CBS 110 • March 2013

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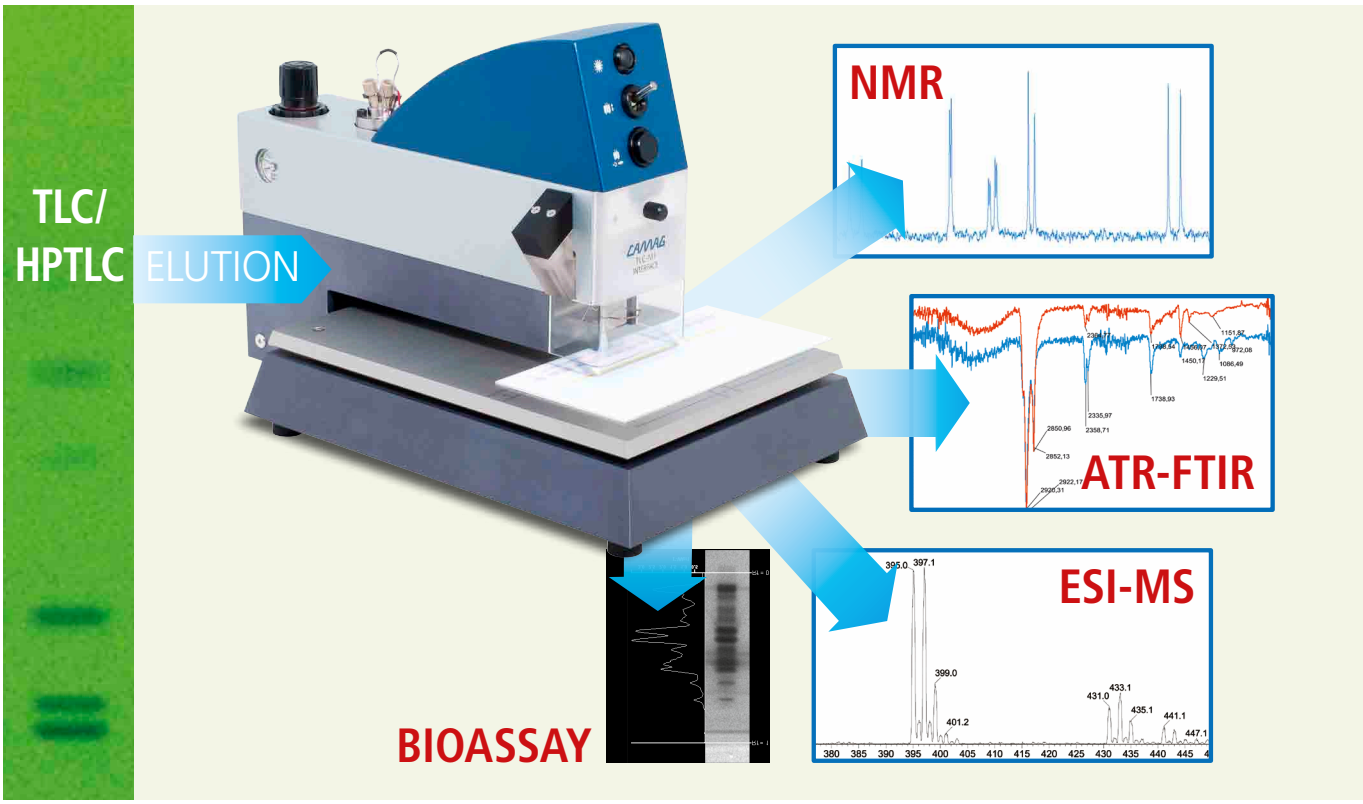

In this issue

Hyphenation of planar chromatography with several kinds of spectroscopy

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110

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No. 110, March 2013

CAMAG Bibliography Service
Planar Chromatography
Edited by Gerda Morlock
cbs@camag.com
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IN THIS ISSUE

Procedures, applications

Products featured in this issue

Column: Know CAMAG

CAMAG (Switzerland)
Sonnenmattstr. 11 • CH-4132 Muttenz 1
Tel. +41 61 467 34 34 • Fax +41 61 461 07 02
info@camag.com • www.camag.com

CAMAG Scientific Inc. (USA)
515 Cornelius Harnett Drive
Wilmington, NC 28401
Phone 800 334 3909 • Fax 910 343 1834
tlc@camag.com • www.camagusa.com

Rapid structure confirmation and quantitation by HPTLC-NMR

From left standing: B.Sc. Stefan Gaugler, Dr. Uta Scherer, Angelo Gössi, Prof. Dr. Götz Schlotterbeck, Stefan Wyss, André Büttler; seated: M.Sc. Timm Hettich and Agnes Baron

The research group of Prof. Schlotterbeck is working on coupling chromatographic methods with mass spectrometry with three purposes in mind: identification, assaying medical preparations for active pharmaceutical ingredients and their decomposition products and analysing natural products and endogen metabolites in biological matrices. The focus is on the application of mass spectrometric detection and NMR spectroscopy.

Introduction

Swift availability of spectroscopic data for structural elucidation is an important element of today's analyses in pharmaceutical research and related fields. Thereby planar chromatography becomes an efficient tool due to its speed and robustness. An important milestone was coupling of HPTLC with mass spectrometry [1].

Combining planar chromatography with nuclear magnetic resonance spectroscopy (NMR) had been discussed in 2010 [2] and has now been realized [3] as presented here. The advantage is that following an easy chromatographic separation, the rapid identification and quantitation using standard NMR equipment is possible without the need of special and expensive NMR hardware. In order to prove the function, three natural substances frequently occurring in plant extracts, *i.e.* caffeic acid, chlorogenic acid and rutin were transferred to offline HPTLC-NMR with the TLC-MS Interface. This illustrated that, in addition to high-resolution MS, ^{1}H NMR spectroscopy can be used for characterizing unknown substances on an HPTLC plate at concentrations above 10 µg/zone.

Standard solutions

For calibration by NMR 20 mg each of caffeic acid, chlorogenic acid and rutin hydrate were dissolved in 1 mL methanol-d4 and diluted 1:250 as stock solutions. These were diluted with methanol-d4 at 10, 25 and 40 µg/mL. Concentrations for NMR recovery determinations were 16.6 mg/mL caffeic acid, 15.0 mg/mL chlorogenic acid and 15.3 mg/mL rutin hydrate.

Layer

HPTLC plates silica gel 60 F_{254} (Merck), 20 x 10 cm, prewashed with methanol and dried under vacuum at 50 °C and ~93 hPa for 30 min.

Sample application

Bandwise with ATS4, band length 3 mm, track distance 6 mm; sample volumes for NMR recovery studies 1 µL caffeic acid, 1.3 µL chlorogenic acid and rutin (depicted are 1.3, 1.5, 1.8 and 2 µL).

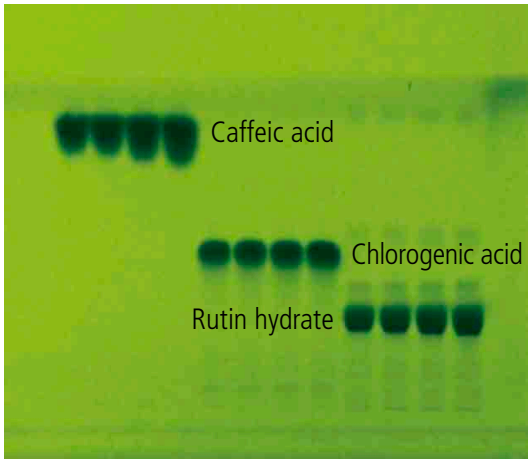
Chromatography

In twin-trough chamber after 5 min chamber saturation with formic acid – ethyl acetate – water – methyl ethyl ketone 5:30:6:18; developing distance 5 cm, drying time 5 min

Editor`s note: After 5 min preconditioning with solvent vapors only partial saturation will be achieved.

Documentation

With TLC Visualizer under UV 254 nm



1

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CAMAG
TLC-MS Interface

Hyphenating planar chromatography with mass spectrometry opens new possibilities of reliable identification of chromatographic fractions. This interface allows rapid and contamination-free elution of TLC/HPTLC zones with online transfer to the respective spectrometer. The advantage is its plug & play integration with any given HPLC/MS system without modification.

Depending on the MS system selected substances can be identified within a minute via its mass spectrum, or for unknown substance zones the respective sum formula can be obtained.

Since 2008 a number of applications using online coupling of HPTLC-MS have been presented whereby substance quantities of up to a few 100 ng were transferred into the mass spectrometer. This article describes the hyphenation of TLC-NMR whereby comparatively large amounts (> 10 µg) have to be transferred, *e.g.* for obtaining a proton-NMR spectrum to confirm a structural formula.

Merck Millipore introduced special precoated plates for such applications. Reference the detailed description in the contribution by Schulz *et al.* on this CBS p. 10–11.

These special plates were not yet available at the time this application was elaborated.

Elution with TLC-MS Interface

Elution of the respective zone into a vial using the round elution head with methanol at a flow rate of 0.3 mL/min for 6 min; evaporation of methanol under nitrogen, residue taken up with methanol-d4

NMR Spectroscopy

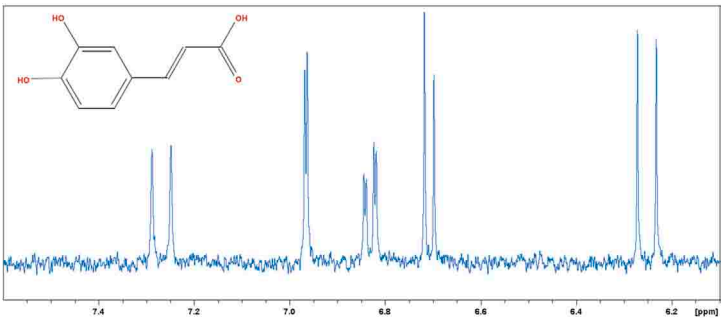
Measurement with 400 MHz Bruker Advance I instrument with 5 mm BBO sample head under TOPSPIN 2.1. Standard 1D proton spectra were recorded with WET (water suppression enhanced through T1 effects). 32678 data points with 256 transients at an acquisition time of 2 s and a spectral width of 8113 Hz were collected. Between transients 5 s relaxation delay was maintained. Before Fourier transformation, multiplication of the free induction decay (FID) with an exponential function (line broadening factor 0.5 Hz) was applied. Measuring time for quantitative NMR acquisition was 30 min.

Results and discussion

At first linearity of ^{1}H NMR spectroscopy of solutions in a concentration range between 10 and 80 µg/mL was investigated. Linearity for all substances in solution and the expected detection and determination limits (LOD, LOQ) could be confirmed. For direct combination of HPTLC and routine NMR spectroscopy, recovery and precision were investigated. For this purpose chromatogram zones were eluted with the TLC-MS Interface with methanol, evaporated to dryness and taken up with deuterized methanol. Recoveries were in the range of 100.5 % for chlorogenic acid and up to 103.4 % for caffeic acid, *i.e.* satisfactory as expected. Precision for these recoveries was all under 3.9 % (*%RSD*, n=3).

This shows that a direct structural confirmation as well as quantification can be achieved with a standard NMR instrument with good quality of the spectra. Exemplarily the ^{1}H NMR spectrum of a caffeic acid zone (15.6 µg/band) is shown after elution with the TLC-MS Interface and treatment as described.

^{1}H NMR	Measurements in solution			After elution from the plate		
Substance	Linearity r^2	LOD (µg)	LOQ (µg)	Quantity (µg/band)	Recovery (%)	*%RSD* (n=3)
Rutin	0.9976	2.3	6.9	20.3	101.8 ± 4.0	3.9
Caffeic acid	0.9978	2.5	7.3	17.1	103.4 ± 1.0	1.5
Chlorogenic acid	0.9991	3.3	10.1	19.6	100.5 ± 3.1	3.1



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Hyphenation of HPTLC-NMR is an interesting complementary method to the well established coupling of HPTLC-MS. Without investment in additional NMR hardware, the potential of NMR spectroscopy for structural confirmation and quantitation can be utilized. It is therefore a cost effective alternative to HPLC-NMR online coupling. The assaying of active pharmaceutical ingredients in preparations or in natural medicine extracts is possible in concentrations >10 µg/mL.

This article has shown that the straight-forward and robust operation of the HPTLC-MS Interface opens new possibilities in the analysis of pharmaceuticals and their degradation products as well as in the analysis of natural products.

Literature

[1] H. Luftmann, M. Aranda, G. Morlock, Rapid Commun. Mass Spectrom. 2007, 21, 3772

[2] G. Morlock, W. Schwack, Trends Anal. Chem. 2010, 29, 1157

[3] A. Gössi, U. Scherer, G. Schlotterbeck, Chimia, 2012, 66, 347

Further information is available from the authors on request.

Contact: Prof. Dr. G. Schlotterbeck, Institut für Chemie und Bioanalytik, Hochschule für Life Sciences, Fachhochschule Nordwestschweiz, Gründenstr. 40, CH-4132 Muttenz, goetz.schlotterbeck@fhnw.ch

Bioautographic HPTLC assays for screening of Gabonese medicinal plants used against Diabetes mellitus

Prof. Dr. Huguette Agnaniet

Dr. Anita Ankli

Professor H. Agnaniet and her team (O. Keita, R. Sanogo and L. Jacques), Laboratory for Natural Products, University of Masuku in Franceville, Gabon, investigate Gabonese medicinal plants for their bioactivity. In collaboration with Dr. Anita Ankli, CAMAG laboratory, various extracts of different plant species and plant parts were screened with bioautographic assays.

Introduction

The study of medicinal plants and their bioactivity is important in the search for new drugs, including their therapeutic application in ethanolic and aqueous extracts for infusions. The detected bioactivity can give hints of new indications for traditional medicines. In this extract screening study two different enzyme assays, *i.e.* α- and β-glucosidase (α-/β-glu.) and acetylcholinesterase (AChE) were used as well as the 2,2-diphenyl-1-picrylhydrazyl reagent (DPPH$^{\bullet}$) for detecting radical scavenging activity. The AChE enzyme test reveals substances which show activity towards the central nervous system and against Alzheimer's disease, whereas the α- and β-glucosidase test detects substances with antiviral properties and activity against Diabetes type 2. The advantages of the enzyme assays on HPTLC plates are the short incubation time of 20–60 min and the easy handling in a conventional laboratory. The extracts were also studied with *in vitro* and *in vivo* tests in Gabon.

Effect-directed bioautographic screening by HPTLC is an important tool in the search for new bioactive compounds from medicinal plants and for scientific information about the use of traditional medicines. Additional benefits of the technique are simplified sample preparation and instant results.

Sample preparation

100 g plant material collected in Franceville were extracted with ethanol 70 % and water. The extracts were dried by lyophilization or concentrated under reduced pressure. 200 mg dry extract were mixed with 10 mL ethanol – water 1:1, then sonicated for 10 min and centrifuged. The bark, leaf and roots of *Hua gabonii* were steam distilled. 10 µL of essential oil were dissolved in 1.5 mL toluene.

Layer

HPTLC plates silica gel 60 F_{254s} (Merck) 20 x 10 cm

Sample application

Bandwise with ATS4, band length 8 mm, distance from lower edge 8 mm, distance from left and right edge 20 mm, track distance min. 10 mm, application volumes 2 and/or 5 µL for samples and standard solutions.

Chromatography

Development in the ADC2 (20 min saturation, 33 % relative humidity, migration distance 70 mm) with

I Toluene – ethyl acetate 19:1

II Chloroform – methanol – water 35:15:2

III Ethyl acetate – acetic acid – formic acid – water 100:11:11:27

IV Acetonitrile – water – formic acid 15:4:1

V 1-Butanol – acetic acid – water 7:1:2

Derivatization

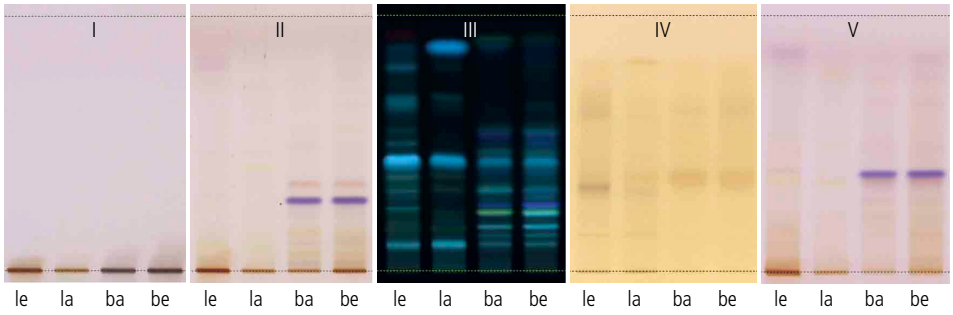
With anisaldehyde (for mobile phases I, II, and V), ninhydrin (for IV) and natural product reagent (for III)

Documentation

TLC Visualizer under UV 254 and 366 nm prior to derivatization; after derivatization under UV 366 nm and white light illumination (direct & transmission mode)

Results and discussion

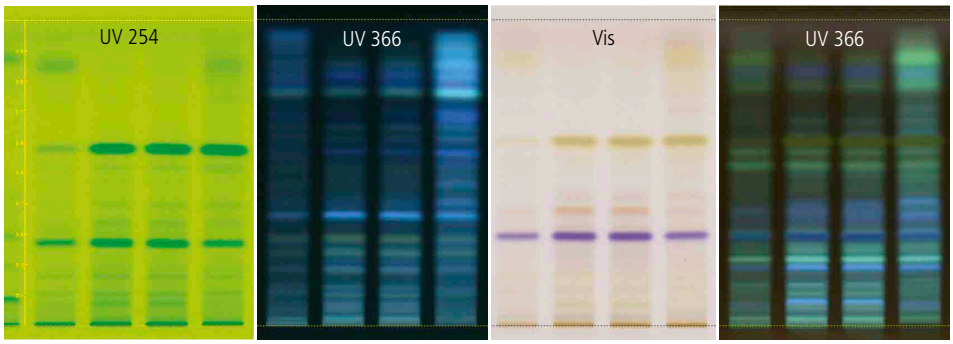
For leaf and bark extracts of *Nauclea diderrichii*, mobile phases II and III were selected for further investigations due to good separation of compounds and sharp zones.



