at 235 nm. The calibration curve was linear between 100 and 500 ng/band for cefixime and 150 to 750 ng/band for erdosteine. The limit of detection and quantification for cefixime was 0.37 µg/mL and 1.14 µg/mL, respectively and for erdosteine 0.33 µg/mL and 1.04 µg/mL, respectively.

quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 214 N. SONI*, S. MANIMARAN, N. MURUGANANTHAM, S. DHANABAL, K. ELANGO (*Dept. of Phytopharmacy & Phytomedicine, JSS College of Pharmacy, Ootacamund, The Nilgiri, Tamil Nadu, India): Validated HPTLC method for the analysis of colchicine. Abstract No. 9933, IHCB (2009). HPTLC of colchicines in *Gloriosa superba* (collected from different parts of India) on silica gel with ethyl acetate - methanol 200:27. The hR_F value of colchicine was 29. Quantitative determination by absorbance measurement at 350 nm. The method was linear in the range of 50-1000 ng/spot. The sample collected from Kerala was found to contain highest level of colchicines (0.24 %).

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 215 B. SPARZAK, Mirosława KRAUZE-BARANOWSKA*, L. POBLOCKA-OLECH (*Medical University of Gdansk, Department of Pharmacognosy with the Medicinal Plants Garden, Hallera 107, 80-416 Gdansk, Poland; krauze@amg.gda.pl): High-performance thin-layer chromatography densitometric determination of beta-sitosterol in *Phyllanthus* species. *J. AOAC Int.* 92, 1343-1348 (2009). HPTLC of beta-sitosterol, beta-amyrin and plant extracts on silica gel with chloroform - *n*-hexane - methanol 13:6:1. Detection by spraying with vanillin-orthophosphoric acid reagent, 5 % phosphomolybdic acid, or anisaldehyde reagent, followed by heating at 110 °C for 5 min. Vanillin reagent provided the best results. Quantitative determination by absorbance measurement at 525 nm.

herbal, quality control, densitometry, quantitative analysis, HPTLC 32e

- 104 216 Malgorzata STAREK*, M. REJDYCH (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Kraków, Poland; mstarek@interia.pl): Densitometric analysis of celecoxib, etoricoxib and valdecoxib in pharmaceutical preparations. *J. Planar Chromatogr.* 22, 399-403 (2009). TLC of celecoxib, etoricoxib, and valdecoxib on silica gel with chloroform - acetone - toluene 12:5:2 with chamber saturation for 15 min at room temperature. Quantitative determination by absorbance measurement at 254 and 290 nm.

pharmaceutical research, quality control, densitometry, quantitative analysis 32a

- 104 217 G.S. SUBRAMANIAN*, A. KARTHIK, A. BALIGA, P. MUSMADE, S. KINI (*Department of Pharmaceutical Quality Assurance, Manipal College of Pharmaceutical Sciences, Manipal University, Manipal, Karnataka, India 576104; ganrajesh@gmail.com): High-performance thin-layer chromatographic analysis of bicalutamide in bulk drug and liposomes. *J. Planar Chromatogr.* 22, 273-276 (2009). HPTLC of bicalutamide and leflunomide (as internal standard) on silica gel with toluene - ethyl acetate 4:5 in a twin trough chamber saturated for 30 min at room temperature. Quantitative determination by absorbance measurement at 273 nm. The limit of detection and quantification was 50 and 200 ng/band, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 218 P. SUBRAMANIAN*, A. RAJENDRAN, V. MADHURAM, R. PADMA (*Drug Standardisation Unit O.U.B. 32, Raod, No.4, Habsiguda, Hyderabad 500007, India, veasha@rediffmail.com): HPTLC fingerprinting of some ethnomedicinally important Cassia species. *Indian Drugs* 46(6), 477-482 (2009). HPTLC of extracts of Cassia auriculata, C. obtusifolia, and C. uniflora and of chrysofanol and emodin on silica gel with toluene - ethyl acetate - formic acid 10:3:1. Detection under UV 254 nm. Based on the fingerprint and by comparison with chemical markers identification of each species was possible.

herbal, densitometry, HPTLC, qualitative identification 32e

- 104 219 A. SUGANTHI*, A. FATHIMUNNISA, T. RAVI (*College of Pharmacy, SRIPMS, Coimbatore, Tamil Nadu, India): HPTLC method for the simultaneous estimation of itopride hydrochloride and pantoprazole in pharmaceutical dosage form. Abstract No. F-264, 61st IPC (2009). HPTLC of pantoprazole and itopride hydrochloride on silica gel with *n*-butanol - chloroform - 25 % ammonia 7:2:1. The hR_F value was 54 and 75 for pantoprazole and itopride hydrochloride, respectively. Quantitative determination by absorbance measurement at 291 nm. The linearity of the method was 80-240 ng/band for pantoprazole and 300-900 ng/band for itopride.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 220 A. SUGANTHI*, M. SRIKANTH, A. GOP, T. RAVI (*College of Pharmacy, Sri Ramakrishna Institute of Paramedical Sciences, Coimbatore, Tamilnadu, India): A validated HPTLC method for the estimation of fenoverine in capsule dosage form. Abstract No. F-270, 61st IPC (2009). HPTLC of fenoverine on silica gel with methanol - butyl acetate 1:4. The hR_F value of fenoverine was 67. The linearity was in the range of 50 to 500 ng/band with a correlation coefficient of 0.9976. The limit of detection and quantification was 30 ng/band and 100 ng/band, respectively. The recovery was 100.1 %.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 221 B. SUHAGIA*, T. RATHOD, S. SINDHU (*L. M. College of Pharmacy, Ahmedabad, Gujarat, India): Quantitative determination of Sapindus saponins in the pericarp of Sapindus mukorossi. Abstract No. F-287, 61st IPC (2009). HPTLC of saponins (a mixture of the seven different sapindosides A, B, C, D, E, F, and G) in the pericarp of Sapindus mukorossi on silica gel with chloroform - methanol - water 50:14:1. Detection by derivatization with sulfuric acid reagent and

evaluation under visible light. The hR_F values of sapindosides A to G are 19, 26, 41, 48, 59, 67, and 75. Quantitative determination by absorbance measurement at 550 nm after derivatization. The linearity ranges for sapinosides A to G were 0.9-4.3, 0.7-5.4, 3.3-15.2, 1.2-9.7, 2.7-12.5, 0.2-1.3, 0.3-1.5 µg/band. The pericarp contained 15.2 % w/w of total sapindosides.

herbal, HPTLC, densitometry, postchromatographic derivatization, quantitative analysis 32e

- 104 222 Divya SUKUMAR*, R. ARIMBOOR, C. ARUMUGHAN (*Agroprocessing & Natural Products Div., National Institute for interdisciplinary Science & Technology, Thiruvananthapuram, Kerala 695010, India, carumughan@yahoo.com): HPTLC fingerprinting and quantification of lignans as markers in sesame oil and its polyherbal formulations. J. Pharm. Biomed. Anal. 47, 795-801 (2008). HPTLC of sesamin and sesamolone (the major lignans in sesamum oil and its herbal formulations) on silica gel with benzene - methanol 50:1 with chamber saturation. Quantitative determination by absorbance measurement at 290 nm. The identity of sesamin and sesamolone was confirmed by UV-VIS spectra, NMR and MS of the compounds obtained by scraping off from the plate and elution. For fingerprint analysis derivatization with 5 % methanolic sulphuric acid was performed, followed by heating at 100 °C for 20 min and densitometry at 450 nm.

pharmaceutical research, herbal, HPTLC, densitometry, quantitative analysis, comparison of methods, postchromatographic derivatization 32e