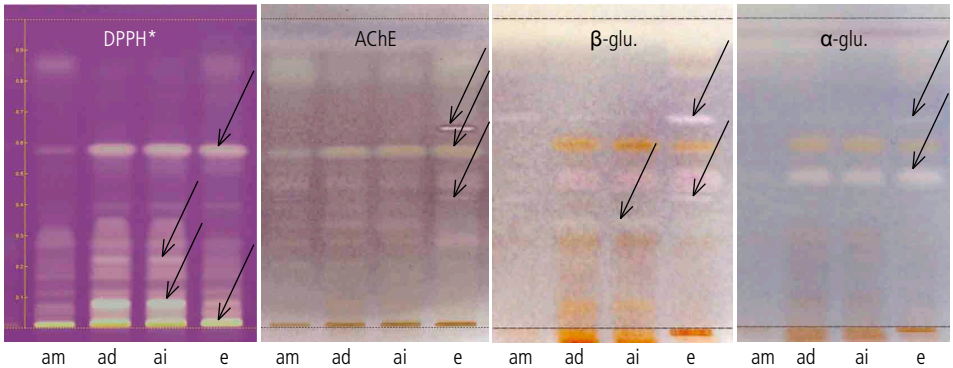
1

Ethanolic (e) and aqueous (a) extracts of Nauclea diderrichii leaf (l) and bark (b) separated with mobile phases I to V

The best fingerprint for *Sarcocephalus pobeguinii* was obtained with mobile phase II. The bioautographic assays showed some very prominent antioxidative zones and several inhibitors of AChE. Some of these zones show the same hR_F value as the antioxidative compounds. A very prominent zone was the distinct white zone of the ethanolic extract at hR_F 65. Possibly the same compound also showed inhibition of α- and β-glucosidase.



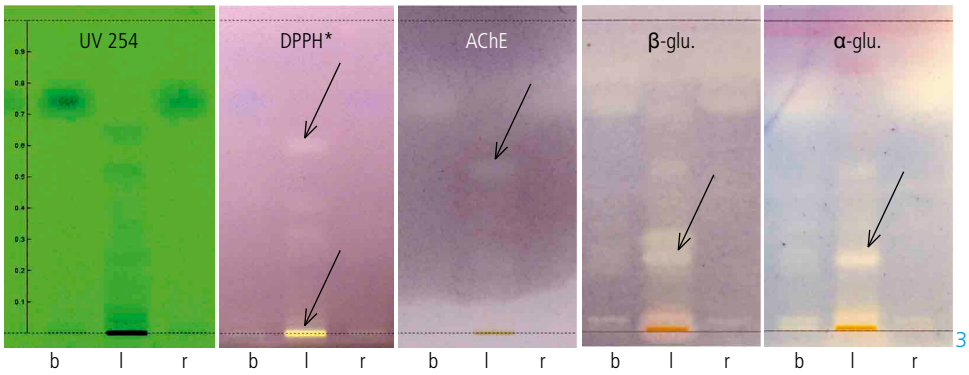
2a



2b

Ethanolic (e) and aqueous (a) extracts of Sarcocephalus pobeguinii bark by maceration (m), decoction (d) and infusion (i) with mobile phase II

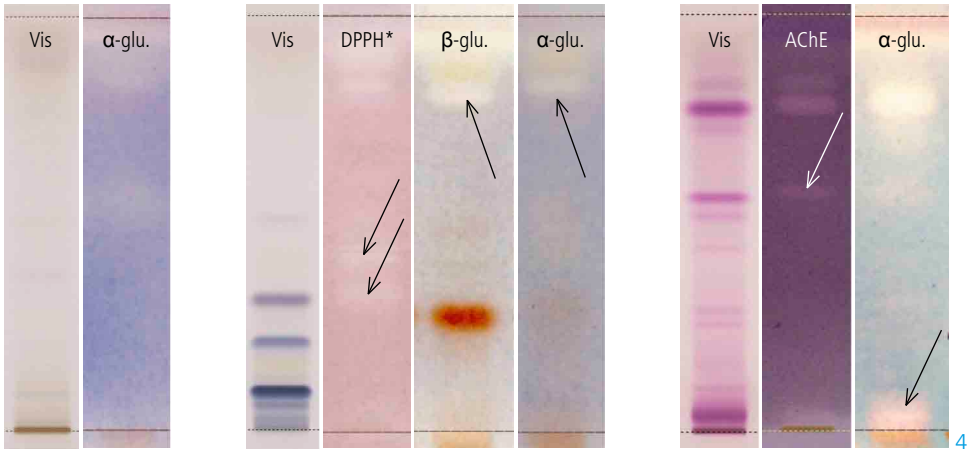
For the lipophilic compounds of the essential oil of *Hua gabonii* mobile phase I was selected. A characteristic zone at hR_F 75 seemed to be active in all three enzyme inhibition tests. The essential oil of the leaves showed the highest activities in all four bioassays. The zones at hR_F 23 showed strong β- and α-glucosidase inhibition, whereas the compound just above was only active for β-glucosidase.



3

Essential oil of Hua gabonii bark (b), leaf (l) and root (r) with mobile phase I

Morinda lucida and *Momordica foetida* are used against Diabetes mellitus in the African traditional medicine. The aqueous (not shown) and ethanolic leaf extracts of *Morinda lucida* showed a faint white zone for α-glucosidase inhibition. The ethanolic bark extract showed weak β- and α-glucosidase inhibition as well as some radical scavenging activities. The ethanolic extracts of *Momordica foetida* contains two active α-glucosidase inhibitiors. The AChE inhibition zones correspond to the pink zones visualized with anisaldehyde reagent.



4

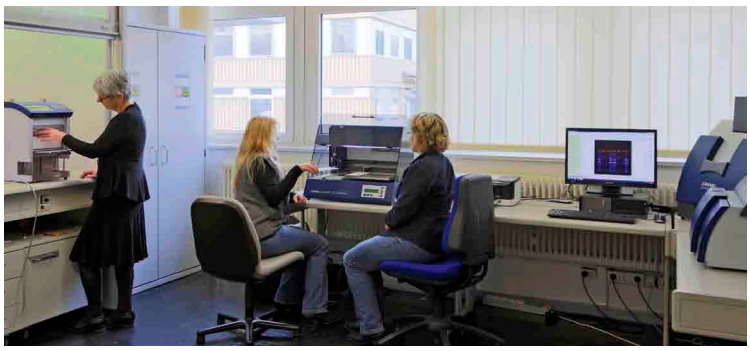
Ethanolic extracts of Morinda lucida leaf (left) and bark (middle) and of Momordica foetida plant (right) with mobile phase II

Effect-directed bioautographic screening by HPTLC proved to be an important tool in the search for new bioactive compounds from medicinal plants as it gave hints for successful indication of traditional medicine.

Further information is available from the authors.

Contact: Dr. Anita Ankli,
CAMAG Laboratory
Sonnenmattstr. 11, 4132 Muttenz,
Switzerland,
anita.ankli@camag.com

NEW
Customer support laboratory at CAMAG Berlin



Margit Werther

For decades the laboratory at our headquarters in Muttenz has promoted planar chromatography as an established analytical method worldwide. From our experience there we decided to set up a laboratory at CAMAG Berlin with the primary objective of supporting our domestic customers more efficiently. This became operative at the end of 2011 and is now available for all to profit from its truly customer oriented services.

Main activities of the CAMAG Berlin laboratory are:

- Feasibility studies for customers who are considering planar chromatography as their method for a given analytical task – either working directly with the customers or just with their samples.
- Demonstration of equipment in cases where on-site presentation at the customer's laboratory is not feasible, *e.g.* in cases where a variety of equipment is being considered.
- Instrument and/or software training for single persons or small groups in cases where their travel to Switzerland to attend regular courses is difficult to arrange. Irrespective of this new endeavor, we will maintain our offer to hold in-house courses at the customer's site, which we have successfully done for over 12 years.

Since the lab has been established at CAMAG Berlin, its services have been well received, indicating that it was the right move. Contact person for lab services in Berlin is Ms Margit Werther (margit.werther@camag.com).

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PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

Fast alle Beiträge in diesem CBS haben die Kopplung der Planar-Chromatographie mit spektroskopischen bzw. spektrometrischen Methoden zum Thema. Seit nunmehr 4 Jahren hat sich das TLC-MS-Interface als zuverlässiges, einfach zu bedienendes Bindeglied zwischen Planar-Chromatographie und Massenspektrometrie bei zahlreichen Aufgabenstellungen bewährt. Eine nicht unbeträchtliche Anzahl Analytiker haben die Verfügbarkeit zum Anlass genommen, ein komplettes HPTLC-System anzuschaffen, was den Bedarf bestätigt.

Der Einsatz des TLC-MS-Interfaces für weitere Kopplungen wurde bereits 2008 für die HPTLC-ATR-FTIR-Spektroskopie gezeigt, bei der man genauso vorging, wie es in diesem CBS nun erstmalig für die Kopplung mit Kernresonanzspektroskopie gezeigt wird. Die Eluate der interessierenden Zonen werden in Probengläschen aufgefangen, eingedampft und mit kleinen Volumina geeigneter Lösungsmittel aufgenommen. Merck hat für derartige Kopplungen nun spezielle Fertigschichten herausgebracht, die ein Vorwaschen der Schicht erübrigen.

Mit dem TLC-MS-Interface eröffneten sich neue Möglichkeiten, MS-, NMR- und ATR FTIR-Spektren von interessierenden Zonen nach online Elution aufzunehmen. Damit sind die vielfältigen Möglichkeiten der Kopplung bei weitem nicht ausgeschöpft, und über weitere werden wir zu gegebener Zeit berichten.

Mit freundlichen Grüssen

Gerda Morlock

Gerda Morlock
cbs@camag.com

Dear friends

This 110th issue of CBS almost exclusively deals with the hyphenation of planar chromatography with spectroscopic methods. For four years now the TLC-MS Interface has proven to be a reliable, convenient link between planar chromatography and mass spectrometry.
A considerable number of analysts have recognized its usefulness to the point that they have invested in a complete HPTLC system, confirming that such capability is vital to their work.

The application of the TLC-MS Interface for additional hyphenation was demonstrated for HPTLC-ATR-FTIR spectroscopy back in 2008 and this same protocol today is the one shown in this CBS for nuclear magnetic resonance spectroscopy for the first time. Eluates from the zones of interest are collected in vials, evaporated to dryness and taken up with small volumes of suitable solvents. Merck has now launched special plates for such hyphenations making plate prewashing unnecessary.

The TLC-MS Interface has opened up new options for recording MS, NMR and ATR FTIR spectra of zones of interest after online elution. Undoubtedly these versatile coupling possibilities will continue to unfold and we will be right here to report them.

Regards from Switzerland,

Gerda Morlock

Gerda Morlock
cbs@camag.com

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MARCH
2013

110

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

110 042 S. BHAWANI *et al.*, see section 18

110 124 J. PATEL *et al.*, see section 32

110 002 P.D. SETHI (Ed.): High-Performance Thin Layer Chromatography, Quantitative Analysis of Pharmaceutical Formulations, Volumes 1-3, CBS Publishers & Distributors, New Delhi, India (2013). The first volume provides a comprehensive introduction to the HPTLC technique, including details for each HPTLC step as well as various factors which influence the performance of a HPTLC analysis. Then presented over 3 volumes, 528 protocols for the HPTLC analysis of pharmaceutical formulations follow. Each protocol provides details on the preparation of samples and standards, chromatographic equipment, parameters for densitometric evaluation, chromatographic conditions, including stationary phase, mobile phase, standard and sample application, chamber saturation, relative humidity, quantity of mobile phase, temperature, migration distance and other critical parameters. References to the original publication are given as well as comments on the validation or any comparative study. Each protocol is illustrated with a typical densitogram, structures of the compounds analysed and overlaid UV spectra of compounds analysed (for selection of suitable wavelength for densitrometric scanning). All in all a comprehensive collection of protocols for pharmaceutical formulations.

quality control, pharmaceutical research, toxicology, HPTLC, densitometry, quantitative analysis, qualitative identification 1

110 003 J. SHERMA (Lafayette College, Department of Chemistry, Easton, PA 18042, shermaj@lafayette.edu): Biennial review of planar chromatography: 2009-2011. J. AOAC Int. 95, 992-1009 (2012). In his profound 22nd planar chromatography biennial review, the author presented the most important advances published between November 1, 2009 and November 1, 2011. The excellent review covered different aspects such as history, student experiments, books, reviews, theory and fundamental studies, chromatographic systems, apparatus and techniques, as well as quantitative analysis with a broad range of applications such as analysis of pharmaceuticals, herbal medicines, and dietary supplements, biological and clinical samples, foods and beverages, environmental samples and chemicals.

review, HPTLC 1b

110 004 E. TYIHÁK*, E. MINCSOVICS, Agnes M. MÓRICZ (*Plant Protection Inst. Hungarian Acad. of Sci., Herman O. Str. 15, POB 102, Budapest 1525, Hungary): Overpressured layer chromatography: From the pressurized ultramicro chamber to BioArena system. J. Chromatogr. A 1232, 3-18 (2012). A review on overpressured-layer chromatography (OPLC). OPLC is a separation technique that combines the advantages of conventional TLC/HPTLC with those of HPLC. Use of a special chromatoplate and a pump to increase and optimize the mobile phase flow velocity through an optional development distance in an adsorbent layer by employing the pressurized ultramicro (UM) chamber as a closed adsorbent layer chamber. Description of the versions of OPLC instruments, the character and achievement of off-line and on-line OPLC systems in analytical and preparative use. Demonstration of the unique advantages of planar-layer systems for detection, isolation and identification of new antimicrobials, antineoplastics, biopesticides and other biologically active substances as well as for studying fundamental biochemical reactions and mechanisms by BioArena which was newly developed as a complex bioautographic system.

qualitative identification, review, preparative TLC, quantitative analysis 1

2. Fundamentals, theory and general

110 005 Virgina COMAN*, S. KREIBIK, C. TUDORAN, Ocsana OPRIS, Florina COPACIU (*Babe-Bolyai University, »Raluca Ripan« Institute for Research in Chemistry, 30 Fantanele Street, 400294, Cluj-Napoca, Romania, coman_virginia@yahoo.com): Dielectroosmotic effects in electric current pulse. J. Planar Chromatogr. 25, 504-508 (2012). Physical properties of polar and non-polar solvents under electric current pulse were studied. In addition, solvents were classified according to their behavior in a pulsating electric field with applications in planar dielectrochromatography.

pharmaceutical research, qualitative identification, planar dielectrochromatography 2e

110 006 A. COZMA, L. VLASE, A. IGNAT, V. ZAHARIA, S, GOCAN*, N. GRINBERG (*Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, 11 Arany Janos, Cluj-Napoca 400082, Romania, gocansimion@gmail.com): Prediction of the lipophilicity of eight new *p*-toluenesulfonyl-hydrazinothiazole and hydrazine-bis-thiazole derivatives: a comparison between RP-HPTLC and RP-HPLC. J. Liq. Chromatogr. Relat. Technol. 35, 1444-1451 (2012). HPTLC of eight new p-toluenesulfonyl-hydrazinothiazole and hydrazine-bis-thiazole derivatives on silica gel RP-18 with mixtures of methanol/water in different methanol proportion between 50 to 70% with 5% increments as mobile phase. Quantitative determination by absorbance measurement at 254 nm. A chromatographic hydrophobicity index was determined and comparable results were obtained using HPLC.

pharmaceutical research, comparison of methods, HPTLC 2d

110 007 E. GOWIN, L. KOMSTA* (*Department of Medicinal Chemistry, Faculty of Pharmacy, Medical University of Lublin, Jaczewskiego 4, 20-090 Lublin, Poland, lukasz.komsta@umlub.pl): Revisiting thin-layer chromatography as a lipophilicity determination tool. Part III - a study on CN adsorbent layers. J. Planar Chromatogr. 25, 471-474 (2012). TLC of 35 simple compounds with known literature lipophilicity on cyano phase with increasing mixtures of methanol - water. Detection by absorbance measurement at 254 nm. Correlation between single RM values and lipophilicity values as well as dependences between the extrapolated log kW and experimental lipophilicity was described.

pharmaceutical research, qualitative identification, HPTLC 2d

110 009 Malgorzata JANICKA*, Katarzyna STEPNIK, Anna PACHUTA-STEC (* Department of Physical Chemistry, Faculty of Chemistry, Maria Curie-Sklodowska University, Maria Curie-Sklodowska Sq. 3, 20-031 Lublin, Poland): Quantification of lipophilicity of 1,2,4-triazoles using micellar chromatography. Chromatographia 75 (9-10), 449-456 (2012). Proposal of application of OPLC and TLC techniques with micellar mobile phases to evaluate the lipophilicity of 21 newly synthesized 1,2,4-triazoles, compounds of potential importance in medicine or agriculture as fungicides. The separation of the compounds 1) by micellar TLC on cyano phase with buffered SDS - tetrahydrofuran 4:1 in a sandwich chamber; 2) by micellar OPLC on cyano phase with buffered SDS - tetrahydrofuran 4:1 in off-line mode; 3) by reversed-phase TLC on RP-8 phase with buffered solutions of acetonitrile and tetrahydrofuran in concentrations varied in the range of volume fraction from 0.3 to 0.7, in constant steps of 0.1. Detection by densitometric scanning at UV 200 nm, or by means of a video camera at UV 254 nm. Application also of micellar HPLC technique on RP-8 column eluted with buffered SDS - acetonitrile 4:1, whereas in OPLC and TLC, cyano phases were applied, which allowed the use of micellar effluents in planar chromatography measurements. Determination of the micellar parameters log km and comparison with extrapolated R M0 values determined from reversed-phase TLC experimental data, as well as with log P values (Alog Ps, AClog P, Alog P, Mlog P, KowWin, xlog P2 and xlog P3) calculated

from molecular structures of solutes tested. Application of principal component analysis (PCA) and linear regression showed the results of considerable similarity between partition and retention parameters as alternative lipophilicity descriptors, and indicated micellar chromatography as a suitable technique to study lipophilic properties of organic substances.

pharmaceutical research, qualitative identification 2

110 010 R. KAISER (Institute for Chromatography, Bad Duerkheim, Germany, rudolf.kaiser@t-online.de): Correct calibration in planar chromatography. J. Planar Chromatogr. 25, 269-276 (2012). Different aspects such as selecting the center of the plate, repairing the unwanted start chromatography by using a solvent, avoiding mobile phase interaction that may influence hR_F values, improving signal integration by multi integration at mini stepwise positioning, applying statistical methods for outlier removal and data correlation of signal over substance mass were defined as important parameters to improve repeatability standard deviation.

review 2

110 011 Jadranka ODOVIC*, B.D. MARKOVIC, R.D. INJAC, S.M. VLADIMIROV, Katarina D. KARLJIKOVIC-RAJIC* (*Dep. of Anal. Chem., Univ. of Belgrade, Faculty of Pharm., Vojvode Stepe 450, 11221 Belgrade, Serbia): Correlation between ultra-high performance liquid chromatography-tandem mass spectrometry and reversed-phase thin-layer chromatography hydrophobicity data for evaluation of angiotensin-converting enzyme inhibitors absorption. J. of Chromatogr. A 1258, 94-100 (2012). Study of seven angiotensin-converting enzyme (ACE) inhibitors (enalapril, quinapril, fosinopril, lisinopril, cilazapril, ramipril, benazepril) to evaluate the correlation between their absorption and UHPLC-MS and RP-TLC hydrophobicity data (ϕ0 or C0 parameters, respectively). Their absorption values were in the range of 25-60 % and calculated KOWWIN logP values ranged from -0.94 to 6.61. In order to obtain reliable correlation ($r^2 = 0.7208$) between absorption and ACE inhibitors lipophilicity the solubility data (logS) must be considered, as independent variable, simultaneously with KOWWIN logP. Study of the relationships between literature available and absorption data predicted by multiple linear regression (MLR) using logS values besides chromatographically obtained hydrophobicity parameters C0 ($r^2 = 0.6424$) or ϕ0 ($r^2 = 0.6762$) indicates that these parameters could be used in ACE inhibitors absorption evaluation. The UHPLC-MS method provides the direct application of experimentally obtained ϕ0 values. Mathematical conversion of C0 parameters to logC0 values was necessary based on requisite for probability value of regression analysis ($P < 0.05$) for better MLR correlation of ACE inhibitors absorption with C0 parameters (RP-TLC) and logS. Definition of the accordance and differences between hydrophobicity parameters obtained by UHPLC-MS and RP-TLC.

HPTLC, qualitative identification 2

110 012 G. OROS, M. SZOGYI, T. CSERHATI* (*Research Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, P.O. Box 17, 1525, Budapest, Hungary, szogyim@t-online.hu): Relationship between the calculated physicochemical parameters and reversed-phase thin-layer chromatographic retention behavior of alkoxy-phenylbenzamide derivatives. J. Liq. Chromatogr. Relat. Technol. 35, 1314-1324 (2012). HPTLC of 16 alkoxy-phenylbenzamide derivatives on silica gel pre-coated overnight with *n*-hexane - paraffin oil 19:1, with a mixture of organic solvent - water. The ratio of the organic solvent component (*n*-hexane, acetone, ethyl alcohol, methyl alcohol, and dioxane) changed in steps of 5 vol %. Quantitative determination by absorbance measurement under UV light (wavelength not given). Lipophilicity parameters (lipophilicity, extrapolated to zero concentration of organic modifier; specific hydrophobic surface area) were calculated from the retention data.

pharmaceutical research, HPTLC, qualitative identification 2d

110 013 H. SHIMIZU-YUMOTO, N. HAYASHI*, K. ICHIMURA, M. NAKAYAMA (*Institute of Vegetable and Tea Science, National Agriculture and Food Research Organization, 2769 Kanaya-Shishidoi, Shimada, Shizuoka 428-8501, Japan): Slantingly cross loading sample system enables simultaneous performance of separation and mixture to detect molecular interactions on thin-layer chromatography. J. of Chromatogr. A 1245, 183-189 (2012). Anthocyanins are major flower pigments that can be affected by copigments, colorless compounds that can modify anthocyanin coloration. Application of TLC to separate and analyze anthocyanins and copigments. By slantingly cross loading samples compounds are symmetrically developed in various angles from the upper origin to individual R_F values and cross each other. Detection of copigments as color change on the developed line of anthocyanin. Demonstration by using pink sweet pea (*Lathyrus odoratus* L.) petals showing a more intense zone and a paler zone on the anthocyanin line. The zones were identified as kaempferol 3-rhamnoside and 2-cyanoethyl-isoxazolin-5-one. Kaempferol 3-rhamnoside is a flavonoid with general copigment effect of more intense and bluer coloration change, whereas the structure 2-cyanoethyl-isoxazolin-5-one is not a conventional copigment, it had a novel effect to change anthocyanin coloration paler while maintaining color tone.

herbal, pharmaceutical research, qualitative identification 2

110 014 K. SHWESHEIN, A. RADOICIC, F. ANDRIC, Z. TESIC*, D. MILOJKOVIC (*Faculty of Chemistry, University of Belgrade, P.O. Box 51, 11158 Belgrade, Serbia, ztesic@chem.bg.ac.rs): Hydrophilic interaction planar chromatography of geometrical isomers of selected Co(III) complexes. J. Liq. Chromatogr. Relat. Technol. 35, 1289-1297 (2012). TLC of fourteen geometrically isomeric Co(III) complexes: three pairs of neutral fac-mer isomers, one pair of cis-trans isomeric complexes of neutral type and three pairs of cationic cis-trans isomers, on silica gel with mobile phases consisting of different ratios of water, methanol and sodium chloride. The retention behavior of the Co(III) complexes was investigated, revealing that the most selective systems were chromatographic systems with low content of water. A significant effect of sodium chloride on the retention was observed.

pharmaceutical research, HPTLC 2c

3. General techniques

110 015 V. BEREZKIN*, A. CHAUSOV (*Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, Leninsky pr. 29, Moscow, 119991 Russia, berezkin@ips.ac.ru): The simple chromatographic chamber and its application in circular and linear TLC. J. Liq. Chromatogr. Relat. Technol. 35, 294-307 (2012). A new chamber configuration, in which the feeding of the mobile phase to the TLC plate is carried out directly over the sorption layer of the plate, was proposed. The chamber is suitable for linear and circular TLC in ascending, horizontal, and descending modes of development.

chromatographic chamber 3d

110 016 D. CSUPOR*, K. BOROS, A. HUNYADI, K. VERES, J. HOHMANN (*University of Szeged, Faculty of Pharmacy, Department of Pharmacognosy, Eötvös u. 6, Szeged, Hungary, csupor.dezso@pharmacognosy.hu): Validation of a densitometric method for the determination of theanine in tea extracts using CP atlas software. J. Planar Chromatogr. 25, 571-574 (2012). HPTLC of theanine in tea extracts on silica gel with *n*-butanol - acetone - acetic acid - water 7:7:2:4. Detection by dipping into a ninhydrin reagent for 3 s, followed by heating at 105 °C for 5-15 min. Quantitative determination by analysis of green channels of photographs using the CP Atlas 2.0 software. The hR_F of theanine was 35. Linearity was in the range of 1.4-14 ng/zone. The intermediate/inter-day/intra-day precision was below 0.7 % (n=3). Recovery (by standard addition) was

between 95.7 and 102.5 %.

quality control, herbal, quantitative analysis, HPTLC 3f, 18

110 017 J. DILLON*, J. APONTE, Y. TSAI, Y. HUANG (*Dep. of Chem., Brown Univ., 324 Brook Street, Providence, RI 02906, USA): Thin-layer chromatography in the separation of unsaturated organic compounds using silver-thiolate chromatographic material. J. of Chromatogr. A 1251, 240-243 (2012). Report of the use of silver-thiolate chromatographic material as a stable material for TLC. The stationary phase operated under the same principles as silver-ion chromatography, separating compounds by degree of unsaturation; however, showed considerable advantages over Ag-TLC in terms of light stability and shelf lifetime. Demonstration of the light stability and its application for separations based on the degrees of unsaturation. TLC of using fatty acid methyl esters (FAMEs) with hexane - ethyl acetate 9:1 and of polycyclic aromatic hydrocarbons (PAHs) with 100 % ethyl acetate.

qualitative identification 3b

110 018 D.S. JENSEN, Supriya S. KANYAL, V. GUPTA, M. A. VAIL, A.E. DADSON, M. ENGELHARD, R. VANFLEET, R.C. DAVIS, M.R. LINFORD* (*Dep. of Chem. & Biochem., Brigham Young Univ., Provo, UT 84602, USA): Stable, microfabricated thin-layer chromatography plates without volume distortion on patterned, carbon and Al_2O_3-primed carbon nanotube forests. J. of Chromatogr. A 1257, 195-203 (2012). Based on the recent description of the fabrication of TLC plates from patterned carbon nanotube (CNT) forests via direct infiltration/coating of the CNTs by low pressure chemical vapor deposition of silicon from SiH_4, followed by high temperature oxidation of the CNTs and Si, an improved microfabrication process for the preparation of these TLC plates has been presented. First, deposition of a few nanometers of carbon and/or a thin film of Al_2O_3 on the CNTs, confirmation of the presence of additional oxygen after carbon deposition by X-ray photoelectron spectroscopy, after priming, coating of the plates by rapid, conformal deposition of an inorganic material that does not require subsequent oxidation, *i.e.*, by a fast pseudo atomic layer deposition (ψ-ALD) of SiO_2 from trimethylaluminum and tris(tert-butoxy)silanol and faithful reproduction of the features in the masks is still observed after oxidation. Fast, highly efficient separations of the fluorescent dyes eosin Y disodium and sulforhodamine B were achieved on amino phase with LiCl - methanol 1:100 over 30 mm migration distance.

HPTLC 3b, 30

110 019 Olivia MARUTOIU, C. TIGAE, C. MARUTOIU*, I. KACSO, I. BRATU, Ioana PERHAITA (*Babe-Bolyai University Cluj-Napoca, Faculty of Orthodox Theology, 18 Piaba Avram Iancu Square, Cluj-Napoca, Romania, cmarutoiu@yahoo.com): Preparation and characterization of some ethyl-phenyl modified stationary phases. J. Planar Chromatogr. 25, 548-553 (2012). New modified stationary phases obtained by chemical modification of diatomaceous earth from Filia and silica gel with trimethoxyethylphenylsilane. Separations on ethyl-phenylmodified adsorbents are similar to the separation on C8.

pharmaceutical research, HPTLC 3b

110 020 Barbara MILZ, K. KLEIN, B. SPANGENBERG* (*University of Applied Sciences Offenburg, Institute of Process Engineering, Badstrasse 24, 77652 Offenburg, Germany, spangenberg@FH-Offenburg.de): Quantitative two-dimensional thin-layer chromatography using a diode-array detector. J. Planar Chromatogr. 25, 493-497 (2012). TLC of 12 sulfonamides on cyano phase with methyl tert-butyl ether - methanol - dichloromethane - cyclohexane - ammonia 25 % 48:2:2:1:1

in the first direction and with water - acetonitrile - dioxane - ethanol 8:2:1:1 in the second direction. The TLC plate was two-dimensional scanned and measured by use of a diode-array scanner.

pharmaceutical research, quantitative analysis 3f

110 021 A.J. OKO, S.R. JIM, M.T. TASCHUK, M.J. BRETT* (*Univ. of Alberta, Dep. of ECE, 2nd Floor ECERF, Edmonton, AB, Canada T6G 2V4): Time resolved chromatograms in ultra-thin layer chromatography. J. of Chromatogr. A 1249, 226-232 (2012). Ultrathin-layer chromatography (UTLC) is a recently developed analytical method intended for compact, rapid separations of nanolitre analyte volumes. However, new measurement techniques compatible with the millimetre length scales and rapid separation dynamics observed in UTLC are required for optimizing the performance of this method. A measurement system which records UTLC separations in full color with 32 μm spatial resolution and 33 ms temporal resolution has been designed, implemented and characterized. It features analysis of multiple tracks per plate, filtering of analyte zones by color, and automatic generation of time-resolved figures. By capture a wealth of information from a UTLC separation it provides insight into UTLC physics and improves the analytical performance.

HPTLC 3

110 022 P. ZARZYCKI*, Magdalena SLACZKA, Magdalena ZARZYCKA, Elzbieta WLODARCZYK, M. BARAN (*Section of Toxicol. & Bioanal., Dep. of Civil & Environm. Engineering, Koszalin Univ. of Technol., Sniadeckich 2, 75-453 Koszalin, Poland): Reprint of: application of micro-thin-layer chromatography as a simple fractionation tool for fast screening of raw extracts derived from complex biological, pharmaceutical and environmental samples. Anal. Chim. Acta 716, 54-60 (2012). Demonstration of the separation and detection capability of micro-TLC technique involving simple one step liquid extraction protocols of complex materials without multistep sample pre-purification. Isolation of the target components (cyanobacteria pigments, lipids and fullerenes) from complex matrices including spirulina dried cells, birds‘ feathers, fatty oils, and soot samples derived from biomass fuel and fossils-fired home heating systems. Completion of an isocratic separation protocol involving less than 1 mL of one component or binary mixture mobile phases within times of 5-8 min in each case, e.g. 1) micro-TLC of dyes and low-molecular mass compounds of cyanobacteria cells (S. platensis) extracted from pharmaceutical formulations on RP-18W phase with acetone - *n*-hexane 3:7 detection under daylight and densitometric evaluation; 2) separation of the main lipids fraction derived from bird feathers and oil samples (rapeseed, grapeseed, sunflower and olive oil) on RP-18 phase with dichloromethane - methanol 3:17, detection by spraying with 10 % phosphomolybdic acid in methanol and heating at 80 °C for 20 min; 3) screening of soot residues in dust samples derived from biomass fuel and fossils home heating systems for the presence of C60/C70 fullerenes on RP-18W phase with *n*-hexane, detection by densitometry at visible light, or at UV 254 nm or UV 366 nm.

environmental, quantitative analysis, qualitative identification, densitometry 3d

4. Special techniques

110 023 L. CIESLA*, I. KOWALSKA, W. OLESZEKA, A. STOCHMALA (*Department of Biochemistry and Crop Quality, Institute of Soil Science and Plant Cultivation - State Research Institute, 8 Czartoryskich Street, 24-100 Pulawy, Poland, lukecarpenter@poczta.onet.pl): Free radical scavenging activities of polyphenolic compounds isolated from *Medicago sativa* and *Medicago truncatula* assessed by means of thin-layer chromatography DPPH rapid test. Phytochem. Anal. 24, 47-52 (2013) TLC of 22 acylated phenolic compounds on silica gel with acetonitrile - water - chloroform - formic acid 12:3:2:1, followed by dipping into 0.2 % methanolic 2,2-diphenyl-1-picrylhdrazyl solution (DPPH radical reagent) for 5 s and kept in the dark for 30 min. Free radical scavenging activity of the acylated phenolic compounds was assessed by coupling with

the image processing software ImageJ.

pharmaceutical research, herbal, qualitative identification, densitometry 4e

110 024 H. GAD*, S. EL-AHMADY, M. ABOU, M. AL-AZIZI (*Department of Pharmacognosy, Faculty of Pharmacy, Ain Shams University, Abbassia, 11566-Cairo, Egypt, haidygad@gmail.com): Application of chemometrics in authentication of herbal medicines: a review. Phytochem. Anal. 24, 1-24 (2013) The application of chemometrics in combination with chromatographic fingerprining was reviewed. The authors described how this combination allowed to find correlation between different variables such as molecular profile and genetic variability and their geographical origins and growing conditions.

herbal, traditional medicine, quality control, review, HPTLC, qualitative identification 4e

5. Hydrocarbons and halogen derivatives

110 025 Anna SOBANSKA*, J. PYZOWSKI (*Department of Analytical Chemistry, Medical University of Lodz, ul. Muszynskiego 1, 90-151 Lodz, Poland, anna.sobanska@umed.lodz.pl): Simultaneous multiple-development HPTLC quantification of water- and oil-soluble sunscreens. J. Planar Chromatogr. 25, 344-348 (2012). HPTLC of two oil-soluble sunscreens, namely avobenzone (1) and octyl salicylate (2) and a water-soluble sunscreen, namely phenylbenzimidazol sulfonic acid (3) on silica gel with cyclohexane - diethyl ether 5:1 for (1) and (2) and ethyl acetate - ethanol - water 14:7:6 for (3). Quantitative determination by absorbance measurement at 300 nm for (2) and (3), and 360 nm for (1). Limits of detection and quantification were found to be 30 and 80 ng/zone for (1), and 20 and 60 ng/zone for both (2) and (3).

cosmetics, quality control, quantitative analysis, HPTLC 5b

7. Phenols

110 026 Silvia CORAN*, S. MULAS, Nadia MULINACCI (*Departimento Scienze Farmaceutiche, Universita di Firence, Via Ugo Schiff 6, 50019 Sesto Fiorentino, Firenze, Italy): Crucial aspects of high-performance thin-layer chromatography quantitative validation. The case of determination of rosmarinic acid in different matrices. J. Chromatogr. A 1220, 156-161 (2012). Description of a new method for determination of rosmarinic acid in different matrices by HPTLC on silica gel with toluene - ethyl formate - formic acid 6:4:1. Quantification by densitometry in absorbance mode at 330 nm. The influence of the main HPTLC operative parameters was figured out in view of a more stringent validation process. Together with the fundamental HPTLC instrumentation an automatic developing chamber is mandatory as it allows for control of the relative humidity and the saturation conditions and thus assures reproducibility. Several commercial preparations containing rosmarinic acid in different amounts were tested and rosmarinic acid in the range of 132-660 ng/band was found. The *%RSD* of repeatability and intermediate precision did not exceed 2.
pharmaceutical research, traditional medicine, herbal, food analysis, HPTLC, densitometry,

quantitative analysis, qualitative identification 7

110 027 M. HAWRYT, R. NOWAK, Monika HAJNOS* (*Department of Inorganic Chemistry, Faculty of Pharmacy, Medical University of Lublin, Chodzki 4A, 20-093 Lublin, Poland, monika.hajnos@am.lublin.pl): Two-dimensional thin-layer chromatographic determination of phenolic antioxidants from Eupatorium cannabinum extracts on cyano-bonded polar stationary phases. J. Planar Chromatogr. 25, 394-402 (2012). 2D-HPTLC of kaempferol, quercetin, rutin, hyperoside, ferulic acid, gallic acid, caffeic acid, chlorogenic acid, chinic acid, *p*-coumaric acid, catechin, epicatechin, and resveratrol in the flowers of *Eupatorium cannabinum* on cyano phase with propan-2-ol mixed with *n*-heptane, and ethyl acetate mixed with *n*-heptane as non-aqueous mobile

phases in the first direction and after turning the plate 90 ° with methanol mixed with water in the second direction of development. Detection by spraying with diphenylborinic acid 2-aminoethylester and PEG 4000 or DPPH radical reagent. Evaluation under UV 254 nm and 366 nm. The 2D-HPTLC system allowed the separation of the phenolic fractions.