- 104 223 Katarzyna SZEWCZYK*, L. KOMSTA, A. SKALSKA.KAMINSKA (*Department of Pharmaceutical Botany, Faculty of Pharmacy, Medical University of Lublin, Chodzki 1, 20-093 Lublin, Poland; k.szewczyk@am.lublin.pl): Densitometric HPTLC method for analysis of triterpenoids in the leaves of *Jovibarba sobolifera* (Sims.) Opiz (Hen nad chickens houseleek). J. Planar Chromatogr. 22, 367-369 (2009). HPTLC of triterpenoids (alpha- and beta-amyrin, oleanolic acid) on silica gel prewashed with methanol and dichloromethane, with dichloromethane - ethyl acetate 37:3. in a horizontal chamber saturated for 15 min. Detection by spraying with 8 % sulfuric acid in ethanol and heating at 105 °C for 3 min. Evaluation in daylight and under UV 366 nm. Quantitative determination by absorbance measurement at 520 nm.

herbal, quality control, HPTLC, densitometry, quantitative analysis 32e

- 104 224 E. TOTH*, G. JANICSAK, I. MATHE, G. BLUNDEN (*Institute of Ecology and Botany of the Hungarian Academy of Sciences, Alkotmay u. 2, 2163 Vacratot, Hungary; totheniko@botanika.hu): Determination of phenylpropanoids in three *Ballota* species. J. Planar Chromatogr. 22, 293-296 (2009). TLC of verbascoside, forsythoside B, caffeoyl-malic acid and plant (*Ballota nigra*, *B. hirsuta*, and *B. rupestris*) extracts on silica gel with formic acid - acetic acid - water - ethyl acetate 15:15:36:134. Quantitative determination by fluorescence measurement at 395 nm. It was observed that amounts of phenylpropanoids in *Ballota nigra* leaves increase during the main and secondary flowering periods in June.

herbal, quality control, densitometry, quantitative analysis 32e

- 104 225 J. VADHAVANA*, B. PATEL, R. PATEL (*K. B. Institute of Pharmaceutical Education and Research, Gandhinagar, Gujarat, India): Simultaneous estimation of nebivolol hydrochloride and S-amlodipine besylate by high-performance thin-layer chromatography. Abstract No. F-247, 62st IPC (2009). HPTLC of nebivolol HCl and S-amlodipine besylate on silica gel (prewashed with methanol) with chloroform - toluene - methanol - acetic acid 50:20:1. The hR_F value was 33 and 48 for S-amlodipine and nebivolol, respectively. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 500-2500 ng/band for nebivolol and 250-1280 ng/band for S-amlodipine, respectively.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 226 D. VASA*, N. VASA, P. GIDE, V. VAGHELA (*A. R. College of Pharmacy & G. H. Patel Institute of Pharmacy, Anand, Gujarat, India): HPTLC method development for estimation of rivastigmine hydrogen tartrate in pharmaceutical dosage form. Abstract No. F-365, 61st IPC (2009).

HPTLC of rivastigmine hydrogen tartrate on silica gel with methanol - 25 % ammonia - acetic acid 200:7:2. The hR_F value was 54. Quantitative determination by absorbance measurement at 215 nm. The method was linear in the range of 1-10 $\mu\text{g}/\text{band}$.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 227 S. VASANTHARAJU*, A. KARTHIK, K. BHAT, C. PRASHANT, M. RAO, N. UDUPA (*Manipal College of Pharmaceutical Science, Manipal, Karnataka, India): HPTLC method development and validation of capsaicin in bulk drug. Abstract No. 9452, IHC (2009). HPTLC of capsaicin on silica gel with toluene - ethyl acetate 3:2. The hR_F value of capsaicin was 38. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 100-1000 ng/spot. Capsaicin was subjected to different stress conditions (alkali, acid, oxidation, thermal). The method is suitable for separation of capsaicin from its degradation products and can be used to indicate stability.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 228 R. VERMA*, H. MUKHTAR, R. SINGH, A. PASRIJA (*S.B.S College of Pharmacy, Patna, Punjab, India): Validated HPTLC method for the determination of 3H-4M-benzaldehyde in crude plant material, extracts and dosage form of Hemidesmus indicus. 60th Indian Pharmaceutical Congress PG-349 (2008). HPTLC of 3H-4M-benzaldehyde (in crude plant material, extracts and dosage form of Hemidesmus indicus) on silica gel with toluene - ethyl acetate - methanol - acetic acid 15:3:1:1. Quantitative determination by absorbance measurement at 230 nm.

herbal, HPTLC, densitometry, quantitative analysis 32e

- 104 229 S. WAKODE*, H. SINGH, V. SINGH (*Delhi Institute of Pharmaceutical Science & Research, New Delhi, India): Development and validation of HPTLC assay method for voriconazole in tablets. 60th Indian Pharmaceutical Congress PA-229 (2008). HPTLC of voriconazole on silica gel with toluene - methanol - glacial acetic acid 78:20:1. Quantitative determination by absorbance measurement at 254 nm. The method was linear in the range of 50-400 ng/spot, recovery was 99.9-100.7 %. The method was suitable for routine quality control of the dosage form.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 230 P. WAVHAL*, J. SANGSHETTI, A. SARKATE, P. WAKTE, D. SHINDE (*Dept. of Chemical Technology, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, Maharashtra, India): Stability-indicating HPTLC determination of tadalafil in API and in its pharmaceutical dosage form. Abstract No. F-257, 61st IPC (2009). HPTLC of tadalafil on silica gel with *n*-hexane - ethyl acetate - acetonitrile 14:3:3. The hR_F value was 65. Quantitative determination by absorbance measurement at 215 nm. The method was linear in the range of 10-60 ng/band. The drug was subjected to different stress conditions (acid, alkali, oxidative, photodegradation, thermal) and showed degradation under all stress conditions. Degradation products and excipients of the formulation were well separated from the main component.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis 32a

- 104 231 T. XONG (Xong Ting) (Pharm. Coll., Wuhan Univ., Wuhan, Hubei 430072, China): (Determination of astragaloside in Tianjian capsule) (Chinese). Chinese J. Hospit. Pharm. 29 (4), 336-338 (2009). TLC of astragaloside in Tianjian capsules on silica gel with chloroform - methanol - water 13:6:2. Detection by spraying with 5 % sulfuric acid in ethanol followed by heating at 105 °C until coloration. Quantification by densitometry at 515 nm. The linearity was between 0.98 and 4.90 $\mu\text{g}/\text{spot}$ ($r^2 = 0.998$), the %RSD was 2.4 % ($n = 6$) within plate and 2.3 % ($n = 6$) inter-plate, standard addition recovery was 99.2 % with RSD = 1.7 % ($n = 6$).

pharmaceutical research, traditional medicine, quality control, herbal, quantitative analysis, qualitative identification, densitometry 32e