herbal, quality control, qualitative identification, HPTLC 7

110 028 L. WANG (Wang Lili)*, Q.WANG (Wang Qunbo), J. HUANG (Huang Jianan), ZH. XU (Xu Zhongxi) (*National Research Center of Engineering & Technol. for Utilization of Functional Ingredients from Botanicals, Hunan Agr. Univ., Changsha 410128, China): (Optimization of the developing solvent system for the separation of catechin in tea by high-performance thin-layer chromatography) (Chinese) . J. of Hunan Agr. Univ. (Natural Sci. Edit.) 38 (1), 102-105 (2012). Catechins are flavanols and the group includes mainly epigallocatechin gallate (EGCG), epigallocatechin (EGC), epicatechin gallate (ECG), and epicatechin (EC), among which EGCG is in majority (50-80 %). Based on investigation of the TLC procedures in literatures for the separation of catechin in tea, an optimized HPTLC system was presented. HPTLC of the extracts of green tea and white tea on silica gel with chloroform - acetone - formic acid 125:75:22. Detection by spraying with 1 % vanillin in concentrated hydrochloric acid and heating at 105 °C for 3 min.

quality control, agricultural, qualitative identification, HPTLC 7

8. Substances containing heterocyclic oxygen

110 029 A. CHAUDHARY, P. KAUR, A. KATIYAR, B. SINGH* (*Natural Plant Products Division, CSIR-Institute of Himalayan Bioresource Technology, Palampur, Himachal Pradesh, 176 061, India, bikram_npp@rediffmail.com): HPTLC-densitometry method for simultaneous determination of major lignans and flavonoids in *Podophyllum hexandrum*. J. Planar Chromatogr. 25, 314-319 (2012). HPTLC of 4-odemethylpodophyllotoxin (1), podophyllotoxin (2), kaempferol (3), podophyllotoxone (4), and deoxypodophyllotoxin (5) in the rhizomes of *Podophyllum hexandrum* on silica gel with toluene - ethyl acetate 2:1 + 1 drop glacial acetic acid. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 1-8 µg/band for (1), (2) and (4) and 2-10 µg/band for (3) and (5). The intermediate/inter-day/intra-day precision was below 2 %. Average recovery for all (1) to (4) were between 96.4 and 101.8 %.

herbal, quality control, HPTLC, quantitative analysis 8a

10. Carbohydrates

110 030 D. HU (De-Jun Hu), K. CHEONG (Kit-Leong Cheong), J. ZHAO (Jing Zhao), S. LI (Shao-Ping Li)* (*State Key Laboratory of Quality Research in Chinese Medicine, Institute of Chinese Medical Sciences, University of Macau, Macao, China, lishaoping@hotmail.com): Chromatography in characterization of polysaccharides from medicinal plants and fungi. J. Sep. Sci. 36, 1-19 (2013). The application of TLC in the characterization of polysaccharides from medicinal plants and fungi were reviewed. Physicochemical and structural characterization as well as fingerprinting methods were also described.

review, HPTLC, quantitative analysis, qualitative identification 10b

11. Organic acids and lipids

110 031 P. BOCHENSKA, A. PYKA* (*Department of Analytical Chemistry, Faculty of Pharmacy, Medical University of Silesia, 4 Jagiellonska Street, PL-41-200 Sosnowiec, Poland, apyka@sum.edu.pl): Determination of acetylsalicylic acid in pharmaceutical drugs by TLC with densitometric detection in UV. J. Liq. Chromatogr. Relat. Technol. 35, 1346-1363 (2012). HPTLC of acetylsalicylic acid in tablets on silica gel with *n*-hexane - diethyl ether - acetic acid 7:2:1. Quantitative

determination by absorbance measurement at 200 nm. The hR_F value of acetylsalycilic acid was 18 and selectivity regarding matrix was given. Linearity was between 4.7 and 19.2 µg/zone. The intermediate/inter-day/intra-day precision was below 2 % (n=3). The limit of detection and quantification was 160 and 480 ng/zone, respectively. Recovery (by standard addition) was 100.4 %.

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

110 032 G. KAMATOU*, W. CHEN (Weiyang Chen), A. VILJOEN (*Department of Pharmaceutical Sciences, Faculty of Science, Tshwane University of Technology, Private Bag X680, Pretoria, 0001, South Africa, kamatougp@tut.ac.za): Quantification of rosmarinic acid in Salvia species indigenous to South Africa by HPTLC. J. Planar Chromatogr. 25, 403-408 (2012). HPTLC of rosmarinic acid in Salvia species on silica gel with ethyl acetate - toluene - formic acid 6:3:1. Quantitative determination by absorbance measurement at 328 nm. The hR_F value of rosmarinic acid was 42. Linearity was in the range of 200-1000 ng. The coefficient of variation (%) of intra-day and inter-day precision were 1.4 and 4.5, respectively (n=9). Recovery (by standard addition) was between 82.3 and 85.9 %.

herbal, HPTLC, quantitative analysis 11a

110 033 K. KUMAR, B. DASH, R. SINGH* (*Department of Chemistry, North Orissa University, Sriramchandra Vihar, Baripada, Mayurbhanja-757003, Orissa, India, rajeshks2001@yahoo.com): HPTLC quantification and antimicrobial activity of ursolic acid from *Diospyros melanoxylon*. J. Planar Chromatogr. 25, 320-325 (2012). HPTLC of ursolic acid in the leaves of *Diospyros melanoxylon* on silica gel with chloroform - methanol 19:1. Detection by dipping in 5 % methanolic sulphuric acid reagent and heating at 105 °C for 3 min. Quantitative determination by absorbance measurement at 540 nm. Linearity was in the range of 50-450 ng/zone for ursolic acid. Limits of detection and quantification were found to be 20 and 40 ng/zone. Recovery (by standard addition) was 97.5 %.

herbal, quality control, quantitative analysis, HPTLC 11a

110 034 S. VAN KERREBROECK*, H. PETIT, J. BEAUPREZ, I. BOGAERT, W. SOETAERT (*Laboratory of Industrial Biotechnology and Biocatalysis, Faculty of Bioscience Engineering, Ghent University, Coupure Links 653, 9000 Ghent, Belgium, simon.vankerrebroeck@ugent.be): The development of a detection method discriminating for mannosylerythritol lipids and acylglycerols. J. Planar Chromatogr. 25, 468-470 (2012). TLC of mannosylerythritol lipids (1), monoacylglycerol (2) and triacylglycerols (3) on silica gel with a mixture of formic acid 0.4 % in diethyl ether. The hR_F values of (1) were 3, 6 and 11, whereas the hR_F of (2) and (3) were 71 and 98, respectively. The method for analysis of biosurfactants allowed to discriminate mannosylerythritol lipids and acylglycerols.

pharmaceutical research, quality control, HPTLC, qualitative identification 11e

13. Steroids

110 035 H. FAN (Fan Hongmei)*, L. LI (Li Lingyun) (*Guizhou Provin. Qianxinan State Inst. of Food & Pharm. Test, Guizhou, Yixing 562400, China): (Quick screening of steroid hormones in some popular medicines) (Chinese). Chinese J. Ethnomed. Ethnopharm. 23, 69-71 (2011). In recent years, some unauthorized specific medicines were available as »traditional cure«, »folk recipe«, or »ancestral folk prescription« in China, such as powders and pills for relieving cough or capsules for rheumatic arthritis. It has been found that certain kinds of steroid hormones were added. Description of a quick method for screening of prednisone acetate and dexamethasone acetate in the chloroform extracts of the popular medicines by TLC on silica gel with dichloromethane - di-

ethyl ether - methanol - water 385:60:15:2. Detection by heating at 105 °C for 10 min followed by spraying with 2 % alkaline tetrazolium solution and viewing under daylight.

pharmaceutical research, traditional medicine, quality control, qualitative identification 13

110 036 S. MUSHARRAF*, Q. ARFEEN, M. SHOAIB (*Center for Molecular and Drug Research, International Center for Chemical and Biological Sciences, University of Karachi, Karachi-75270, Pakistan, musharraf1977@yahoo.com): Development and validation of TLC-densitometric method for the quantification of a steroidal drug, danazol in its pharmaceutical formulations. J. Planar Chromatogr. 25, 331-337 (2012). HPTLC of danazol on silica gel with hexane - acetone 3:2. Quantitative determination by absorbance measurement at 291 nm. The hR_F value of danazol was 55. Linearity was in the range of 200-1200 ng/zone. Limits of detection and quantification were 1 and 4 ng/zone.

pharmaceutical research, quality control, HPTLC, quantitative analysis 13

110 037 J. XING (Xing Junbo)*, H. CAO (Cao Hong), N. WU (Wu Ningqi) (*Inst. of Med. & Instrum. Test, Health Section of General logistics Dep. of the Chinese PLA, Beijing 100071, China): (Fast identification of hormones in traditional Chinese preparations by thin-layer chromatography) (Chinese). J. Trad. Chinese Med. & Pharm. Consult. 3 (7), 368-369 (2011). Hormones have been widely applied in clinical practice, however, overdose hormones are liable to cause general anaphylaxis, and even serious harm to physical health. For quick identification of hormones in traditional Chinese preparations, TLC of the extracts of the preparations on silica gel with chloroform - petroleum ether (30-60 ºC) - methanol 5:3:1, detection at UV 254 nm. Identification of methyltestosterone, prednisolone acetate, estrone, ethinyloestradiol, prednisolone acetate, testosterone undecanoate, fluocinolone acetonide, dexamethasone, betamethasone sodium phosphate, and hydrocortisone acetate by fingerprint comparison with the standards. The method has been applied to the real life samples of four varieties of preparation. The results were compatible with those obtained by HPLC and MS-MS.

pharmaceutical research, traditional medicine, quality control, qualitative identification, comparison of methods 13

14. Steroid glycosides, saponins and other terpenoid glycosides

110 038 H. LI (Li He)*, B. SONG (Song Bing), SH. ZHENG (Zheng Shimei), X. DING (Ding Xiaoying), Y. MA (Ma Yunchao), P. WANG (Wang Peiwu) (*Siping Munic. Administr. of Agr. Economy, Siping, Jilin Prov. 136000 China): (Development of a procedure for thin-layer chromatography of soybean saponin) (Chinese). Anhui Agri. Sci. Bull. 18 (1), 43-45 (2012). Soybean saponin is a component of soybean which has physiological activities and is composed of a variety of monomers. Presentation of a procedure for the separation of saponin extracted from soybean using ethanol. TLC on silica gel with 1) chloroform - ethyl acetate - methanol - water 15:40:22:10; 2) chloroform - ethyl acetate - methanol - water 3:4:2:1; 3) chloroform - ethyl acetate - methanol - water - acetic acid 3:4:2:1:0.05; 4) chloroform - ethyl acetate - methanol - water - acetic acid 30:80:40:20:1. Detection by spraying with 10 % sulfuric acid in ethanol and heating at 95 ºC until the zones are clearly visualized. Detection under UV 366 nm. The results showed that the mobile phase 1 gave the optimum separation of the soybean saponin.

quality control, agricultural 14

15. Terpenes and other volatile plant ingredients

110 039 L. PAILLAT*, Christine PÉRICHET, J.-P. PIERRAT, Sophie LAVOINE, J. FILIPPI, U. MEIERHENRICH, X. FERNANDEZ (*Charabot S.A., 10 avenue Yves Emmanuel Baudoin, 06130

Grasse, France): Purification of vetiver alcohols and esters for quantitative high-performance thin-layer chromatography determination in Haitian vetiver essential oils and vetiver acetates. J. of Chromatogr. A 1241, 103-111 (2012). Development of a simple, fast, and efficient HPTLC method for the simultaneous quantitative determination of alcohols and acetates in Haitian vetiver essential oils (*Chryzopogon zizanioides*) and its acetylated form. TLC on silica gel with *n*-hexane - chloroform - ethyl acetate 16:12:1 at 47 % relative humidity. Detection with vanillin - sulfuric acid reagent. Quantification of the analytes by densitometry in absorbance mode at 530 nm. Preparation of the reference mixtures of alcohols and acetates by fractionation of Haitian vetiver oil or vetiver acetates, followed by purification of the fractions of interest by means of OPLC. Determination of the chemical composition of each reference fraction by using GC-MS and GC×GC-MS, and of their overall purity by HPTLC with good linearity (r^2 = 0.9995 for alcohols, r^2 = 0.9996 for acetates) in a concentration range of 40-200 ng/zone.

herbal, quality control, HPTLC, qualitative identification, densitometry, quantitative analysis 15b

17. Amines, amides and related nitrogen compounds

110 040 U. HUBICKA, J. KRZEK*, Barbara WITEK (*Department of Inorganic and Analitycal Chemistry, Medical College of Jagiellonian University, 9 Medyczna Str, 30-688 Krakow, Poland, jankrzek@cm-uj.krakow.pl): TLC-densitometric determination of tolperisone and its impurities 4-methylpropiophenone and piperidine in pharmaceutical preparations. J. Liq. Chromatogr. Relat. Technol. 35, 1325-1335 (2012). HPTLC of tolperisone (1) and its impurities 4-methylpropiophenone (2) and piperidine (3) on silica gel with cyclohexane-1,4 - dioxane - isopropanol - ethanol 32:1:2:8 + 1 drop glacial acetic acid. Detection by dipping in a 0.3 % methanolic ninhydrin solution for 10 min, followed by heating at 105 °C for 5 min. Quantitative determination by absorbance measurement at 570 nm. The hR_F values of compounds (1) to (3) were 10, 76 and 60, respectively. Linearity was in the range of 60-1500 ng/band for (1), 90-400 ng/band for (2) and 40-250 ng/band for (3). Limits of detection and quantification were 20 and 60 ng/band for (1), 30 and 90 ng/band for (2) and 20 and 40 ng/band for (3), respectively. The intermediate precisions (level 2) for (1) to (3) were 1.6 %, 2.6 % and 2.4 % (n=5), respectively. Recovery for compounds (1) to (3) was between 84.6 and 99.7 %.

pharmaceutical research, quality control, HPTLC, qualitative identification 17

110 180 K. RANKOVIC, S. FILIPIC, K. NIKOLIC, D. AGBABA* (*Department of Pharmaceutical Chemistry, Faculty of Pharmacy, University of Belgrade, Vojvode Stepe 450, 11000 Belgrade, Serbia, danica@pharmacy.bg.ac.rs): TLC determination of tiapride hydrochloride and its impurities in pharmaceuticals. J. Liq. Chromatogr. Relat. Technol. 35, 1336-1345 (2012). HPTLC of tiapride hydrochloride and impurities III, VII, and VIII on silica gel with methylene chloride - methanol 45:8 + 1 drop ammonia. Quantitative determination by absorbance measurement at 240 nm. Migration distances were 61 mm, 19 mm, 25 mm and 32 mm for (1) and impurities III, VII, and VIII, respectively. Limits of detection for impurities III, VII, and VIII were 4, 7, and 6 ng/band, where as the limit of quantitation for impurities III, VII, and VIII were found equal to 10, 20, and 20 ng/band. Recovery for every concentration level was in the range of 95.1 % - 100.4 %.

pharmaceutical research, quality control, HPTLC, quantitative analysis 17

110 041 M. ROTHENHÖFER, ROSMARIE SCHERÜBL, G. BERNHARDT, J. HEILMANN*, A. BUSCHAUER (*Lehrstuhl für Pharmazeutische Biologie, Universität Regensburg, Universitätsstr. 31, 93040 Regensburg, Germany): Qualitative and quantitative analysis of hyaluronan oligosaccharides with high-performance thin layer chromatography using reagent-free derivatization on amino-modified silica and electrospray ionization-quadrupole time-of-flight mass spectrome-

try coupling on normal phase. J. of Chromatogr. A 1248, 169-177 (2012). Purified oligomers of hyalobiuronic acid are indispensable tools to elucidate the physiological and pathophysiological role of hyaluronan degradation by various hyaluronidase isoenzymes. Establishment and validation of a novel sensitive, convenient, rapid, and cost-effective HPTLC method for the qualitative and quantitative analysis of small saturated hyaluronan oligosaccharides consisting of 2-4 hyalobiuronic acid moieties. HPTLC on amino phase with 1-butanol - formic acid - water 3:5:2 or 3:4:1. Detection 1) by spraying with orcinol in various concentrations of sulfuric acid; 2) by dipping into the reagent of orcinol in 10 % sulfuric acid and Morgan-Elson reagent; 3) by illuminating with white light and UV 366 nm after heating. The simple reagent-free in situ derivatization of 3) resulted in a detection limit of 7-19 pmol/band and LOQ of 37-71 pmol/band depending on the analyzed saturated oligosaccharide. Identification of the analytes by TLC-ESI-MS. The validated HPTLC method, as an alternative to sequential techniques such as HPLC and CE, can easily be automated and is applicable to the analysis of multiple samples in parallel.

pharmaceutical research, HPTLC, qualitative identification, comparison of methods, densitometry, postchromatographic derivatization,quantitative analysis 17

18. Amino acids and peptides, chemical structure of proteins

110 042 S. BHAWANI*, M. MOHAMAD, O. SULAIMAN, R. HASHIM, A. MOHAMMAD, S. HENA (*Bhawani, School of Chemical Sciences, University Sains Malaysia, 11800, Pulau Pinang, Malaysia, sabhawani@gmail.com): Thin-layer chromatography of amino acids: a review. J. Liq. Chromatogr. Relat. Technol. 35, 1497-1516 (2012). The authors reviewed stationary phases, solvent systems, and detection reagents developed for the analysis of amino acids. Polar and nonpolar layers as well as impregnated layers mainly with metal ions and also with chiral agents were described for the separation and identification of amino acids. On the other hand, over fifty mobile phases were reviewed for the analysis of amino acids, with a recent tendency in the use of surfactants as less toxic reagents. Methodologies for the separation of amino acid enantiomers, such as the use of beta-cyclodextrin as chiral mobile phase as well as derivatization methods such as iodine azide reaction to enhance sensitivity in detection were also described. TLC has a privileged position due to its simplicity, convenience, and cost-effectiveness for separation of amino acids.

review, HPTLC 18a, 1b

110 016 D. CSUPOR *et al.*, see section 3

110 043 Monika DABROWSKA*, Emilia SIECZKA, M. STAREK (*Jagiellonian University Collegium Medicum, Department of Inorganic and Analytical Chemistry, 9 Medyczna St, 30-688, Poland, mtylka@cm-uj.krakow.pl): TLC assay of L-carnitine in dietary supplements. J. Planar Chromatogr. 25, 450-455 (2012). HPTLC of L-carnitine in dietary supplements on cellulose with methanol - water 5:1 + 1 drop glacial acetic acid. Detection by spraying with ninhydrin reagent. Quantitative determination by absorbance measurement at 420 nm. The hR_F of L-carnitine was 65. Linearity was in the range of 10-40 µg/zone. Limits of detection and quantification were 3 and 8 µg/zone, respectively. Precision (*%RSD*) was below 1.6 %. Recovery was in the range of 99.5 and 103.6 %.