- 104 232 L. XU (Xu Li)*, Y. ZHAO (Zhao Yuan), Y. LUAN (Luan Yuquan), Y. YANG (Yang Yongshou), CH. WANG (Wang Chengjun) (*Basic Med. Coll., Dali Acad., Dali, Yunnan 671000, China): (Study of the content of puerarin in different parts of Radix Puerariae) (Chinese). Learned J. Dali Acad. (General Issue) 8 (10), 3-6 (2009). TLC of puerarin (in extracts obtained from different parts of Radix Puerariae) on silica gel with trichloromethane - methanol - water 14:5:1. Detection under UV 254 nm. The maximum amount of puerarin was found in the bine of the drug, followed by that in the root, whereas no puerarin was found in the flower and fruit.
- pharmaceutical research, traditional medicine, herbal, quality control, quantitative analysis, qualitative identification 32e
- 104 233 A. YADAV, R.M. SINGH*, S.C. MAHTUR, P.K. SAINI, G.N. SINGH (*Indian Pharmacopoeia Commission, Govt. of India, Ministry of Health & Family Welfare, Sect-23, Rajnagar, Ghaziabad (U.P), India 201 002; raman19662002@yahoo.co.in; ipclab@vsnl.net): A simple and sensitive HPTLC method for simultaneous analysis of domperidone and paracetamol in tablet dosage form. J. Planar Chromatogr. 22, 421-424 (2009). TLC of domperidone and paracetamol on silica gel with acetone - toluene - methanol 2:2:1 in a twin trough chamber saturated for 30 min at room temperature. Quantitative determination by absorbance measurement at 285 nm for domperidone and at 248 nm for paracetamol.
- pharmaceutical research, quality control, quantitative analysis, densitometry 32a
- 104 234 A. YADAV*, N. TIWARI, P. SRIVASTAVA, S. SINGH, K. SHANKER, R. VERMA, M. GUPTA (*Analytical Chemistry Div. Central Institute of Medicinal and Aromatic Plants, Lucknow 226015, India, guptammg@rediffmail.com): Iridoid glycoside-based quantitative chromatographic fingerprint analysis: A rational approach for quality assessment of indian medicinal plant Gambhari (Gmelina arborea). J. Pharm. Biomed. Anal. 47, 841-846 (2008). HPTLC of iridoid glycosides in the aerial part of Gambhari (Gmelina arborea) with iridoid glycoside 6-O-(2'', 3''-dibenzoyl)-o-L-rhamnopyranosylcatalpol as a chemical marker for the standardization of G. arborea plant extracts on silica gel with chloroform - methanol 4:1. Quantitative determination by absorbance measurement at 240 nm and at 430 nm after derivatization with vanillin - sulfuric acid reagent. The linear working range was between 1000-5000 ng/spot with a good correlation coefficient of 0.994.
- pharmaceutical research, quality control, HPTLC, densitometry, comparison of methods, quantitative analysis, postchromatographic derivatization 32e
- 104 235 H. YAN (Yan Hua)*, Y. ZHANG (Zhang Yumei), J. SONG (Song Jincui), J. LU (Lu Jing) (*Nat. Inst. Cont. Pharm. & Biolog. Prod., Beijing 100050, China): (Analysis of adenosine and adenine in Lingzhi capsules by TLC and HPLC) (Chinese). Chinese J. Pharm. Anal. 28 (11), 1800-1803 (2008). TLC of adenosine and adenine in Lingzhi capsules on silica gel with chloroform - ethyl acetate - i-propanol - water 16:6:3:1. Detection under UV 254 nm. The method is simple, fast and accurate and can be used for the quality control of the medicine.
- pharmaceutical research, traditional medicine, quality control, herbal, quantitative analysis, qualitative identification 32c
- 104 236 L. YANG (Yang Li)*, SH. ZHANG (Zhang Shengwan), W. DU (Du Wen), W. WANG (Wang Wei), M. LI (Li Meiping) (*Coll. Life Sci. & Technol., Shanxi Univ., Taiyuan 030036, China): (Determination of hyperoside in the raw extract of Hypericum perforatum by thin-layer chromatography) (Chinese). Chinese J. Pharm. Anal. 28 (4), 608-610 (2008). TLC of hyperoside in the raw extract of Hypericum perforatum on silica gel with petroleum ether (60-90 °C) - ethyl acetate - methanol 1:4:2. Detection under daylight. Quantification by densitometry at 591 nm. Linearity was given between 0.1 and 14.4 µg/spot ($r^2 = 0.9892$), recovery was between 96.4 and 100.1 %.
- pharmaceutical research, quality control, traditional medicine, herbal, qualitative identification, quantitative analysis, densitometry 32c

- 104 237 H.E. ZAAZAA*, S.S. ABBAS, M. ABDELKAWY, M.M. ABDELRAHMAN (*Analytical Chemistry Department, Faculty of Pharmacy, Cairo University, Kasr El-Aini St., 11562 Cairo, Egypt): Spectrophotometric and spectrodensitometric determination of clopidogrel bisulfate with kinetic study of its alkaline degradation. *Talanta* 78 (3), 874-884 (2009). Presentation of a sensitive, selective and precise stability-indicating method for the determination of clopidogrel bisulfate in presence of its alkaline degradate and in pharmaceutical formulations. TLC on silica gel with hexane - methanol - ethyl acetate 87:10:3. Quantification by densitometry at 248 nm in the range of 0.6-3 µg/band. Recovery was 99.9 %. Clopidogrel could be determined in the presence of up to 90 % of its alkaline degradate. Method selectivity was evaluated using laboratory prepared mixtures. The analysis of clopidogrel in pharmaceutical dosage forms is possible without interference from additives.

pharmaceutical research, quality control, quantitative analysis, qualitative identification, comparison of methods

32c

- 104 238 CH. ZHENG (Zheng Cheng)*, T. YAO (Yao Tongwei), ZH. BAI (Bai Zhimin) *(Pharm. Coll., Zhejiang Univ., Hangzhou 310080, China): (Study of the quality standard for Jinlu pills) (Chinese). *J. Chinese Trad. & Herb. Drugs* 40 (6), 900-903 (2009). TLC of the TCM drug extracts on silica gel with 1) ethyl acetate - formic acid - glacial acetic acid - water 15:1:1:2; 2) petroleum ether (30-60 °C) - ethyl acetate - formic acid 15:5:1; 3) chloroform - ethyl acetate - methanol - water 31:81:25:2. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration; 2) under UV 254 nm.

pharmaceutical research, traditional medicine, quality control, qualitative identification, quantitative analysis

32e

- 104 239 Y. ZHOU (Zhou Ying)*, X. NIU (Niu Xiuhua) (*Nat.Inst. Cont. Pharm. & Biolog. Prod., Beijing 100050, China): (Determination of related impurity substances in nisoldipine by thin-layer chromatography) (Chinese). *Drug Standards of China* 9 (2), 144-146 (2008). TLC of nisoldipine silica gel with chloroform - acetone - triethylamine - water 90:5:1. Detection under UV 254 nm. Semiquantification of impurities by comparison of spots. The method was successfully used for the quality control of real life samples.

pharmaceutical research, quality control, quantitative analysis, qualitative identification

32c

33. Inorganic substances

- 104 240 A. RADOICIC, H. MAJSTOROVIC, T. SABO, Z. TESIC, Dusanka MILOJKOVIC-OPSENI-CA* (*Faculty of Chemistry, University of Belgrade, P. O. Box 51, 11158 Belgrade, Serbia; dusankam@chem.bg.ac.yu): Hydrophilic-interaction planar chromatography of some water-soluble Co(III) complexes on different adsorbents. *J. Planar Chromatogr.* 22, 249-253 (2009). Investigation of the chromatographic behavior of twelve neutral, mixed cobalt(III) complexes of the uns-cis-edda-type in six planar chromatographic systems. Four different stationary phases - silica gel, cyano phase, cellulose, and alumina - were combined with water-organic solvent (methanol or acetone) binary mobile phases. Hydrophilic-interaction chromatography was assumed to be the mechanism determining separation under normal-phase conditions (use of mobile phases with small amounts of water), whereas the use of mobile phases with high water content lead to reversed-phase chromatography.

qualitative identification

33a

35. Other technical products and complex mixtures

- 104 241 Dorina CASONI*, C. SARBU (*Department of Analytical Chemistry, Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, Arany Janos Str. No 11, 400028 Cluj-Napoca, Romania): Lipophilicity of some preservatives estimated by RP-TLC using stationary phases with different polarity. *Chromatographia* 70 (7-8), 1277-1282 (2009). HPTLC of preservatives on three stationary phases of different polarity: RP-18, RP-18W and cyano phase, with methanol - water mixtures in different volume proportions. The resulting R_M values showed a linear decrease with increasing methanol concentration of the mobile phase (determination coefficients for all stationary phases were >0.98). The retention behavior of the preservatives on RP phase is in

good agreement with their polarity. Principal component analysis showed that for all three stationary phases the same lipophilic interactions take place.