food analysis, HPTLC, quantitative analysis 18a

110 044 A. SAHANA, S. DAS*, R. SAHA, M. GUPTA, S. LASKAR (*Dep. of Chem., The Univ. of Burdwan, Burdwan 713104, India): Identification and interaction of amino acids with leucine-anthracene reagent by TLC and spectrophotometry: experimental and theoretical studies. J. Chromatogr. Sci. 49 (8), 652-655 (2012). Synthesis of a new reagent by coupling anthracene moiety to L-leucine. The reagent has been characterized by different analytical techniques and is suitable

for easy identification of various amino acids on TLC plates by developing distinguishable colors with a detection limit between 0.1-0.5 µg in cold condition and 0.1-0.4 µg after heating. It was found that this reagent also binds with different amino acids very strongly in methanolic solution. Estimation of equilibrium binding constants with different amino acids showed that the values of the binding constants ranged from the lowest for L-tyrosine of 6.86 × 103 dm3/mol to the highest for L-arginine monohydrochloride of 8.86 × 105 dm3/mol at 25 °C. A theoretical study (Hartree-Fock) was also performed to investigate the interaction of the reagent with a representative amino acid (glycine).

qualitative identification, postchromatographic derivatization 18

21. Purines, pyrimidines, nucleic acids and their constituents

110 045 A. KOWALSKA*, K. PLUTA (*Department of Organic Chemistry, The Medical University of Silesia, Jagiellon´ska 4, 41-200 Sosnowiec, Poland, kowalska@sum.edu.pl): RP TLC assay of the lipophilicity of new azathioprine analogs. J. Liq. Chromatogr. Relat. Technol. 35, 1686-1696 (2012). Reversed-phase TLC of azathioprine and nineteen of its derivatives on RP-18 with acetone-TRIS buffer pH 7.4 mixtures containing amounts of acetone in the range 40-80 % (v/v) in 5 % increments; detection at UV 254 nm. The lipophilicity parameters of these compounds were determined experimentally and compared with theoretical values.

pharmaceutical research, qualitative identification 21

23. Other substances containing heterocyclic nitrogen

110 046 A. COZMA, V. ZAHARIA, A. IGNAT, S. GOCAN*, N. GRINBERG (*Univ. Babes-Bolyai, Anal. Chem., Dep., Cluj-Napoca, Romania): Prediction of the lipophilicity of nine new synthesized selenazoly and three aroyl-hydrazinoselenazoles derivatives by reversed-phase high performance thin-layer chromatography J. Chromatogr Sci. 50 (3), 157-161 (2012). Study of the lipophilicity of 12 new synthesized derivatives, the first eight compounds have as a basic chemical structure aryliden-hydrazino-selenazoles and the second group of the three compounds belongs to aroyl-hydrazinoselenazoles. HPTLC on RP-18 with methanol-water mixtures in different ratios. The linear correlation between RMw and the methanol-water ratios showed high values for the correlation coefficient. Determination of the chromatographic hydrophobic index by using the ratio -RMw/S, giving the values ranged between 99 and 73, and a good linear correlation between RMw and the slope. The log P values are also calculated. Formation of the matrices with RMw and log P and a principal component analysis (PCA) was implemented. Extraction of the information from PCA by plotting the obtained matrices. The compounds can be grouped by analyzing the scores into one containing nine compounds, and other containing three compounds, each group of compounds with the same basic chemical structure.

HPTLC, qualitative identification 23

27. Vitamins and various growth regulators

110 047 A. PYKA*, D. NABIALKOWSKA, K. BOBER, M. DOLOWY (*Department of Analytical Chemistry, Faculty of Pharmacy, Medical University of Silesia, 4 Jagiellon´ska Street, 41-200, Sosnowiec, Poland, apyka@sum.edu.pl): Comparison of NP-TLC and RP-TLC with densitometry to quantitative analysis of tocopherol acetate in pharmaceutical preparation. J. Liq. Chromatogr. Relat. Technol. 35, 2548-2564 (2012). HPTLC of tocopherol acetate in pharmaceutical preparation on silica gel with chloroform - cyclohexane 11:9. Quantitative determination by absorbance measurement at 272 nm. The hR_F values for tocopherol acetate and its related substance were 47 and 38, respectively. Limits of detection and quantification were 50 and 150 ng/zone, respectively. The intermediate/inter-day/intra-day precision was below 1.4 % (n=3). Recovery was between 99.8 and 101.5 %, respectively. A better separation of tocopherol acetate was obtained

using NP-TLC technique than by RP-TLC technique.

pharmaceutical research, quality control, quantitative analysis, HPTLC 27

28. Antibiotics, mycotoxins

110 048 V. OSTRY*, J. SKARKOVA, J. RUPRICH (*National Institute of Public Health, Center for Health, Nutrition and Food, Palackeho 3a, 612 42 Brno, Czech Republic, ostry@chpr.szu.cz): Densitometric high-performance thin-layer chromatography method for toxigenity testing of Alternaria alternata strains isolated from foodstuffs. J. Planar Chromatogr. 25, 388-393 (2012). HPTLC of alternariol (1), alternariol monomethyl ether (2), altenuene (3), L-tenuazonic acid (4) in Alternaria alternata strains isolated from foodstuffs on silica gel pre-coated with oxalic acid in methanol with toluene - ethyl acetate - formic acid 6:3:1. Quantitative determination by absorbance measurement at 254 nm. The hR_F values of compounds (1) to (4) were 25, 36, 49 and 30, respectively. The *%RSD* of repeatability were 7-19. Limit of detection for compounds (1) to (3) was 0.3 mg/kg and limit of quantification for (1) to (4) was 5.0 mg/kg in rice cultures with *Alternaria alternata mycelium*.

toxicology, quantitative analysis, HPTLC 28b

29. Pesticides and other agrochemicals

110 049 M. ACIKKOL*, S. SEMEN, Z. TURKMEN, S. MERCAN (*Institute of Forensic Sciences, Istanbul University, Istanbul, Turkey, acikkolm@istanbul.edu.tr): Determination of alpha-cypermethrin from soil by using HPTLC. J. Planar Chromatogr. 25, 48-53 (2012). HPTLC of alpha-cypermethrin in soil on silica gel with hexane - toluene 1:1. Quantitative determination by absorbance measurement at 220 nm. The hR_F values for alpha-cypermethrin was 38. Linearity was in the range of 12.5-1000 ng/zone. Limits of detection and quantification were 2 and 6 ng/spot, respectively. Precision was found in the range 1.7-6.3 % (n=6). Recovery was between 90.8 and 91.2 %.

environmental, HPTLC, quantitative analysis 29f

110 050 R. AKKAD, W. SCHWACK* (*University of Hohenheim, Institute of Food Chemistry, Garbenstrasse 28, 70599 Stuttgart, Germany, wolfgang.schwack@uni-hohenheim.de): Determination of organophosphorus and carbamate insecticides in fresh fruits and vegetables by high-performance thin-layer chromatography-multienzyme inhibition assay. J. AOAC Int. 95, 1371-1377 (2012). HPTLC of organophosphate and carbamate pesticides such as chlorpyrifos (1), paraoxon (2), parathion (3) and pirimicarb (4) in fresh fruits and vegetables on silica gel in an automatic development chamber with methanol - dichloromethane 1:9 and *n*-hexane - ethyl acetate - dichlormethane 13:4:3. Matrix interferences were visualized at 366 nm after dipping in primuline reagent (0.5 g/L in acetone - water 4:1). Detection by immersion in a solution of rabbit liver esterase or cutinase, followed by horizontal incubation for 30 min at 37 °C. The enzymatic reaction was stopped by heating the plate at 50 °C for 5-7 min. Staining was performed with a mixture of 60 mL Fast Blue Salt B (2.5 g/L in water) and 30 mL alpha-naphythyl acetate (2.5 g/L in ethanol). Recoveries were in the ranges of 86-109, 95-129, 96-114 and 90-111 % for pesticides (1) to (4), respectively. Mean *%RSD* was 8.5 for all samples.

food analysis, toxicology, HPTLC, quantitative analysis 29b

110 051 B. MILZ, I. IDROS, B. SPANGENBERG* (*University of Offenburg, Institute of Process Engineering, Badstrasse 24, 77652 Offenburg, Germany, spangenberg@FH-Offenburg.de): Limits of quantification of some neonicotinoid insecticides measured by thin-layer chromatography. J. Liq. Chromatogr. Relat. Technol. 35, 1404-1414 (2012). HPTLC of neonicotinoid insecticides nitenpyram (1), thiamethoxam (2), acetamiprid (3), imidacloprid (4), thiacloprid (5), and clothianidin

(6) on RP-18 with methyl-t-butyl ether - 2-butanone 5:2 + 1 drop ammonia. Quantitative determination by absorbance measurement at 300 nm. The hR_F values of (2) to (6) were 25, 42, 53, 55 and 63, respectively. Limits of quantification by UV detection were in the range from 12 ng to 26 ng/zone.

agricultural, HPTLC, quantitative analysis 29f

110 052 B. SPANGENBERG (University of Applied Sciences Offenburg, Institute of Process Engineering, Badstrasse 24, D-77652 Offenburg, Germany, spangenberg@HS-Offenburg.de): Standard addition method for the quantification of paraquat, diquat, difenzoquat, mepiquat, and chloromequat in water by thin-layer chromatography. J. Planar Chromatogr. 25, 262-268 (2012). HPTLC of paraquat (1), diquat (2), difenzoquat (3), mepiquat (4), and chloromequat (5) in water on silica gel with 1-propanol - methanol - 2.5 N NaCl 1:1:3. Quantitative determination by absorbance measurement between 500 and 535 nm. The hR_F values of compounds (1) to (5) were 21, 30 36, 52 and 56, respectively. Limits of quantification were 5 ng/zone for (1), 2 ng/zone for (2), 25 ng/zone for (3), 15 ng/zone for (4) and 9 ng/zone for (5). Recovery rates for compounds (1) to (5) were 50.7, 65.2, 59.6, 45.1 and 33.7 %, respectively.

environmental, quality control, toxicology, HPTLC, quantitative analysis 29d

110 053 T. TUZIMSKI (Department of Physical Chemistry, Chair of Chemistry, Faculty of Pharmacy with Medical Analytics Division, Medical University in Lublin, 4A Chodz´ki Street, 20-093 Lublin, Poland, tomasz.tuzimski@umlub.pl): Determination of pesticides in wine samples by HPLC-DAD and HPTLC-DAD. J. Liq. Chromatogr. Relat. Technol. 35, 1415-1428 (2012). HPTLC of prometryn in wine samples on silica gel with tetrahydrofuran - *n*-heptane 1:4 in the first direction and with methanol - water 7:3 in the second direction after the plate was turned by 90°. Quantitative determination by absorbance measurement at 223 nm. The hR_F value of prometryn was 35. Linearity was in the range of 220-1320 ng/zone. Limits of detection and quantification were 110 and 330 ng/zone. The method was compared with a validated HPLC-DAD method and both methods are useful for correct identification of pesticides in complicated mixtures.

toxicology, HPTLC, quantitative analysis, comparison of methods 29d

30. Synthetic and natural dyes

110 054 S. COBZAC, D. CASONI, A. FAZAKAS, C. SARBU* (*Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, Arany Janos 11, 400 028, Cluj-Napoca, Romania, csarbu@chem.ubbcluj.ro): Determination of food synthetic dyes in powders for jelly desserts using slit-scanning densitometry and image analysis methods. J. Liq. Chromatogr. Relat. Technol. 35, 1429-1443 (2012). HPTLC of azorubin (1) and sunset yellow (2) in powders for jelly desserts on silica gel with *n*-butyl alcohol - acetic acid - ethanol - water 10:2:1:5. Quantitative determination by absorbance measurement at 485 nm for (2) and 515 nm for (1). The hR_F values of (1) and (2) were 45 and 53, and selectivity regarding matrix was given. Linearity was between 100-400 ng/zone for (1) and 200-450 ng/zone for (2). The *%RSD* values for repeatability studies were below 2. The limits of detection and quantification were 9 and 19 ng/zone, for (1) and 11 and 23 ng/zone for (2), respectively. Recovery was higher than 96.9 % for (1) and 95.7 % for (2).

food analysis, quality control, HPTLC, quantitative analysis 30a

110 055 Simona COBZAC*, Dorina CASONI, S. POP (*Babes-Bolyai University, Faculty of Chemistry and Chemical Engineering, Arany Janos 11, 400028, Cluj-Napoca, Romania, codruta.cobzac@yahoo.com): Tartrazine determination from mustard sample by TLC-photodensitometry and TLC-digital processing of images. J. Planar Chromatogr. 25, 542-547 (2012). HPTLC of tartra-

zine in mustard on silica gel with isopropyl alcohol - ammonia 7:3. Quantitative determination by absorbance measurement at 425 nm (1) and by digital processing using a TLC scanner at an optical resolution of 300 dpi in visible mode followed by image digitalization (2). The hR_F of tartrazine was 61. Linearity was in the range of 32-878 ng/zone using (1) and 92-600 ng/zone using (2). Limits of detection and quantification were 5 and 11 ng/zone using (1) and 51 and 74 ng/zone using (2). Average recovery with both methods was greater than 100 %. Lower contents of tartrazine in mustard samples were determined when TLC image processing was applied for quantification probably due to poor sensibility of calibration with this method.

food analysis, quality control, quantitative analysis, HPTLC 30a

110 001 Florina COPACIU, Virginia COMAN*, Mihaela VLASSA, Ocsana OPRIS (*Babe-Bolyai University, Faculty of Environmental Science and Engineering, 30 Fântânele Street, 400294, Cluj-Napoca, Romania, coman_virginia@yahoo.com): Determination of some textile dyes in wastewater by solid phase extraction followed by high-performance thin-layer chromatography. J. Planar Chromatogr. 25, 509-515 (2012). HPTLC of textile dyes Lanasyn Blue F-L 150 (1), Lanasyn Dark Brown M-GLN (2), Lanasyn Red M-GA (3), Nylosan Dark Brown S-MBL (4), and Nylosan Red N-2RBL (5) on RP-18 with *n*-butanol - ethyl acetate - 5 % ammonium hydroxide 4:4:1. Quantitative determination by absorbance measurement at 550 nm. Linearity was in the range of 20-60 ng/band. Limits of detection for (1) to (5) were 7, 6, 3, 5 and 1 ng/band, respectively.

HPTLC, quantitative analysis, textile dyes 30a

110 018 D.S. JENSEN *et al.*, see section 3

110 056 Z. RODIC, B. SIMONOVSKA, A. ALBREHT, I. VOVK* (*EN-FIST Centre of Excellence, Dunajska 156, 1000 Ljubljana, Slovenia): Determination of lutein by high-performance thin-layer chromatography using densitometry and screening of major dietary carotenoids in food supplements J. of Chromatogr. A 1231, 59-65 (2012) Reversed-phase HPTLC of lutein, lycopene and beta-carotene standards on RP-18 (pre-washed by development with dichloromethane - methanol 1:1) with methanol - acetone 1:1 with 0.1 % of 2-tert-butylhydroquinone. The hR_F value was 4 of lutein esters, 24 of beta-carotene, 32 of lycopene, and 68 of lutein. Quantitative determination of lutein by densitometry at 450 nm. The repeatabilities were *%RSD* = 3.4, 1.3 and 1.6 at levels of 5 ng, 15 ng and 25 ng, respectively (n = 6). The calibration curve was best fit with a polynomial function in the range of 5-30 ng. The LOD was 1.5 ng, the LOQ 5 ng. With these chromatographic conditions also dietary carotenoids lutein esters, lycopene, free lutein and beta-carotene from food supplements were identified. It was shown that the standards remain stable on the plate for 1 h after chromatogram development.

quality control, food analysis, HPTLC, qualitative identification, densitometry, quantitative analysis 30b

110 057 A. SZABO, K. TAKACS, C. KALINAK, B. ERDELYI* (*Fermentia Ltd., Berlini u. 47-49, 1045 Budapest, Hungary, info@fermentia.hu): Thin-layer chromatographic method for separation of wheatgrass pigments on sucrose impregnated silica gel plates. J. Planar Chromatogr. 25, 361-362 (2012). HPTLC of pigments in wheatgrass on sucrose-impregnated silica gel prepared by dipping the plates into a sucrose solution (400 g granulated sucrose/1 L distilled water), followed by heating at 110 °C for 20 min. Development with *n*-hexane - acetone 37:13. Detection by absorbance measurement between 400 and 800 nm. Sucrose impregnation prevents the degradation of the pigments during the analysis.

herbal, quality control, pharmaceutical research, HPTLC, qualitative identification 30b

110 058 L. ZHANG (Zhang Lingyan)*, Z. PAN (Pan Ziqin), Q. QI (Qi Qun), J. RONG (Rong Jingyue), H. CHEN (Chen Hao) (*Coll. of Criminal Investigation, Southwest Univ. of Political Sci. & Law, Chongqing 401120, China): (Identification of the stamping time of atomic seal ink on paper by thin-layer chromatography) (Chinese). Chinese J. of Anal. Instruments (Fenxi Yiqi) (1), 58-62 (2012). The knowledge of the time when a document was created is one of the primary coverages in expert testimony. Especially the identification of the stamping time of atomic seal ink on paper is quite different from that of handwriting and common seals on paper, due to the special feature of its particular ingredient. The coloring agents such as bronze red C and phthalocyanine blue are not the same on paper and on developed silica plates because they may be oxidized and permeate gradually into the deeper layer of the paper. Also permeation of adjuvant materials to the color zone may cause cross-linked reactions. Thus the character of the ink in the extracts from the paper may be changed as time passes. The samples of atomic seal ink on paper with different stamping times were extracted with the optimized solvent system N,N-dimethylformamide - *n*-butanol - dichloromethane 2:1:1. TLC of the extracts on silica gel with *n*-amyl alcohol - cyclohexane - ethyl acetate 2:1:1. Determination of the coloring agent in the extracts by densitometry at 470 nm. Calculation of the extraction rate R = Ct / C, where Ct is the scanning peak area of the extracts obtained at different extraction duration and C is that obtained at the time when extraction reaches its equilibrium stage. Determination of the stamping time of a sample by using the calibration curves of the extraction rate against the stamping time. Demonstration of the method by determination of ten varieties of atomic seal inks containing bronze red C and and stamping times ranging from 3 to 36 months.

qualitative identification 30

32. Pharmaceutical and biomedical applications

110 059 N. ATIA*, S. AHMED (*Department of Pharmaceutical Analytical Chemistry, Faculty of Pharmacy, Assiut University, Assiut 71526, Egypt, nohanahedj@yahoo.com): A validated high-throughput chromatographic method for simultaneous determination of vitamin K homologues. J. Liq. Chromatogr. Relat. Technol. 35, 484-498 (2012). HPTLC of vitamin K homologues including phylloquinone (1), menaquinone-4 (2), and menaquinone-7 (3) on silica gel with methanol - ethanol - isopropanol - water 15:1:1:3. Quantitative determination by absorbance measurement at 254 nm. The hR_F values of compounds (1) to (3) were 56, 43 and 23, respectively. Linearity was in the range of 1-200 ng/band. Limits of detection and quantification were in the range of 0.2-0.9 and 0.7-2.5 ng/band, respectively. The intermediate/inter-day/intra-day precisions for (1) to (3) were in the range of 0.5-1.3 % (n=5). Recoveries were ranged from 95.3 to 100.8 %.