HPTLC, autoradiography

35b

38. Chiral separation

- 104 242 R. BHUSHAN*, H. BRÜCKNER, V. KUMAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee 247 667, India): Indirect resolution of enantiomers of penicillamine by TLC and HPLC using Marfey's reagent and its variants. *Bio. Chromatogr.* 21(10), 1064-1068 (2008). Indirect chiral TLC separation of penicillamine (3,3-dimethylcysteine) enantiomers after derivatization with Marfey's reagent (FDNP-Ala-NH₂) and two of its structural variants, FDNP-Phe-NH₂ and FDNP-Val-NH₂ on silica gel and RP-18 with phenol - water 3:1 and solvent combinations of acetonitrile and triethylamine phosphate buffer. The methods were applied for determination of the enantiomeric impurity of l-penicillamine, d-penicillamine, and pharmaceutical formulations of d-penicillamine.

pharmaceutical research, quality control, quantitative analysis,
qualitative identification, comparison of methods

38, 28a

- 104 243 R. BHUSHAN*, S. TANWAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee - 247667, India): Different approaches of impregnation for resolution of enantiomers of atenolol, propranolol and salbutamol using Cu(II)-l-amino acid complexes for ligand exchange on commercial thin-layer chromatographic plates. *J. Chromatogr. A* 1217(8), 1396-1398 (2010). Separation of enantiomers of atenolol, propranolol, and salbutamol using different loading/impregnation techniques for the Cu(II) complexes of l-proline, l-phenylalanine, l-histidine, N,N-dimethyl-l-phenylalanine, and l-tryptophan. TLC on silica gel with acetonitrile - methanol - 2 mM aqueous solution of Cu(II) 3:4:5. The different techniques were: A) using the Cu(II)-l-amino acid complex as chiral mobile phase additive, B) development of plates in solutions of Cu-complex, and C) with a solution of Cu(II)acetate as mobile phase additive for plates impregnated with the amino acids. Detection of zones by exposure to iodine vapor.

pharmaceutical research, quality control, quantitative analysis,
qualitative identification, comparison of methods

38

- 104 244 R. BHUSHAN*, S. TANWAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee, 247667, India): Direct TLC resolution of the enantiomers of three beta-blockers by ligand exchange with Cu(II)-L-amino acid complex, using four different approaches. *Chromatographia* 70 (5-6), 1001-1006 (2009). Chiral TLC of the enantiomers of atenolol, propranolol, and salbutamol by complexation with Cu(II) cation and five L-amino acids using different techniques: 1) using the Cu(II)-L-amino acid complex as chiral mobile phase additive with untreated silica gel plates, 2) by mixing the Cu-complex with silica gel before preparing the TLC plates, 3) by development with solutions of the Cu-complex on untreated silica gel plates, and 4) by using a solution of Cu(II) acetate as mobile phase additive for plates prepared by mixing the L-amino acid with silica gel. Detection of zones by exposure to iodine vapor.

pharmaceutical research, qualitative identification

38

International Symposium for High-Performance Thin-Layer Chromatography BASEL, 06th–08th July 2011



We are enthusiastic to learn how many analysts are now confronted with situations where HPTLC is a suitable solution to their problems and is favored over better known and more widely used analytical methods. At the same time it is rather difficult nowadays to find a place for this “old technique” in the minds of opinion leaders, even if the need exists in analytical laboratories. This has arisen through inadequate information and training.

How to select the method; how to use HPTLC when it is described as a method of choice; avoidance of usual mistakes; which other samples may be well covered by this technique;... To address these issues an exchange of knowledge is foremost, from which sprang our motivation to hold again an international event with the Interlaken series spirit, last held in 1997. After Lyon 2003, Berlin 2006, and Helsinki 2008, we are pleased to announce that the 4th International Symposium for High-Performance Thin-Layer Chromatography will be organized on 06th–08th July 2011, in the Congress Centre of Basel, Switzerland. This new major issue will include a workshop, a symposium, and a manufacturers session.

The scientific program will feature invited keynote speakers, selected submitted lectures and poster presentations. Contributions are invited from all areas of high-performance thin-layer chromatography, but especially from colleagues working in the pharmaceutical, food, environmental and medical fields. Papers on theory, method development, validation, instrumental methods, hyphenated techniques, and quantitative applications in all areas of chemistry would be most welcome.

Deadlines for

Abstract submission (oral and poster): **March 1st 2011**

Final registration:

May 30th 2011

The **participation fee** includes the full scientific program, and added to that, lunches, coffee breaks, the symposium dinner, and the social events.

Industrial **500 €**

Academic **400 €**

Students **200 €**

Reduction of **100 €** with ISPS or CCCM membership.

Special accommodation rates are available for hotels close to the symposium venue.

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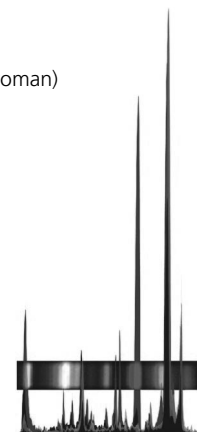
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Dr. Irena Vovk, Slovenia

Dr. Vicente Cebolla, Spain

Dr. Eike Reich, Switzerland

Prof. Dr. Joseph Sherma, USA



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2 min (silica gel plate) or 30 s (RP18 plate).

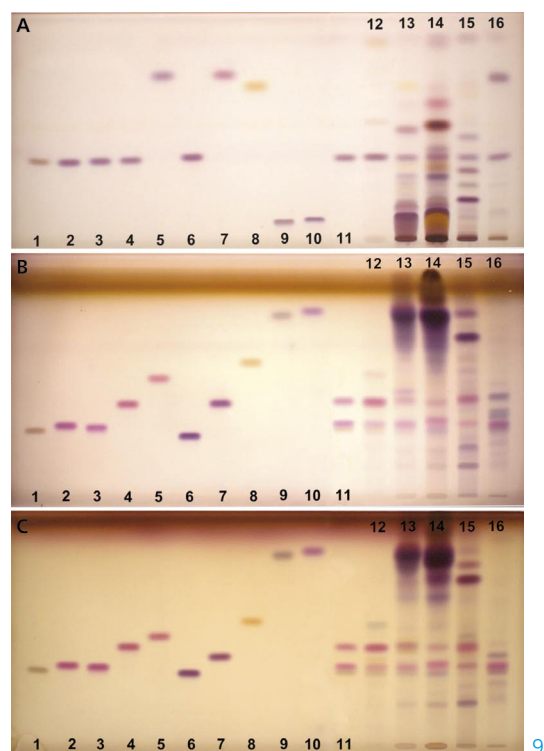
Note (editor): In case of quantification the plate has to be placed on the cold TLC Plate Heater, being heated to 110 °C. By doing so, homogeneous heating across the whole plate is assured, and thus a good precision guaranteed.

Documentation

With DigiStore 2 under UV 366 nm and under white light illumination

Results and discussion

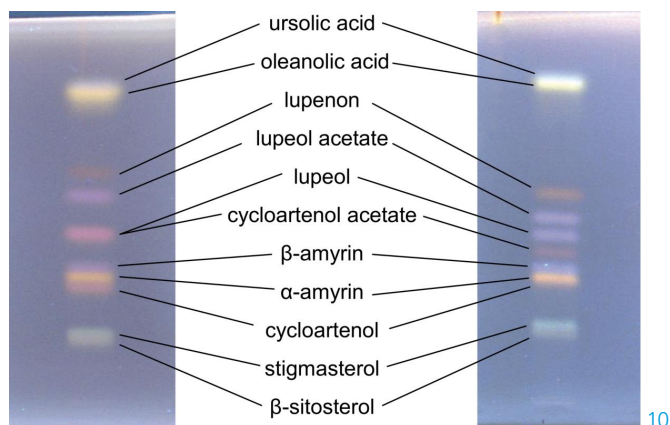
The separation of triterpenoids with different functional groups (alcohols, acids, ketones and esters) has been generally achieved on silica gel plates, although the use of RP18 plates was crucial for the separation of four isomeric triterpenols and two triterpenol esters.