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

110 060 J. BADR*, F. BAMANE, N. EL-SHAER (*Department of Pharmacognosy, Faculty of Pharmacy, Suez Canal University, Ismailia 41522, Egypt, jihanbadr2010@hotmail.com): Application of high-performance thin-layer chromatography for determination of nicotine in different brands of cigarettes. J. Liq. Chromatogr. Relat. Technol. 35, 1213-1221 (2012). HPTLC of nicotine in cigarettes on silica gel with *n*-hexane - methylene chloride - methanol 4:16:3. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 0.1-1 mg/mL. Precision was estimated with an *%RSD* below 2.0. Limits of detection and quantification were 0.008 mg/mL and 0.02 mg/mL, respectively. The method provides acceptable intra-day and inter-day precision for nicotine. Recovery was between 97.5 and 98.4 %, respectively.

toxicology, HPTLC, quantitative analysis 32d

110 061 Anindita BEHERA*, Dannana SANKAR, S. MOITRA, S. SI (*School of Pharmaceutical Sciences, Siksha »O« Anusandhan University, Bharatpur, Ghatikia, Bhubaneswar, Orissa, India, an-

indita02@gmail.com): Densitometric thin-layer chromatography of protease inhibitors in pharmaceutical preparations. J. Planar Chromatogr. 25, 374-379 (2012). HPTLC of atazanavir sulfate (1) and ritonavir (2) in combined dosage forms on silica gel with toluene - methanol - glacial acetic acid - ethyl acetate 14:1:3:4. Quantitative determination by absorbance measurement at 254 nm. The hR_F values for (1) and (2) were 50 and 63, respectively. Linearity was in the range of 30-300 ng/zone for (1) and 10-100 ng/zone for (2). The limit of detection and quantification was 16 and 49 ng/zone for (1) and 18 and 55 ng/zone for (2), respectively. The intermediate/inter-day/intra-day precision was below 0.7 % (n=6). Recovery for (1) and (2) was in the range of 99.6-100.0 %.

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

110 062 Anindita BEHERA*, K. SETHY, Dannana SANKAR, S. MITRA, S. SI (*School of Pharmaceutical Sciences, Siksha O Anusandhan University, Bharatpur, Ghatikia, Bhubaneswar - 751003, Orissa, India, anindita02@gmail.com): Statistical correlation and simultaneous estimation of atazanavir sulfate and ritonavir in fixed dosage form by high performance liquid chromatography and high-performance thin-layer chromatography. J. Liq. Chromatogr. Relat. Technol. 35, 1731-1749 (2012). HPTLC of atazanavir sulfate (1) and ritonavir (2) in fixed dosage on silica gel with toluene - methanol - glacial acetic acid - ethyl acetate 14:1:3:4. Quantitative determination by absorbance measurement at 254 nm. The hR_F values of (1) and (2) were 50 and 63, respectively. Linearity was 30-300 ng/zone for (1) and 10-100 ng/zone for (2). The intermediate/inter-day/intra-day precision was 0.3 % for (1) and 0.7 % for (2) (n=6). The limit of detection and quantification was 16 and 49 ng/zone for (1) and 18 and 55 ng/zone for (2), respectively. Recovery (by standard addition) was 99.9 % for (1) and (2). The results were in accordance with those by a validated HPLC method.

pharmaceutical research, quality control, HPTLC, quantitative analysis, comparison of methods 32a

110 063 S. BORISAGAR, H. PATEL*, C. PATEL (*Department of Quality Assurance, Shree Sarvajanik Pharmacy College, Nr. Arvind Baug, Mehsana 384001, Gujarat, India, harshaupatel@yahoo.co.in): A validated stability-indicating HPTLC method for the estimation of gemcitabine HCl in its dosage form. J. Planar Chromatogr. 25, 77-80 (2012). HPTLC of gemcitabine on silica gel with toluene - methanol - chloroform 6:6:5. Quantitative determination by absorbance measurement at 268 nm. The hR_F value of gemcitabine was 48. Linearity was in the range of 500-3000 ng/band. Intraday and interday precisions (*%RSD*) were 1.4-1.6 % and 1.6-1.8 %, respectively. Recovery was found to be in the range of 98.3-101.8 %.

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

110 064 R. BRAZ, Luciana WOLF, G. LOPES, J. DE MELLO* (*Programa de Pós-graduação em Ciências Farmacêuticas, Universidade Estadual de Maringá Av. Colombo, 5790, 87020-900 Maringá-PR, Brazil, mello@uem.br): Quality control and TLC profile data on selected plant species commonly found in the Brazilian market. Brazilian Journal of Pharmacognosy 22, 1111-1118 (2012). TLC of *Schinus terebinthifolius* (1), *Arctium lappa* (2), *Trichilia catigua* (3), *Camellia sinensis* (4), *Mikania glomerata* (5), *Croton moritibensis* (6), *Achyrocline satureioides* (7), *Heteropterys aphrodisiaca* (8), *Plantago major* (9) and *Arctostaphylos uva-ursi* (10). The hR_F value of caffeic acid in (7) and (9) was 35, chlorogenic acid in (2) and (9) was 58, gallic acid in (1) and (10) was 20, *o*-coumaric acid in (5) was 39, catechin in (1) was 81, cinchocain Ib in (3) was 58, coumarin in (5) was 79, epicatechin in (4) was 80 and epicatechin-3-O-gallate in (4) was 66. Summary of the applied TLC methods: silica gel layer for all plants. Mobile phases: (1) Toluene - ethyl acetate - methanol - formic acid 75:25:10:6; (2) ethyl acetate - toluene - formic acid - water 16:2:1:1; (3)

ethyl acetate - water - formic acid - acetic acid 100:27:11:11, (4) chloroform - acetic acid - methanol - water 16:8:3:2; (5) toluene - dichloromethane - acetone 9:5:6; (6) toluene - dichloromethane - acetone 9:6:5; (7) ethyl acetate - formic acid - water 18:1:1. Detection by spraying with (A) ferric chloride 1 % in methanol; (B) vanillin perchloric acid, followed by heating at 105 ºC for 5 min; (C) diphenylboric acid 2-amino-ethyl ester and polyethylene glycol 400, evaluation at UV 366 nm; (D) potassium hydroxide 10 %, evaluation at UV 366 nm; (E) anisaldehyde, followed by heating at 105 ºC for 5 min. For plant 1, mobile phase (MP) (1) and detection (det) A as well as MP (2) and det B was applied, for plant 2, MP (3) and det C, for 3, MP (4) and det B, for 4, MP (2) and det B, for 5, MP (5) and det D, for 6, MP (6) and det E, for 7, MP (1) and det C, for 8, MP (7) and det A, for 9, MP (1) and det C, for 10, MP (1) and det A.

quality control, herbal, qualitative identification 32e

110 065 SH. CAI (Cai Shujuan)*, G. XING (Xing Guirong), Z. WANG (Wang Zhiping) (*Tangshan Inst. for Drug Control, Tangshan, Hebei 063000, China): (Study of the quality standard for Chuangshang No 1 compound oral liquid) (Chinese). J. China Pharm. Univ. 20 (9), 27-28 (2011). Chuangshang No 1 compound oral liquid is a herbal TCM effective specially for promoting blood circulation for removing blood stasis and relieving pain. TLC of the extracts of the medicine 1) for Angelica sinensis and *Ligusticum wallichii* on silica gel with *n*-hexane - ethyl acetate 9:1, detection by viewing under UV 366 nm; 2) for Stigma Croci on silica gel with ethyl acetate - methanol - water 200:33:27, detection by viewing under daylight; 3) for *Radix Paeoniae rubrathe* (unpeeled root of common peony) on silica gel with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 4:1 and heating until the zones were visualised.

pharmaceutical research, quality control, herbal, qualitative identification 32e

110 066 SH. CAO (Cao Shujuan)*, L. LING (Ling Liying), Y. ZHANG (Zhang Yaya) (*Tangshan Municip. Inst. of Drug Contr., Hebei, Tangshan 063000, China): (Study of the method for the quality control of Zhongtongxiao capsules) (Chinese). J. China Pharm. Univ. 21 (20), 34-35 (2012). Zhongtongxiao capsules are a herbal TCM preparation to treat blood stasis and sore swellings caused by fracture. TLC of the extracts of the medicine on silica gel 1) for *Angelica sinensis* and the rhizome of Chuanxiong, with *n*-hexane - ethyl acetate 9:1, detection at UV 366 nm; 2) for *Radix Paeoniae*, with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in ethanol - sulfuric acid 200:1 and heating at 105 °C until the zones are visible, viewing in daylight; 3) for Teasel, developed with toluene - ethanol 9:2, detection by spraying firstly with potassium iodobismuthate - hydrochloric acid - water 10:1:200 and then with 5 % nitrous acid in ethanol, followed by viewing in daylight; 4) for Licorice, with the lower phase of chloroform - methanol - water 13:7:2, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones are visible, viewing in daylight.

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

110 067 J. CHANG (Chang Jimei)*, J. CHANG (Chang Juan) (*Affiliated Hosp. with Xinxiang Med. Coll., Henan, Xinxiang 453003, China): (Preparation of Qichai Hugan pills and its quality control) (Chinese). Chinese J. Prac. Med. 7 (3), 251-252 (2012). Presentation of a procedure for preparation and quality control of Qichai Hugan pills. TLC of the extracts 1) for *Astragalus mongholicus*, on silica gel with the lower phase of chloroform - ethyl acetate - methanol - water 10:20:11:5, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C for 5 min and viewing under daylight and UV 366 nm; 2) for the root of Chinese thorowax, on silica gel with the lower phase of chloroform - methanol - water 7:3:1, detection by spraying with a

solution of *p*-dimethylaminobenzaldehyde - sulfuric acid - ethanol 1:2:18 followed by heating at 105 °C and viewing under daylight; 3) for the fruit of Chinese wolfberry, on silica gel with toluene - ethyl acetate - formic acid 15:5:2, detection under UV 366 nm; 4) for the root of red-rooted salvia, on silica gel with chloroform - acetone - formic acid 60:5:2, detection by spraying with a solution of 2 % ferric chloride - 1 % potassium ferricyanide 1:1 and viewing under daylight.

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

110 068 J. CHEN (Chen Jinling), X. HUANG (Huang Xuesong)* (*Coll. of Sci. & Engineering, Jinan Univ., Guangzhou 510632, China): (Separation of fructo oligosaccharides (FOS) in garlic by thin-layer chromatography) (Chinese). Chinese J. of Guangdong Agr. Sci. (9), 103-105 (2012). Garlic (*Allium sativum* L.) contains high levels of carbohydrates (up to 75 %), the main of which are fructo oligosaccharides (FOS). FOS have antioxidative activity. The quality control of garlic has to monitor the content of FOS because it changes during its storage. TLC of the extracts of garlic on silica gel twice with *n*-butanol - isopropanol - water - acetic acid 7:5:4:2, detection by spraying with vanillin - sulfuric acid - ethanol - water 3:13:81:26 and heating at 100 °C for 5 min. Up to seven monosaccharides in garlic are well separated by using the procedure.

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

110 069 L. CHENG (Cheng Lihui)*, L. LU (Lu Lixia) (*Guangdong Inst. of Pharm., Guangzhou 510627, China): (Study of the method for the quality control of Compound Prescription Cortex Phellodendri Chinensis fluid) (Chinese). Chinese J. of Subtropical Plant Sci. 41 (2), 13-18 (2012). Compound Prescription *Cortex Phellodendri Chinensis* fluid is a herbal TCM preparation, having remarkable curative effect to osteomyelitis, swelling and ulcer on the body surface and traumatic infection. For quality control, TLC on silica gel 1) for *Cortex Phellodendri Chinensis* with *n*-butanol - glacial acetic acid - water 9:2:1, detection by viewing at UV 366 nm, identification by fingerprint comparison in parallel with the standard drug and berberine chloride standard submitted to the same procedure; 2) for *Flos Lonicerae* and *Fructus Forsythiae* with the upper phase of ethyl acetate - formic acid - water 14:5:5, detection by viewing at UV 366 nm, identification by fingerprint comparison in parallel with the standard drug and chlorogenic acid standard submitted to the same procedure.

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

110 070 S. DEVKAR, Y. BADHE, S. JAGTAP, M. HEGDE* (*Interactive Research School for Health Affairs, Bharati Vidyapeeth Deemed University, Medical College Campus, Pune-411043, India, mahabaleshwarh@yahoo.com): Development and validation of an HPTLC method for simultaneous estimation of thiocolchicoside and aceclofenac in combined dosage form. J. Planar Chromatogr. 25, 290-294 (2012). HPTLC of withaferine A (1), 1,2 deoxy-withastramonolide (2), withanolide A (3), and withanolide B (4) in *Withania somnifera* on silica gel with dichloromethane - toluene - methanol - acetone - diethyl ether 15:15:6:2:2. Quantitative determination by absorbance measurement at 235 nm. The hR_F values of (1) to (4) were found to be 58, 61, 68 and 79, respectively. Linearity was in the range of 200-1200 ng/band. Average recoveries were 98, 99.5, 98 and 99 % for compounds (1) to (4), respectively.

herbal, quality control, quantitative analysis, HPTLC 32e

110 071 N. DUBEY*, A. JAIN, A. RAGHUWANSHI, D. JAIN (*College of Pharmacy, IPS Academy, Indore-452015, Madhya Pradesh, India, nitindubeympharm@yahoo.com): Stability-indicating

HPTLC method for simultaneous estimation of amlodipine besylate, hydrochlorothiazide and olmesartan medoxomil in combined tablet dosage forms. J. Planar Chromatogr. 25, 475-480 (2012). HPTLC of amlodipine besylate (1), hydrochlorothiazide (2) and olmesartan medoxomil (3) on silica gel with chloroform - ethyl acetate - toluene - methanol - glacial acetic acid 39:39:77:39:6. Quantitative determination by absorbance measurement at 230 nm. The hR_F of compounds (1) to (3) were 31, 56 and 81, respectively. Linearity was in the range of 200-4800 ng/band for (1), 100-4000 ng/band for (2) and 200-5200 ng/band for (3). Limits of detection and quantification were 35 and 101 ng/band for (1), 20 and 59 ng/band for (2) and 48 and 144 ng/band for (2), respectively. Intermediate/inter-day/intra-day precision was below 1.0 % (n=6). Mean recovery was between 100.0 and 100.3 % for all active agents.

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

110 072 SH. FAN (Fan Shangtan)*, Y. ZHANG (Zhang Yong), B. CAI (Cai Bin), Q. YOU (You Qiufeng) (*Section of Pharm., Fuzhou General Hosp., Nanjing Military Region, The Chinese PLA, Fujian, Fuzhou 350025, China): (Study on the method for the determination of emodin in Chuanyushaoshang cream) (Chinese). J. China Pharm. Univ. 21 (10), 31-32 (2012). Chuanyushaoshang cream is a herbal TCM preparation for external application for protecting wounds, promoting wound healing, antibiosis, invigorating the circulation of blood, and relieving pain. For quality control, TLC of emodin, one of the key active constituents in the extracts of the preparations, on silica gel with petroleum ether (60-90 ºC) - ethyl acetate 7:3, detection at UV 366 nm.

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

110 073 M. FANG (Fang Mingyu) (Hebei Provincial Tangshan Fengnan Hosp., Dep. Of TCM, Hebei, Fengnan 063300, China): (Study on the identification of Compound Kuhuang lotion by thin-layer chromatography) (Chinese). Hebei J. Trad. Chinese Med. 34 (3), 433-434 (2012). Compound Kuhuang lotion is a TCM preparation used clinically for clearing heat and eliminating dampness, eliminating stagnations of blood, and relieving pains. Description of a method for the quality control of the preparation. TLC of the extracts of the preparations on silica gel 1) for *Rheum officinale* and the standards emodin, rhein, chrysophanol and aloe emodin, with petroleum ether (60-90 ºC) - ethyl acetate - formic acid 15:5:1, detection in daylight; 2) for *Radix Sophorae flavescentis* and the standard matrine, with ethyl acetate - propanone - benzene - ammonia 30:10:30:1, detection by spraying with a solution of potassium iodobismuthate - water - hydrochloric acid 10:200:1, detection in daylight; 3) for Golden Cypress and the standard berberine, with benzene - ethyl acetate - methanol - isoamyl alcohol - water 20:10:5:5:1, detection at UV 366 nm.