Separation of triterpenoids on silica gel (A) and RP18 phases (B: ethyl acetate – acetonitril 3:2, C: acetone – acetonitrile 5:1). Tracks: 1 α -amyrin; 2 β - amyrin; 3 δ - amyrin; 4 lupeol; 5 lupeol acetate; 6 cycloartenol; 7 cycloartenol acetate; 8 lupenon; 9 ursolic acid; 10 oleanolic acid; 11 α -amyrin, β -amyrin and lupeol; 12 cabbage; 13 rosemary; 14 sage; 15 oak bark; 16 tomato

The separation of isomeric triterpenols (α -amyrin, β -amyrin, lupeol and cycloartenol) was achieved using the solvent system ethyl acetate - acetonitril 3:2, but with this system the separation of isomeric esters failed. On the other hand, solvent system acetone –

acetonitrile 5:1 separated the mentioned isomeric esters, but cycloartenol was not fully separated from α -amyrin.



Characteristic fluorescence of bands under UV 366 nm after derivatization with anisaldehyde sulfuric acid reagent; separation on RP18 plates with ethyl acetate – acetonitrile 3:2 (left) and acetone – acetonitrile 5:1 (right)

Moreover, both reversed phase methods enabled to some extent the separation of triterpenoids with different functional groups, yet with no interference from the structurally related sterols.

For identification of compounds, the characteristic colors and fluorescence of bands obtained after derivatization with anisaldehyde sulfuric acid reagent were helpful. Through this selective, post-chromatographic derivatization for all samples in parallel, HPTLC provides easy, fast and inexpensive screening of triterpenoids.

Further information is available on request from the author(s).

[1] M. Martelanc, I. Vovk, B. Simonovska, J. Chromatogr. A 1164 (2007) 145 and [2] dito 1216 (2009) 6662

* Dr. Irena Vovk, National Institute of Chemistry, Laboratory for Food Chemistry, Hajdrihova 19, 1000 Ljubljana, Slovenia, irena.vovk@ki.si

Validated determination of secoisolariciresinol diglucoside in flaxseed by HPTLC



Prof. Silvia Coran

Getting the very best results from any analytical system requires a clear understanding of the capabilities of each instrument in the system, as well as any attachments, the sampling procedure and of course the software. This is particularly true with HPTLC where the components for each step must be coordinated in order to realize the technique's full problem solving capabilities. No where is this more apparent than at the University of Florence as promoted by Silvia A. Coran*, Associate Professor of Pharmaceutical Chemistry of Department of Pharmaceutical Science. Since HPTLC affords much flexibility for operative parameter optimization, novel analytical HPTLC methods have been set up as powerful alternative to HPLC. Even validation based on the more demanding HPLC protocols has been successfully carried out here at the University.

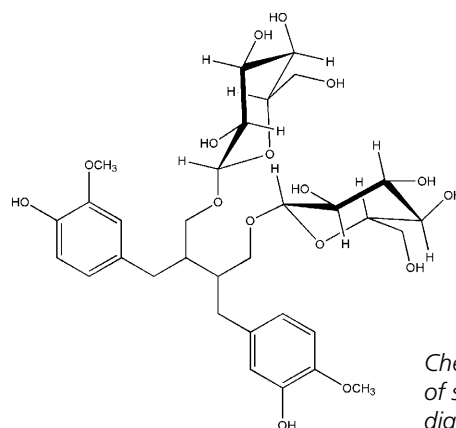
Introduction

Secoisolariciresinol diglucoside (SDG) is a plant lignan most notably found in flaxseed. The considerable interest in SDG is driven by the observation that it is the main precursor for mammalian lignans that potentially protect against hormone related cancers. Since SDG is plant-derived and shows a weak estrogenic activity, it is classified as phytoestrogen.

Following a previously proposed novel method for the quantitative determination of SDG in flaxseed using silica gel plates [1], a significant improvement

of the procedure was set up by shifting from silica gel phases to C18 phases to assure the essential run to run reproducibility without any need for control of the plate activity [2].

With this change, a selective and robust method was obtained and fully validated by applying the SFSTP (Société Française des Sciences et Techniques Pharmaceutiques) Commission protocol [3] tailored for HPLC determinations. This protocol encompasses the calculation of accuracy profiles, the total error measurement and the associated risk. The validated method is competitive with the more popular HPLC approach in terms of simplicity of execution and routine throughput. But above all, HPTLC allows to exclude the time-consuming flaxseed defatting from the sample preparation step.



Chemical structure of secoisolariciresinol diglucoside (SDG)

Sample preparation

Flaxseed was frozen in liquid N₂ and very finely ground. 100 mg of the resulting undefatted meal were suspended in 2 mL of aqueous NaOH 0.1 M and sonicated for 60 min at 40 °C. After cooling to room temperature the sample was neutralized with HCl, acidified to pH 3 with 50 µL HCOOH and diluted to 10 mL with MeOH. For the assay, 5 µL of this solution were applied.

Standard solution

The stock solution was prepared by dissolving

4.3 mg of SDG in 5 mL of MeOH (0.86 mg/mL). This solution was diluted with HCOOH 0.1 % to obtain the standard solution of 214 ng/ μ L.

Chromatogram layer

HPTLC plates RP18 W F_{254s}, 10 x 10 cm (Merck) pre-washed by immersion in MeOH over night, dried in N₂ stream under vacuum.

Note (editor): W stands for with water wettable layers, suited for application of aqueous samples.

Sample application

Bandwise with Linomat 5 on the two opposite plate sides resulting in 18 tracks, band length 7 mm, track distance 8.7 mm, distance from the edges 15 mm, application volume 1–5 μ L for standard and 5 μ L for sample solutions, delivery speed 60 nL/s.

Chromatography

In the Horizontal Developing Chamber 10 x 10 cm with 8 mL MeOH – HCOOH 0.1% 2:2 (4 mL per side), migration distance 50 mm (development time 12 min).

Densitometry

TLC Scanner 3 with winCATS software, absorption measurement at 282 nm, slit dimension 5 mm x 0.45 mm, scanning speed 20 mm/s, data resolution 50 μ m/step, polynomial regression by peak area.

Results and discussion

The validation was a two-step strategy based on the accuracy profiles. The acceptance limit was set at 15 % according to the FDA guidelines for bio-analysis. In the pre-validation step five calibration points ranged 214–1071 ng/band were obtained in quadruplicate and repeated on three different days. Four regression functions were calculated: linear model, weighted linear (1/x) model, quadratic model and weighted (1/x) quadratic model. The mean bias, the repeatability and the intermediate precision were back-calculated for each level using the four regression models. The accuracy profiles obtained using the confidence intervals indicated the quadratic model as the best choice giving a dosing range index of 1.00 and a smaller bias.

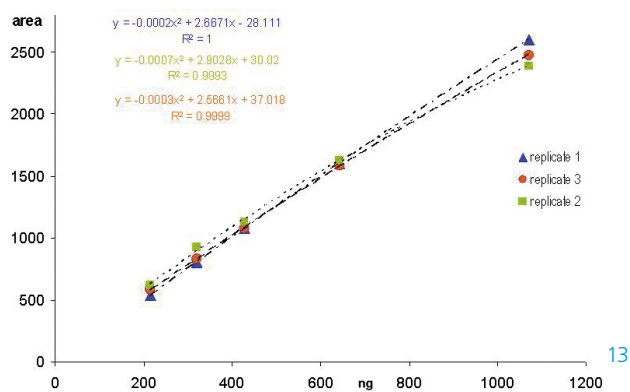


Horizontal Developing Chamber (HDC)

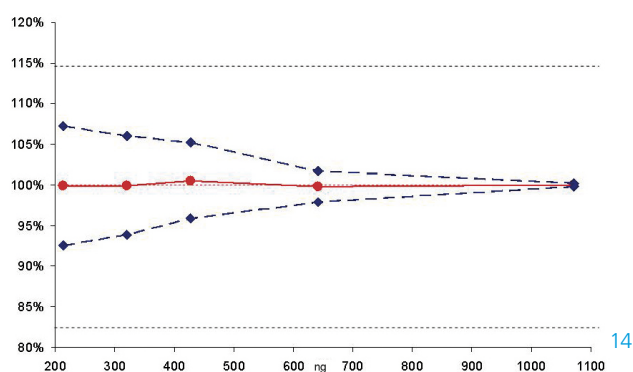
The Horizontal Developing Chamber (HDC) allows the development of a plate from both opposite sides toward the middle, thus doubling the number of samples per plate compared to conventional developing techniques. Here the HDC is used for determination of secoisolariciresinol. In this application, using the HDC 10 x 10 cm, 18 samples are simultaneously developed in 12 min. Use of the HDC 20 x 10 cm would develop 36 samples in 12 min! This would result in 40 and 20 seconds per sample, respectively.

For development, 4 mL solvent were used for chromatography, but already 2.5 mL solvent per side are sufficient. Thus, compared to other chamber types the HDC consumes less solvents, e.g. 75 % less compared to a flat bottom chamber. Also the disposal costs are far below 0.01 Cent per sample.

Easy handling is combined with a high degree of flexibility for conditioning in the tank configuration or for chromatography in the sandwich configuration. The HDC is unsurpassed from an economic point of view, in flexibility and reproducibility of the result in routine work. Test it!



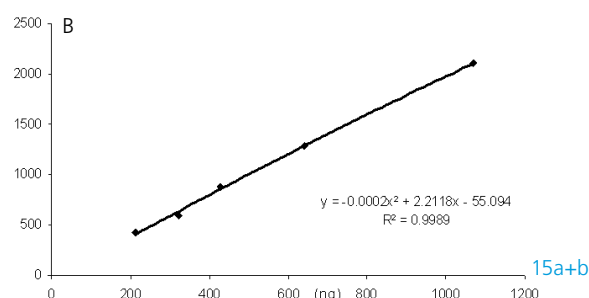
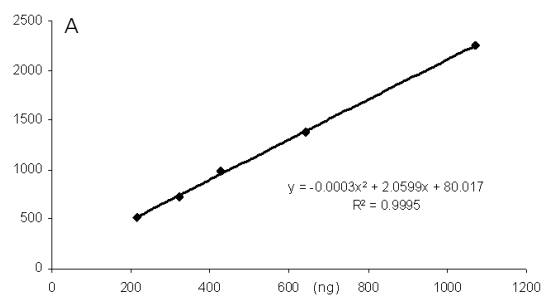
Polynomial calibration curves of the three replicates



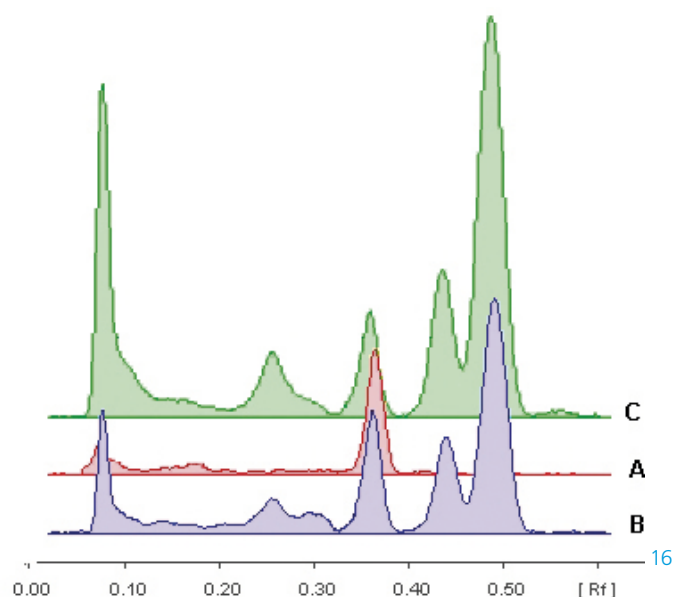
Accuracy profile of SDG obtained by the quadratic regression model: ◆ confidence interval, ● relative trueness

As blank matrix being not available, the standard addition method was used with the interpolation approach. The standard addition curve, calculated with the quadratic regression function, coincided with the calibration curve obtained for the pure analyte in absence of matrix, assessing the validity of the calibration mode. Both determination coefficients were better than 0.999. Using 10 x 10 cm plates, three-point calibration curves (321, 642 and 1071 ng/band) were admitted for routine analysis after successful validation.

The validation standards for accuracy purpose were obtained by spiking a pre-analyzed hydrolysed sample with different standard concentrations. Recoveries were between 100.4 and 103.2 %. The method fulfilled the requirements, having bias largely inside the acceptance limits (95 %) and the relative standard deviation of repeatability and intermediate precision between 2.0 and 3.6 %. To test the method, the SDG content in flaxseed of five different cultivars was determined to be between 0.9 and 1.3 % with residual standard deviations (%RSD) of ≤ 2.3 % ($n = 3$).



Polynomial calibration of SDG: (A) without matrix, (B) in matrix



HPTLC densitograms at 282 nm of SDG standard (A) and flaxseed samples (B and C)

Further information is available on request from the author.

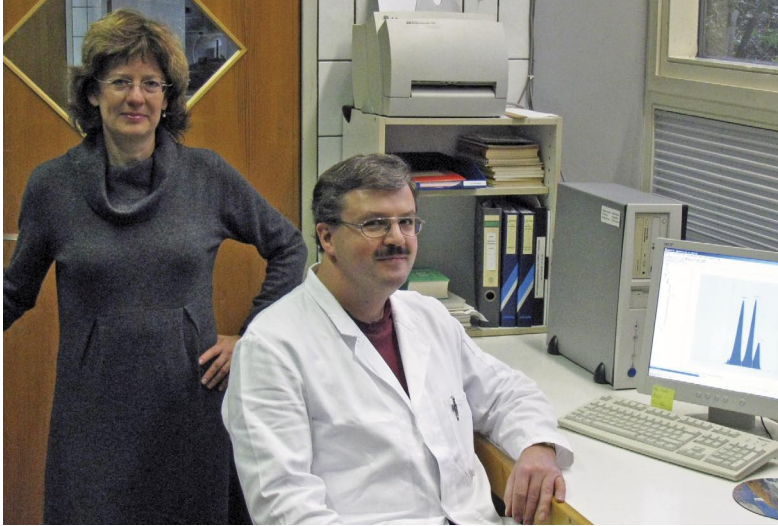
*Prof. Silvia A. Coran, University of Florence, Faculty of Pharmacy, Department of Pharmaceutical Science, Via Ugo Schiff 6, 50019 Sesto Fiorentino (Florence), Italy, coran@unifi.it

[1] S.A. Coran, V. Giannellini, M. Bambagiotti-Alberti, J Chromatogr A 1045 (2004) 217.

[2] S.A. Coran, G. Bartolucci, M. Bambagiotti-Alberti, J Chromatogr A 1207 (2008) 155.