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

110 074 M. FILIP, Mihaela VLASSA, Florina COPACIU, Virginia COMAN* (*Babe-Bolyai University, »Raluca Ripan« Institute for Research in Chemistry, 30 Fântânele Street, 400294, Cluj-Napoca, Romania, coman_virginia@yahoo.com): Identification of anthocyanins and anthocyanidins from berry fruits by chromatographic and spectroscopic techniques to establish the juice authenticity from market. J. Planar Chromatogr. 25, 534-541 (2012). TLC of anthocyanins and anthocyanidins in berry fruits on cellulose layers with hydrochloric acid - glacial acetic acid - water 10:1:3. Quantitative determination by absorbance measurement at 520 nm. The hR_F values obtained for pelargonidin and cyanidin were 78 and 58, respectively. The TLC method was complementary to an HPLC method and allowed for identification of the major anthocyanidins characteristic for each berry fruit.

food analysis, quality control, herbal, qualitative identification 32e

110 075 V. GHOSH, S. BHOPE*, V. KUBER, A. SAGULATE (*Department of Analytical Development (R&D), Tulip Lab Pvt. Ltd. F-20=21, MIDC Ranjangaon, Pune 412220, India, bshrinivas16@gmail.com): An improved method for the extraction and quantitation of diosgenin in *Tribulus terrestris* L. J. Liq. Chromatogr. Relat. Technol. 35, 1141-1145 (2012). HPTLC of diosgenin in various parts of *Tribulus terrestris* L. on silica gel with toluene - ethyl acetate - methanol 7:3:1. Detection by dipping in anisaldehyde reagent consisting of anisaldehyde - acetic acid - sulfuric acid - methanol 1:20:10:170, followed by heating at 110 °C for 2 min. Quantitative determination by absorbance measurement at 430 nm. The hR_F value of diosgenin was 48 and selectivity regarding matrix was given. Linearity was between 50 and 240 ng/zone. The method provides acceptable intra-day and inter-day precision for diosgenin. The limits of detection and quantification were 2 and 7 ng/spot, respectively. Recovery (by standard addition) was 100.6 %. The method provided comparable results with HPLC.

herbal, quality control, HPTLC, quantitative analysis, comparison of methods 32e

110 076 P. GHOSH*, A. KATIYAR (*Natural Plant Products Division, CSIR-Institute of Himalayan Bioresource Technology, Post Box No. 6, Palampur-176061, Himachal Pradesh, India, ghoshpatu75@rediffmail.com): Densitometric HPTLC analysis of juglone, quercetin, myricetin, rutin, caffeic acid, and gallic acid in *Juglans regia* L. J. Planar Chromatogr. 25, 420-425 (2012). HPTLC of juglone (1), quercetin (2), myricetin (3), rutin (4), caffeic acid (5), and gallic acid (6) in the stem bark of *Juglans regia* L. on RP-18 with methanol - water - formic acid - acetic acid 61:58:3:3. Quantitative determination by absorbance measurement at 254 nm. The hR_F values of compounds (1) to (6) were 16, 20, 31, 42, 55 and 78, respectively. Linearity was in the range of 13-800 ng/zone for (1) and (5), 100-1000 ng/zone for (2) and (6) and 50-800 ng/zone for (3) and (4). Limits of detection and quantification were 4 and 13 ng/zone for (1) and (5), 30 and 100 ng/zone for (2), 15 and 50 ng/zone for (3), (4) and (5), respectively. The intra-day and inter-day precisions for compounds (1) to (6) were in the range of 0.6-1.3 % and 0.5-1.9 %, respectively. Average recovery was between 98.6 and 101.1 %.

herbal, quality control, quantitative analysis, HPTLC 32e

110 077 B. GHULE*, P. YEOLE (*Institute of Pharmaceutical Education and Research, Borgaon (Meghe), Wardha-442001, Maharashtra State, India, ghulebv@rediffmail.com): Rapid isolation and HPTLC validated method for the quantification of echioidinin-5-O-beta-D-glucopyranoside in *Andrographis echioides*. J. Planar Chromatogr. 25, 575-580 (2012). HPTLC of echioidinin-5-O-beta-D-glucopyranoside in *Andrographis echioides* on silica gel with chloroform - methanol 31:9. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 200-1400 ng/zone. Limits of detection and quantification were found to be 54 and 68 ng/zone, respectively. Recovery (by standard addition) was 96.7 %.

herbal, quality control, quantitative analysis, HPTLC 32e

110 078 B. GHULE*, S. PALVE, L. RATHI, P. YEOLE (*Institute of Pharmaceutical Education and Research, Borgaon (Meghe), Wardha 442001, Maharashtra State, India, ghulebv@rediffmail.com): Validated HPTLC method for simultaneous determination of shanzhiside methyl ester and barlerin in *Barleria prionitis*. J. Planar Chromatogr. 25, 426-432 (2012). HPTLC of shanzhiside methyl ester (1) and barlerin (2) on silica gel with chloroform - methanol 4:1. Quantitative determination by absorbance measurement at 240 nm. The hR_F of compounds (1) and (2) were 30 and 48, respectively. Linearity was in the range of 200-1000 ng/zone. Limits of detection and quantification were found to be 13 and 22 ng/zone for (1) and 18 and 31 ng/zone for (2), respectively. Recovery

was found to be 99.2-99.5 % for (1) and 98.9-99.2 % for (2), respectively. The method showed comparable results to a validated HPLC method.

quality control, herbal, HPTLC, quantitative analysis, comparison of methods 32e

110 079 V. GLAVNIK, B. SIMONOVSKA, A. ALBREHT, Irena VOVK* (*National Institute of Chemistry, Laboratory for Food Chemistry, Hajdrihova 19, SI-1001 Ljubljana, Slovenia, irena.vovk@ki.si): TLC and HPLC screening of p-coumaric acid, trans-resveratrol, and pterostilbene in bacterial cultures, food supplements, and wine. J. Planar Chromatogr. 25, 251-258 (2012). TLC of trans-resveratrol (1), pterostilbene (2), and *p*-coumaric acid (3) in samples of recombinant bacterial cultures, food supplements, and wine on silica gel with *n*-hexane - ethyl acetate - formic acid 20:19:1. Quantitative determination by absorbance measurement at 286 nm (3) and 303 nm for (1) and (2).

quality control, herbal, HPTLC, qualitative identification 32e

110 080 J. GUO (Guo Jiangong)*, X. HAO (Hao Xufeng), CH. GAO (Gao Chaoxu), Y. ZHOU (Zhou Yiqing) (*Zhengzhou Yimi Pharm. Com., Ltd., Henan, Zhengzhou 452392, China): (Study on the method for the quality control of Dangshi tablets) (Chinese). J. of Chinese Med. 26 (160), 1088-1090 (2011). Dangshi tablets are a herbal TCM preparation for cleaning heat, diuresis, and dissolving stones, and are prescribed to cure urinary tract infections and acute nephritis. For quality control, TLC of the extracts of the medicine on silica gel 1) for *Poria cocos*, with petroleum ether (30-60 ºC) - acetone - ethyl acetate 85:15:1, detection at UV 366 nm; 2) for Licorice, with chloroform - methanol - water 40:10:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 ° until the zones are visible, evaluation in daylight.

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

110 081 M. GUO (Guo Min)*, M. XU (Xu Mei) (*Chongqing Municip. Hosp. at Jiangbei District, Chongqing 400020, China): (Study on the method for the identification of Shujian capsules by thin-layer chromatography) (Chinese) J. China Pharm. Univ. 20 (9), 19-20 (2011) Shujian capsules are a herbal TCM preparation prescribed clinically to treat shoulder arthritis. For quality control, TLC on silical gel for 1) for Cassia twig, with petroleum ether (60-90 ºC) - ethyl acetate 17:3, detection by spraying with 2 % 2,4-dinitrophenylhydrazine in ethanol and viewing in daylight; 2) for *Radix Sileris*, with chloroform - methanol 4:1, detection at UV 254 nm; 3) for *Angelica sinensis* and *Ligusticum wallichii*, with toluene - ethyl acetate - formic acid 40:10:1, detection by spraying with 1 % ferric chloride - 1 % potassium ferricyanide 1:1 and viewing in daylight; 4) for the root of Kudzu vine, with chloroform - methanol - water 28:10:1, detection under UV 366 nm.

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

110 082 L. HAO (Hao Lihua), L. DONG (Dong Lingling), X. YU (Yu Xiaohui), R. WAN (Wan Renling)* (*National Inst. for Veterinary Drug Contr., Beijing 100081, China): (Rapid screening of the additives illegally mixed in astragalus polysaccharides injection by thin-layer chromatography) (Chinese). Chinese J. of Veterinary Drugs 46 (5), 29-31 (2012). TCM is more and more important in veterinary medicine. It has been found that some medicines were illegally mixed with certain chemicals harmful to the animal body. Rapid screening of the additives illegally mixed in *Astragalus polysaccharides* injection by TLC on silica gel with 1) chloroform - methanol - ace-

tone - ammonia 9:2:1:0.05 and 2) ethyl acetate - ethanol 1:1. Detection 1) under UV 254 nm; 2) by spraying with 5 % potassium iodobismuthate solution and evaluation in daylight; 3) by exposure to iodine vapor for a few minutes and evaluation under UV 254 nm. Identification of lincomycin, enrofloxacin, novalgin, 4-acetamino phenol, moroxydine hydrochloride, and ribavirin by comparison of the fingerprint with individual standards, with detection limits of 2.0, 0.5, 1.0, 0.5, 1.0, and 4.0 mg/mL, respectively. The method was applied to 25 batches of market samples and the results were compatible with those obtained by HPLC.

quality control, pharmaceutical research, traditional medicine, herbal, agricultural, qualitative identification, comparison of methods 32e

110 083 K. HARIBABU, M. AJITHA, B. RAMESH, K. SURESH*, J. RAO (*Natural Product Chemistry Division, Indian Institute of Chemical Technology, Hyderabad-500 007, India, suresh@iict.res.in): Quantification of bergenin from *Mallotus philippinensis* by HPTLC-MS and study on different extraction methods. J. Planar Chromatogr. 25, 445-449 (2012). HPTLC of bergenin in the stem-bark and roots of *Mallotus philippinensis* on silica gel with ethyl acetate - methanol - acetic acid - formic acid 16:2:1:1. Quantitative determination by absorbance measurement at 284 nm and by TLC-MS using the TLC-MS Interface. The hR_F of bergenin was 49. Linearity was in the range of 100-600 ng/zone. Limits of detection and quantification were 29, 37 and 98 ng/zone, respectively. Intermediate intra-day/inter-day precision was 1.1 % (n=6). Recovery was between 96.6 and 98.9 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 084 H. HU (Hu Hongyan) (Xiaogan Municipal Central Hosp., Hubei, Xiaogan 432000, China): (Study on the method for the quality control of Biqiang Bidou flushing fluid) (Chinese). J. of Chinese Med. 27 (166), 344-345 (2012). Biqiang Bidou flushing fluid is a herbal TCM preparation for treatment of forehead headaches and sinusitis. For quality control, TLC on silica gel 1) for Honeysuckle and the standard chlorogenic acid, with ethyl acetate - formic acid - water 15:12:5, detection under UV 366 nm; 2) for Baical Skullcap, with ethyl acetate - acetone - acetic acid - water 10:4:5:3, detection by spaying with 5 % ferric chloride in ethanol and viewing in daylight.

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

110 085 J. HUANG (Huang Jian) (Pharm. Preparation section, Wenzhou Municip. Trad. Chinese Med. Hosp., Subsidiary Hosp. of Zhejiang Univ. of Trad. Chinese Med. & Pharm., Zhejiang, Wenzhou 325000, China): (Identification of *Atractylodes macrocephala* Koidz in Yishen Antai ointment by thin-layer chromatography) (Chinese). J. Strait Pharm. 24 (2), 65-66 (2012). Yishen Antai ointment is a herbal TCM recipe specialized for tonifying the kidney and miscarriage prevention of pregnant woman with marked clinical curative effect. Description of a procedure for the analysis of *Atractylodes macrocephala* Koidz, one of the key components of the preparation. TLC on silica gel with petroleum ether (60-90 ºC) - ethyl acetate 50:1, detection by spraying with 5 % vanillin in a solution of ethanol - sulfuric acid 100:1 and heating at 105 °C until the zones were visualized.

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

110 086 L. JIANG (Jiang Lei), X. MENG (Meng Xiangsong), J. LI (Li Jun), L. YI (Yi Lei), X. MA (Ma Xinyu)* (*Bozhou Inst. for Food & Drug Control, Anhui, Bozhou 236800, China): (Study on the method for the quality control of Pudilan Xiaojan oral liquid) (Chinese). J. of Qilu Med. & Pharm.

31 (2), 75-78 (2012). Pudilan Xiaojan oral liquid is a herbal TCM preparation effective in clearing heat and removing toxicity, because of its antiinflammatory activity it is used to cure swelling caused by furuncles, pharyngitis, lymphadenitis and tonsillitis. Dandelion is the key component drug of the preparation. Analysis of the components of dandelion in the preparation by TLC on silica gel with chloroform - methanol - formic acid 18:2:1, detection under UV 366 nm.

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

110 087 L. JIANG (Jiang Li), L. SUN (Sun Li), J. CHANG (Chang Junmin)* (*Xinjiang Med. Univ., Coll. of Pharm., Teach. & Res. Section, Urumqi 830039, China): (Study on the method for the quality control of the effective parts of garlic by thin-layer chromatography) (Chinese). Chinese J. of Medication & Clinics 12 (6), 768-769 (2012). Alliin (*s*-allyl-L-cysteine sulfoxide) is a sulfur containing amino acid found in garlic, which has antibacterial and antioxidant activities and is widely used as a component in Chinese drug formulations. For quality control, TLC on silica gel with *n*-butanol - glacial acetic acid - water 4:1:1, detection by spraying with 0.2 % ninhydrin and heating at 105 ºC for 5 min and viewing in daylight.

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

110 088 C. JOSHI, J. SAVAI, A. VARGHESE, N. PANDITA* (*Department of Applied Pharmaceutical Sciences, Shobhaben Pratapbhai Patel, School of Pharmacy and Technology Management, Mumbai, India, nancy.pandita@nmims.edu): Development and validation of HPTLC method for simultaneous determination of quercetin and kaempferol in leaves of two chemotypes of *Centella asiatica*. J. Planar Chromatogr. 25, 433-438 (2012). HPTLC of quercetin (1) and kaempferol (2) in the leaves of Centella asiatica on silica gel with toluene - ethyl acetate - chloroform - formic acid 6:4:4:1. Quantitative determination by absorbance measurement at 240 nm. The hR_F of compounds (1) and (2) were 35 and 48, respectively. Linearity was in the range of 100-1000 ng/band. Limits of detection and quantification were 54 and 165 ng/band for (1) and 68 and 207 ng/band for (2), respectively. Intra-day relative standard deviation of (1) was between 5.3 and 6.5 % and of (2) between 5.1 and 11.4 %. Inter-day relative standard deviation of (1) was 2.1-6.6 % and of (2) 2.6-5.8 % (n=6). Recovery was found to be 98.1 % for (1) and 90.1 % for (2).

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

110 089 S. KATHIRVEL, S. SATYANARAYANA, G. DEVALARAO* (*Department of Pharmaceutical Analysis, K.V.S.R Siddhartha College of Pharmaceutical Sciences, Vijayawada, A.P., India, devalarao2007@gmail.com): Densitometric evaluation of stability-indicating HPTLC method for the analysis of darifenacin hydrobromide in bulk and in tablet dosage form. J. Liq. Chromatogr. Relat. Technol. 35, 280-293 (2012). HPTLC of darifenacin hydrobromide in bulk and in tablet dosage form on silica gel with toluene - acetone - methanol 3:1:1. Quantitative determination by absorbance measurement at 286 nm. The hR_F value of darifenacin hydrobromide was 34 and selectivity regarding matrix was given. Linearity was between 50 and 450 ng/zone. The intermediate/inter-day/intra-day precision was below 1.4 % (n=6). The limits of detection and quantification were 30 and 91 ng/spot, respectively. Recovery (by standard addition) was between 98.8 and 100.8 %.

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

110 090 P. KAUR, A. CHAUDHARY, A. KATIYAR, B. SINGH*, R. SINGH (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology, (CSIR) Palampur, Himachal Pradesh, 176061, India, bikram_npp@rediffmail.com): Rapid validated RP-HPTLC method for the quan-

tification of major bioactive constituents of *Crataegus oxyacantha* L. J. Planar Chromatogr. 25, 415-419 (2012). HPTLC of apigenin (1), quercetin (2), hyperoside (3), vitexin (4) and vitexin-2«-O-rhamnoside (5) on silica gel with acetonitrile - methanol - water 1:1:2 + 1 drop formic acid. Quantitative determination by absorbance measurement at 254 nm. The hR_F of (1) to (5) were 12, 20, 48, 53 and 59, respectively. Linearity was in the range of 400-1250 ng/zone for (1) and (2) and 800-2500 ng/zone for (3) to (5). Limits of detection and quantification were 100 and 310 ng/zone for (1), 200 and 630 ng/zone for (2) and (3) and 300 and 960 ng/zone for (4) and (5), respectively, The intermediate/inter-day/intra-day precision was below 2.2 % (n=3). Recovery for all (1) to (5) was between 97.1 and 100.2 %.

110 091 S. LAKAVATH, B. AVULA, Y. WANG, C. RUMALL, S. GANDHE, A. BELVOTAGI, P. ACHANTA, R. KUMAR, I. KHAN, A. NARASIMHA* (*Kakatiya University, University College of Pharmaceutical Sciences, Department of Pharmaceutical Chemistry, Warangal-506009, Andhra Pradesh, India, avnapparao@yahoo.com): Differentiating the gum resins of two closely related indian Gardenia species, *G. gummifera* and *G. lucida*, and establishing the source of dikamali gum resin using high-performance thin-layer chromatography and ultra-performance liquid chromatography-UV/MS. J. AOAC Int. 95, 67-73 (2012). HPTLC fingerprint of four cycloartanes dikamaliartane-A (1) and dikamaliartane-B (2) and gummiferartane-1 (3) and gummiferartane-2 (4) in the gum resin from the leaf buds of *Gardenia lucida* or *G. gummifera* on silica gel with toluene - acetone 11:9. Detection by dipping in anisaldehyde reagent (anisaldehyde 0.5 % in methanol - acetic acid - sulfuric acid 17:2:1), followed by heating at 100 °C for 5 min. The hR_F of compounds (1) to (4) were 60, 80, 90 and 70, respectively.

quality control, herbal, HPTLC, qualitative identification 32e

110 092 C. LI (Li Cai Dong)*, Y. LIANG (Liang Yun), W. ZHANG (Zhang Wei), B. WU (Wu Bin) (*Inst. of Hepatology, Lanzhou Municip. People's Hosp. No. 2, Gansu, Lamzhou 730046,China): (Identification of *Phyllanthus niruri* Linn. and quantitative determination of gallic acid in Zhuqi Fugan granules) (Chinese). Chinese J. of Inform. on TCM 19 (4), 47-48 (2012). Zhuqi Fugan granules, a herbal TCM preparation, are effective for clearing heat and removing toxicity, nourishing the liver, and strengthening the body resistance and are clinically prescribed to cure viral hepatitis type B. For quality control, TLC of the extracts of the preparations on silica gel with cyclohexane - chloroform - ethyl acetate 4:1:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 ° until the zones are visible.