[3] P. Hubert et al., J Pharm Biomed Anal 45 (2007) 82.

Determination of aloe vera gel in cosmetics



17

Evamaria Kratz, Jürgen Geisser

The CVUA Karlsruhe* is one of the 4 Official Food Control and Animal Health Laboratories for the Federal state of Baden-Württemberg in southern Germany. Its cosmetic team analyzes official samples that have been collected from retail outlets, regional manufacturers and importers in order to verify their compliance with cosmetics regulations.

Introduction

The gel from the inner part of the aloe vera leaves is traditionally used for skin care. In recent years, numerous cosmetic products have been launched where aloe vera extract was labelled as active ingredient. Aloe vera is an expensive raw material, a fact which may tempt product producers to blend the gel with water and gelling agent, resulting in the dose in the final product being very low, despite promotional activity to the contrary. Aloe vera gel is also offered as concentrated raw material (e.g. 10-times or 200-times).

In the following, an investigation of aloe vera product is described in which aloe vera has been promoted through advertisement in such a way that it would be expected to contain at least 5 %. With a 5% concentration the assumed efficacy is supposed, for example an increase of skin moisture or special care effects. Alooverose was chosen as a marker compound for aloe vera addition. This acetylated polymannose (molecular weight 50–500 kDa, approx. 1.1 acetyl groups per mannose

molecule) makes approx. 20 % of the approx. 0.5 % solids content of the fresh aloe vera gel and is therefore better suited for analytical monitoring than other ingredients like malic acid and glucose.

Up to now, only a ¹H-NMR method was established for the aloe vera raw material, where alooverose is quantitated by the signals of its acetyl groups. With a newly developed HPTLC method it is possible for the first time to test whether cosmetic finished products containing aloe vera, match the expectation of the consumer and/or the statements of efficacy, or if it is a deception. The quantitation of aloe vera addition is carried out via mannose after hydrolysis of alooverose. HPLC methods, which have been developed in the food industry for determination of sugars, turned out not to be suitable because of their insufficient limits of detection.

Standard solution

For stock solutions, 20 mg alooverose are dissolved in water and added to 20 mL; for glucose and galactose, 50 mg at 50 mL are used (can be stored deep-frozen for 1 year). Alooverose stock solution (0.8 mL) are hydrolyzed according to the sample solution and neutralized. For standard mixture solutions, each 1 mL glucose and 1 mL galactose stock solution are added to the hydrolyzed alooverose stock solution (0.8/100 mL and 1 mg/100 mL, respectively).

Sample preparation

Depending on the product, 1 to 5 g sample are homogenized with water. Lipophilic sample components are separated from the acidulated sample solution by solvent extraction with diethyl ether. Alooverose is degraded to mannose by hydrolysis with sulfuric acid (3.35 M, 3 h, 85 °C) and then neutralized. Some products contain hydrogen peroxide, which has to be destroyed by potassium iodide before hydrolysis.

Layer

HPTLC plates silica gel 60 (Merck), 20 × 10 cm, impregnated by immersion in 0.5 M NaH₂PO₄ solution (immersion time 1 s, vertical speed 4 cm/s), pre-drying at room temperature followed by drying on the TLC plate heater (5 min, 60 °C).

Sample application

Bandwise with Automatic TLC Sampler 4, 18 bands per plate, band length 6 mm, application volume 1–10 µL standard solution and, depending on the product, 1–15 µL sample solution, distance from the lower edge 10 mm, distance from the sides 12 mm, track distance 10 mm.

Application of the sample as rectangular area is recommended due to the high matrix load. This is enabled by the ATS4 (see p. 15).

Chromatography

In the flat bottom chamber 20 × 10 cm with 20 mL acetone – *i*-propanol – formic acid (0.1 M) 2:2:1, migration distance 80 mm (developing time approx. 75 min, double development for products with high glucose content is recommendable), drying of the plate on the TLC plate heater (5 min, 60 °C).

Note (editor): It is possible to achieve greater differences in adjacent zone distances using a development of >60 mm, however, this also leads to peak broadening.

Post-chromatographic derivatization

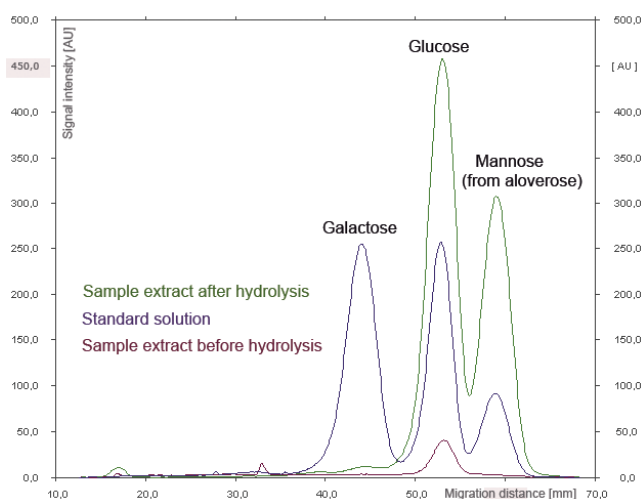
By immersion of the plate (immersion time 1 s, vertical speed 4 cm/s) in 4-aminobenzoic acid reagent (1 g 4-aminobenzoic acid are dissolved in 36 mL pure acetic acid, then 40 mL water, 2 mL of 85 % phosphoric acid and 120 mL acetone are added) and heated on the TLC plate heater (10 min, 110 °C). This reagent is stable for 2 weeks if cooled and stored under nitrogen.

Densitometry

TLC Scanner 3 with winCATS software, fluorescence measurement with Hg lamp at 366 nm, evaluation via peak area with winCATS Planar Chromatography Manager.

Results and discussion

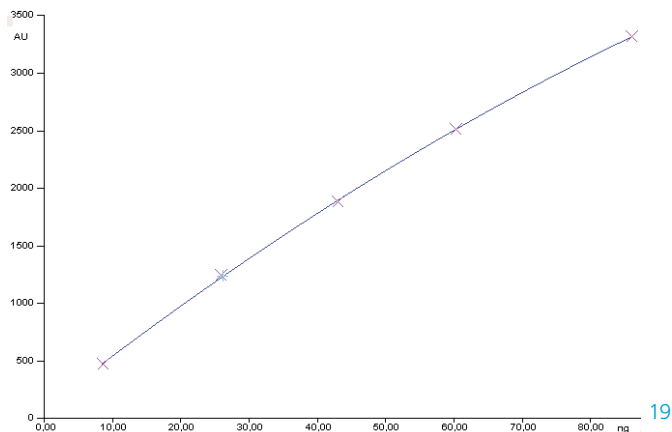
After hydrolysis of alooverose mannose is well separated by HPTLC from the other carbohydrates (except fructose) in the sample. Derivatization of the mannose with 4-aminobenzoic acid forms a fluorescent product, making detection more sensitive. The sample is compared before and after hydrolysis to find out, if mannose is a regular compound of the sample. If aloe vera is present, glucose as a natural compound of aloe vera gel can be detected before hydrolysis.



Comparison of the sample extract before and after hydrolysis

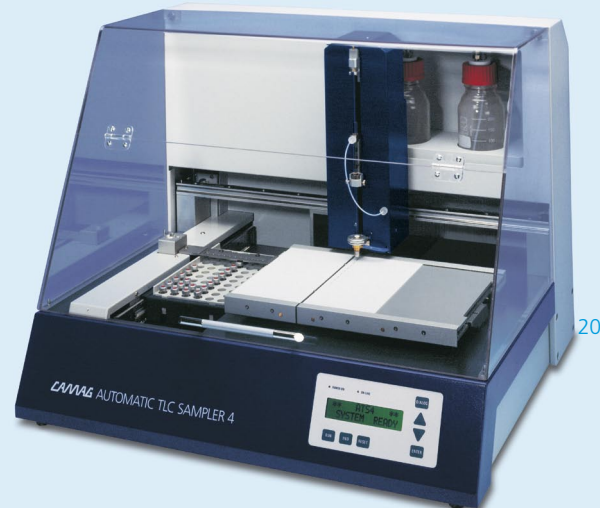
In some cosmetics' formulation maltodextrin might be present, which increases the glucose concentration after hydrolysis. Thickening agents also containing mannose are assigned after hydrolysis by comparing the characteristic building blocks like glucuronic acid in xanthan (organic acids are determined by a separate HPTLC method) and galactose in galactomannanes (guar gum or locust bean gum) and their amounts set in relation to mannose.

The limit of quantitation of aloe vera gel in products is approx. 3 % which is sufficient for this analytical task. The residual standard deviation of the polynomial calibration is 2.1 % for mannose ranged 8–80 ng/band and 4.0 % using matrix calibration.



Polynomial calibration (peak area) of mannose (from aloverose) ranged 8 – 80 ng/band 19

The new method enabled the cosmetic team at the CVUA Karlsruhe to verify the high aloe vera content in cosmetic finished products that had been promoted through advertisements. In the last two years several official complaints have led to improved diligence on the part of these producers to actually meet their label claims.



Area application with the Automatic TLC Sampler (ATS 4)

For determination of aloverose in cosmetic finished products, the high salt content present after the sample preparation and further constituents like tensides limit the sample volume for bandwise application. In such cases, area application of samples is advantageous: Despite the high matrix loading the sample volume can be increased and the limit of quantification be lowered. Depending on the solvent, a subsequent focusing of the analytes is required.

Syringe size:	25	μL	Band length:	8.0	mm
Application mode:	Spray area		Band width:	5.0	mm
Positioning					
Number of tracks:	18		Status:	OK	
First application position X:	15.0	mm			
Application position Y:	8.0	mm			
Distance between tracks:	10.0	mm		<input type="radio"/> Automatic <input checked="" type="radio"/> Manual	
Track	Application position	Application volume	Rack col	Rack row	
1	15.0 mm	2.0 μl	A	1	



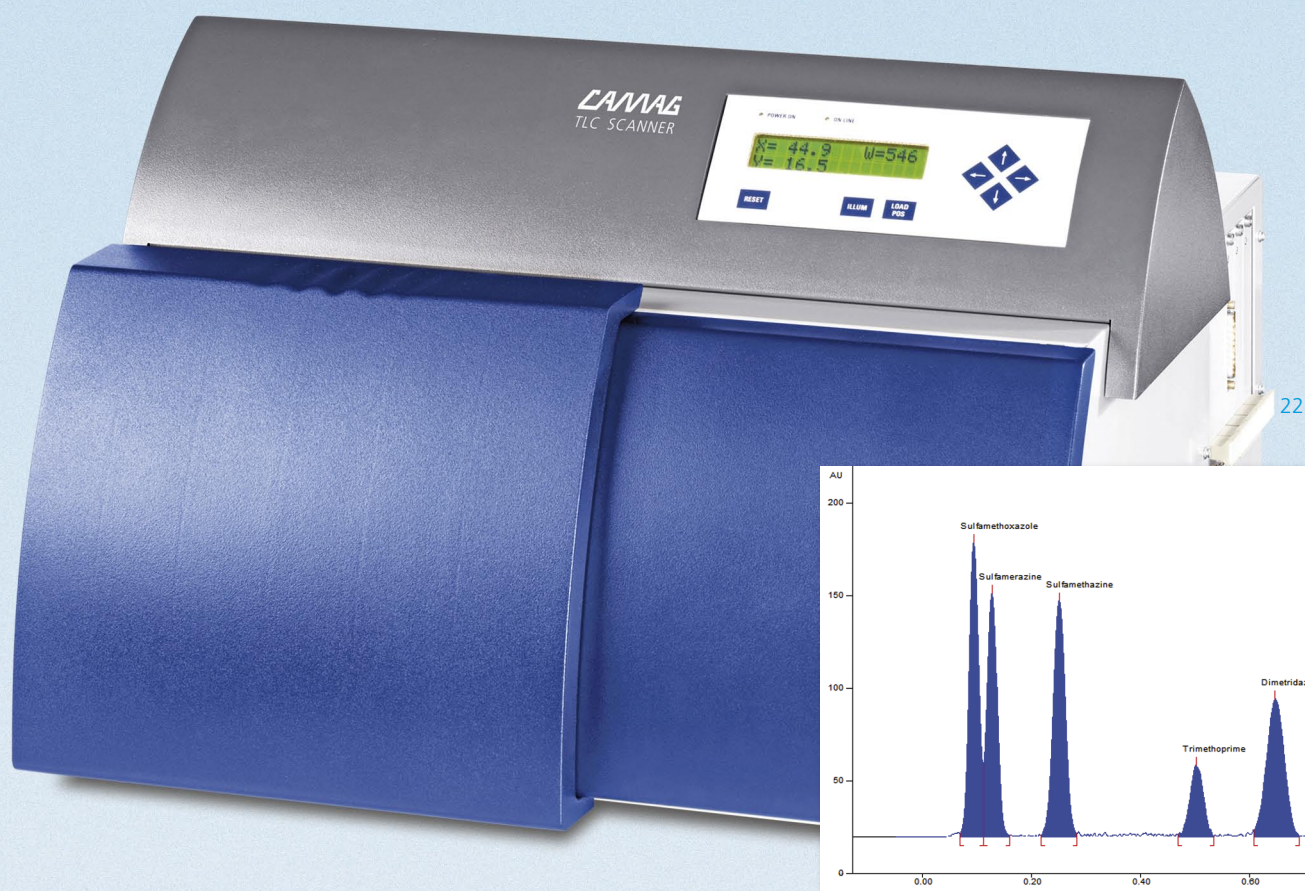
Additionally, for larger, aqueous sample volumes, the ATS4 option with a heated spray nozzle (up to 60 °C) is recommended as it allows a considerably reduced application time.

Further information is available on request from the authors.

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CAMAG TLC Scanner

CAMAG TLC Scanner with winCATS software is the most advanced workstation for densitometric evaluation of planar chromatograms.

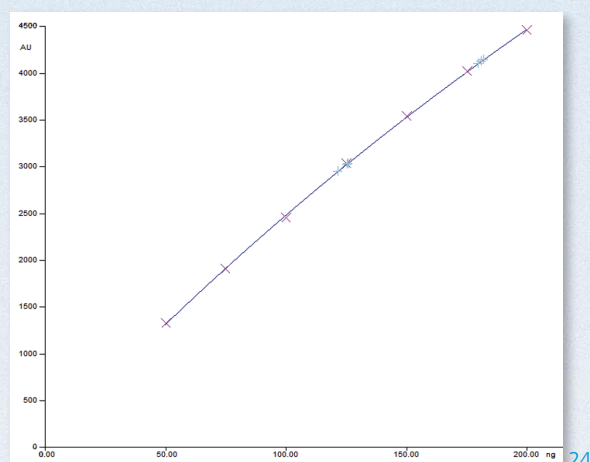


Key features:

- Measurement of reflection, either in absorption or fluorescence mode
- Object formats up to 200 x 200 mm
- Spectral range 190–900 nm
- Automatic start of all lamps: deuterium-, halogen-tungsten, and high pressure mercury lamp
- Data step resolution 25–200 μm
- Scanning speed 1–100 mm/s
- Spectrum recording up to 100 mm/s
- Automatic selection of electronic amplification
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Densitogram of antibiotics after their development



Calibration curve of sulfamethazine

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