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

110 093 H. LI (Li Haiyan)*, J. KANG (Kang Jian) (*Henan Provinc. Inst. for Food & Drug Control, Henan, Zhengzhou 450003, China): (Study of the quality standard for Guanxin Shengmai oral liquid) (Chinese). J. China Pharm. Univ. 20 (1), 18-20 (2011). Guanxin Shengmai oral liquid is a herbal TCM preparation for promoting the secretion of saliva or body fluid and invigorating the circulation of blood. TLC of the extracts of the preparations 1) for *Radix Paeoniae Rubra*, on silica gel plates developed with chloroform - acetone 4:1, detection by spraying with 10 % sulfuric acid in ethanol and heating mildly, viewing under daylight; 2) for *Ophitopogin japonicum* (L.f) Ker-Gawl, on silica gel with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in ethanol - sulfuric acid 4:1 and heating mildly, viewing under daylight; 3) for Ginseng and *Panax notoginseng* (Burk.) F.H.Chen, on silica gel with the lower phase of chloroform - methanol - water 13:7:2 at 2-10 °C, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C, viewing under daylight; 4) for *Schisandra*

chinensis Turcz. Baill, on silica gel with petroleum ether (30-60 ºC) - formyl acetate - formic acid 10:5:1, detection by viewing under UV 254 nm.

pharmaceutical research, qualitative identification 32e

110 094 J. LI (Li Jie)*, W. LUO (Luo Wenhui), J. YIN (Yin Jianhua), ZH. TAN (Tan Zhican), S. LI (Li Sumei)* (*Guandong Provinc. Hosp. of Trad. Chinese Med., Guangdong, Guanzhou 510095, China): (Study on the thin-layer chromatographic fingerprint profiles of flavonoids in *Microcos paniculata* Linn.) (Chinese). Jiangxi J. of Trad. Chinese Med. 43 (351), 67-68 (2012). Flavonoids are the main active component in dried leaves of Microcos paniculata Linn. This traditional Chinese herbal crude drug lowers the blood pressure and blood fat, prevents from cardiovascular diseases and anti-aging effects. In order to develop a quality control method for *Microcos paniculata* Linn. the reference substances vitexin, isovitexin and narcissoside were analyzed with 16 samples of *Microcos paniculata* Linn. available from different places of origin. TLC of the extracts of the drug samples and the reference substances on silica gel with ethyl acetate - methanol - water 100:17:13, detection by spraying with 10 % sulfuric acid in ethanol, followed by heating at 105 °C for 5 min and viewing in UV 366 nm. Densitometric analysis at 366 nm with a mercury lamp in reflection mode, a scanning speed of 20 mm/s and a resolution of 25 µm/step. Identification of the flavonoids in the samples to be tested by fingerprint comparison of both the fluorescence chromatograms and densitograms.

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

110 095 S. LI (Li Suqin), R. MA (Ma Renqiang), R. ZHANG (Zhang Ronghua)*, X. ZHU (Zhu Xiaofeng), L. YANG (Yang Li), M. PAN (Pan Meiying), ZH. SONG (Song Zhijuan) (*Sec. of Teach. & Res. of Trad. Chinese Med., Coll. of Pharm., Jinan Univ., Guangzhou 510632, China): (Study on the method of the quality control of compound Shuyu granules) (Chinese). J. of Jinan Univ. (Natural Sci.) 33 (3), 289-293 (2012). Compound Shuyu granule is a herbal TCM preparation prescribed clinically to treat insomnia, neurosis and melancholia. TLC of the extracts of the medicine on silica gel 1) for *Caulis Polygoni Multiflori* and the standard emodin, with the upper phase of petroleum ether (60-90 ºC) - formyl acetate - formic acid 15:5:1, detection under UV 366 nm and by exposiure to iodine vapors and viewing under UV 366 nm; 2) for *Salvia miltiorrhiza* and the standard salvianolic acid B, with cyclohexane - dichloromethane - acetone - formic acid - glacial acetic acid 2:4:2:2:1, detection by spraying with 5 % ferric chloride in ethanol and heating at 105 ° until the zones are visible; 3) for white Paeony root, with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 1:200 and heating mildly until the zones are seen.

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

110 096 W. LI (Li Wanjiang), X. XIA (Xia Xinzhong)* (*Jingzhou Municip. Inst. for Cont. & Test of Drugs, Hubei, Jingzhou 434000, China): (Identification of the components of *Angelica sinensis* in the traditional Chinese medicines for gynaecological diseases by thin-layer chromatography) (Chinese). J. of Yangtze Univ. (Nat. Sci., Med.) 9 (1), 64-65 (2012). *Angelica sinensis* is a herbal TCM drug widely used in the preparations for gynaecological diseases, effective in promoting blood circulation, ease of pain and against inflammations. TLC of the extracts of the preparation on silica gel with *n*-hexane - ethyl acetate 9:1. Detection under UV 366 nm.

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

110 097 Y. LI (Li Yan)*, H. HU (Hu Hongyan), Q. HUANG (Huang Qiuming) (*Xiaogan Minicip. Centre Hosp., Hubei, Xiaogan 432000, China): (Identification of three component herbal drugs in Baizhuhugan compound oral liquid by thin- layer chromatography) (Chinese). J. of Chinese Med. 27 (166), 343-344 (2012). Baizhuhugan compound oral liquid is effective for enriching the blood, tonifying spleen, soothing the liver, and is clinically prescribed to cure chronic hepatitis and cirrhosis. For quality control, identification of the three contained herbal drugs by TLC of the extracts of the preparation 1) for Chinese Angelica, on silica gel with *n*-hexane - ethyl acetate 9:1, detection at UV 366 nm; 2) for Bighead Atractylodes rhizome, on silica gel with cyclohexane - ethyl acetate 7:3, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C, viewing in daylight; 3) for Chinese herbaceous Peony, on silica gel with chloroform - ethyl acetate - methanol - ammonia 8:1:4:1, detection by spraying with 5 % vanillin in ethanol - sulfuric acid 4:1 and heating mildly, viewing in daylight.

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

110 098 Y. LIANG (Liang Yuanyuan)*, Y. HUANG (Huang Yuping) (*New Drug R & D Center, Guangzhou Univ. of Trad. Chinese Med. & Pharm., Guangzhou 510006, China): (Study on the method for the quality control of E Ling capsules by thin-layer chromatography) (Chinese). J. China Pharm. Univ. 26 (5), 503-504 (2012). E Ling capsule is a herbal TCM for activating blood circulation to dissipate blood stasis, relieving pain, eliminating stagnation, and is prescribed clinically to treat pelvic endometriosis. For quality control, TLC of the extracts of the medicine on silica gel 1) for the root of red-rooted salvia, with toluene - ethyl acetate 19:1, detection by viewing in daylight; 2) for *Fructus Aurantii*, first over 4 cm with ethyl acetate - methanol - water 100:17:10, then over 8 cm with toluene - ethyl acetate - formic acid - water 20:10:1:1, detection at UV 366 nm after spraying with 1 % aluminium chloride in ethanol; 3) for *Rhizoma Corydalis*, with *n*-hexane - chloroform - methanol 15:8:2, detection at UV 366 nm.

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

110 099 R. LIMGAVKAR*, P. TRIVEDI, A. PATEL (*Institute of Pharmaceutical Education and Research, Sector-23, Gh-6, Gandhinagar 382023, Gujarat, India, ratnalimgavkar@gmail.com): Development and validation of reverse phase high-performance liquid chromatographic and high-performance thin-layer chromatographic methods for simultaneous estimation of melitracen hydrochloride and flupentixol hydrochloride in bulk and combined dosage form. J. Liq. Chromatogr. Relat. Technol. 35, 2753-2764 (2012). HPTLC of melitracen hydrochloride (1) and flupentixol hydrochloride (2) on silica gel with methanol - chloroform - toluene 2:9:9 + 1 drop ammonia. Quantitative determination by absorbance measurement at 291 nm. The hR_F value of compounds (1) and (2) were 53 and 34 and selectivity regarding matrix was given. Linearity was in the range of 1600-6400 ng/band and 80-320 ng/band for (1) and (2), respectively. Limit of detection was found to be 27 ng/band and 5 ng/band for (1) and (2), respectively. Limit of quantification was found to be 82 ng/band and 14 ng/band for (1) and (2), respectively. The intermediate/inter-day/intra-day precision was below 0.2 % for (1) and 1.1 % for (2) (n=3). Recovery (by standard addition) was between 99.1 and 101.8 % for both (1) and (2). The method showed comparable results with HPLC.

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

110 100 M. LIU (Liu Min), ZH. XIE (Xie Zhigang), H. YIN (Yin Hongping), M. WANG (Wang Min)* (*Coll. of life sci. & technol., China Pharm. Univ., Jiangsu, Nanjing 210009, China): (Study on

the method for the determination of glucosamine sulfate potassium chloride by thin-layer chromatography (Chinese). J. Strait Pharm. 24 (5), 36-38 (2012). Glucosamine sulfate is a kind of glycosaminoglycan, which, as a medicine, is able to stimulate cartilage cells, to supplement the cartilage matrix and to inhibit matrix metalloproteinase expression, thus to promote the repair of cartilage. For better quality control of the medicine a method is presented for the analysis of the related substances in glucosamine sulfate potassium chloride. TLC on silica gel with dichloromethane - methanol - ammonia 2:2:1, detection by exposure to iodine vapors for 30 min and evaluation in daylight. The LOD was 25 µg/mL. Investigation of the stability of the medicines by analysis of a sample submitted to typical stress conditions such as acid, alkali, water bath, hydrogen peroxide, high temperature and UV light. Degradation occurred only for the samples treated with alkali and in the water bath.

quality control, pharmaceutical research, qualitative identification 32c

110 101 X. LIU (Liu Xiaochuan)*, X. LI (Li Xixiang), ZH. JIAO (Jiao Zhenghua), Q. GU (Gu Qiuli), L. WANG (Wang Lanxia) (*Gansu Provinc. Hosp. of TCM, Gansu, Lanzhou 730050, China): (Study of the method for the quality control of Fuyanshuan suppository) (Chinese). Chinese J. of Inform. on TCM 17 (12), 48-50 (2010). Fuyanshuan suppository is a herbal TCM preparation for heat clearing, detoxifying, eliminating stagnation, regulating the flow of vital energy and invigorating the circulation of blood. For quality control TLC of the extracts of the preparation 1) for *Ajuga becumbens* Thunb., on silica gel with cyclohexane - chloroform - ethyl acetate - glacial acetic acid 40:10:16:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 100 °C, evaluation under UV 366 nm or daylight; 2) for *Lonicera japonica*, on silica gel with chloroform - acetone - formic acid 3:2:1, detection by exposure to daylight for 10 min and viewing under UV 366 nm.

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

110 102 Y. LIU (Liu Yuan)*, W. WEI (Wei Wei), ZH. SONG (Song Zhizhao) (*Guangxi Inst. of Trad. Chinese Med. & Pharm., Guangxi, Nanning 530022, China): (Study on the identification of *Cissus hexangularis* Thorel ex Planch by thin-layer chromatography) (Chinese) Chinese J. Ethnomed. Ethnopharm. (1), 53-54 (2012). *Cissus hexangularis* Thorel ex Planch is a TCM herb. Its dried rattan is effective in relieving rheumatic pains and invigorating the circulation of blood, and is often used in drug preparations against rheumatism, pain or numbness, lumbar muscle degeneration and traumatic injury. TLC of the extracts and standards resveratrol and bergeninum on silica gel with chloroform - ethyl acetate - formic acid 30:20:1, detection by spraying with a solution of vanillin - ethanol - sulfuric acid 10:200:1.

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

110 105 J. LU* (Lu Jianwei), Y. TANG (Tang Yongchen), H. CHEN (Chen Huihong), G. PAN (Pan Gehui) (*Guanxi Liuzhou Municip. Trad. Chinese Med. Hosp., Guangxi, Liuzhou 545000, China): (Identification of Ruoshiming oral liquid by thin-layer chromatography) (Chinese). Modern J. of Integrated Trad. Chinese & Western Med. 21 (16), 1786-1788 (2012). Ruoshiming oral liquid is a herbal TCM preparation for the treatment of children‘s optic nerve hypoplasia, myopia, hyperopia, and astigmatism. For identification, TLC of the extracts of the preparations on silica gel 1) for *Radix Rehmanniae Preparata*, with benzene - diethyl ether 5:2, detection by spraying with 2 % ferric chloride - 2 % potassium ferricyanide 1:1 and viewing in daylight; 2) for *Pericarpium Citri Reticulatae*, first with ethyl acetate - methanol - water 100:17:13 and then with the upper phase of toluene - ethyl acetate - formic acid - water 20:10:1:1, detection by spraying with 3 % alu-

minium chloride in ethanol and viewing at UV 366 nm; 3) for *Rhizoma Acori Tatarinowii*, with chloroform - diethyl ether 8:1, detection by viewing in daylight; 4) for Polygonatum sibiricum, with benzene - ethyl acetate - formic acid 50:40:1, detection by spraying with potassium iodobismuthate - HCl - water 10:1:200 and viewing in daylight; 5) for *Fructus Lycii*, developed with benzene - diethyl ether 2:1, detection by viewing in UV 366 nm.

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

110 106 Y. MA (Ma Yanni)*, L. LIU (Liu Li), J. WEI Wei Junlian), X. LU (Lu Xingwen), H. ZHOU (Zhou Hanhua) (*Guiyang Coll of Trad. Chinese Med., Gyizhou, Guiyang 550002, China): (Identification of Blood ginseng by thin-layer chromatography) (Chinese). Chinese J. Ethnomed. Ethnopharm. (23), 70-71 (2011). Blood ginseng, the dried root of *Indigofera stachyoides* Lindl. is a traditional Chinese herbal crude drug with special effect in invigorating the circulation of blood, reducing phlegm, treating yin deficiency by reinforcing body fluid and nourishing the blood. It is used clinically to treat encephalalgia, woman‘s abdominal pain, and metrorrhagia. For quality control 1) TLC of the extracts on silica gel with toluene - ethyl acetate - formic acid 6:3:1, detection in daylight; 2) determination of the moisture by drying in the oven; 3) determination of general ash content by firing, and of the acid insoluble ash content by acid pickling; 4) determination of ethanol extract by lixiviating with 50 % ethanol.

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

110 107 C. MACWANA*, A. PATEL, V. PARMAR, S. PATEL (*Institute of Pharmaceutical Education and Research, Gh-6, Sec 23, Gandhinagar, India, chhayamacwana111@gmail.com): Simultaneous HPTLC analysis of atorvastatin calcium, ezetimibe, and fenofibrate in tablet. J. Liq. Chromatogr. Relat. Technol. 35, 524-532 (2012). HPTLC of atorvastatin calcium (1), ezetimibe (2), and fenofibrate (3) in tablet on silica gel with toluene - chloroform - methanol 23:15:7 + 1 drop glacial acetic acid. Quantitative determination by absorbance measurement at 253 nm. The hR_F values of active agents (1) - (3) were 20, 33 and 80, respectively. Linearity was 200-800 ng/zone for (1) and (2) and 4-16 µg/zone for (3). The intermediate/inter-day/intra-day precision was below 1.1 % for (1), 1.3 % for (2) and 1.5 % for (3) (n=6). The limits of detection and quantification were 19 and 59 ng/zone for (1), 23 and 68 ng/zone for (2), and 1449 and 4390 ng/zone for (3), respectively. Recovery (by standard addition) was between 99.1 and 99.8 % for compounds (1) to (3).

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

110 108 X. MAN (Man Xixia)*, H. DING (Ding Hongqiang), H. LIN (Lin Hengkuan), R. YUAN (Yuan Ruili) (*Henan Furen Pharm. Co. Ltd., Henan, Zhengzhou 450003, China): (Study on the method for the quality control of Yanreqing capsules) (Chinese). J. China Pharm. Univ. 19 (22), 41-43 (2010). Yanreqing capsules are a herbal TCM preparation specially effective for heat clearing and detoxifying, and prescribed clinically to cure bronchitis, pneumonia, acute tonsillitis, urinary system infections and biliary tract infections. TLC of the extracts of the medicine on silica gel 1) for Baical Skullcap, with ethyl acetate - butanone - formic acid - water 5:3:1:1, detection by spraying with 2 % ferric chloride in ethanol and viewing in daylight; 2) for Cape jasmine, with ethyl acetate - acetone - formic acid - water 5:5:1:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 110 °C until the zones are visible.

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

110 109 L. MENG (Meng Lin)*, J. SUN (Sun Junhui) (*Shandong Inst. of Commerce & Vocational Technol., Shangdong, Jinan 250103, China): (Determination of picroside-l in *Picrorhiza scrophulariiflora* Pennell by thin-layer chromatography) (Chinese). J. of Shandong Inst. of Commerce & Vocational Technol. 12 (1), 93-94 (2012). Description of a procedure for determination of picroside-l in *Picrorhiza scrophulariiflora* Pennell by TLC of its root extracts on silica gel with chloroform - methanol - ethyl acetate 7:3:5. Detection at UV 254 nm. Quantification of picroside-l by densitometry in absorbance mode at 282 nm. Validation of the procedure by investigation of the linearity range (0.6-3.0 µg/zone, R = 0.999, n = 5); of the stability (*%RSD* = 1.5, n = 5 in 90 min); of the precision (*%RSD* = 0.6, n = 5 within plate); and of the standard addition recovery (98.4 %, *%RSD* = 1.9, n = 5).

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

110 110 S. MENNICKENT*, J. CONTRERAS, B. SCHULZ, Marta DE DIEGO (*Departamento de Farmacia, Facultad de Farmacia, Universidad de Concepción, P.O. Box 237, Concepción, Chile, smennick@udec.cl): High-performance thin-layer chromatographic determination of nifedipine in human serum after liquid-liquid extraction. Quim. Nova. 35, 411-415 (2012). HPTLC of nifedipine in human serum on silica gel with chloroform - ethyl acetate - cyclohexane 19:2:2. Quantitative determination by absorbance measurement at 238 nm. The hR_F of nifedipine was 31. Linearity was in the range of 0.02-0.25 ng/µL. Limits of detection and quantification were 0.7 and 0.9 ng/band, respectively. Intraday precision was in the range of 0.6-3.6 % (n=3) and inter-day precision was in the range of 1.2-3.6 % (n=9). Recovery was between 93 and 102 %.

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

110 111 S. MENNICKENT*, M. VEGA, Marta DE DIEGO, R. FIERRO (*Department of Pharmacy, Faculty of Pharmacy, University of Concepción, P.O. Box 237, Concepción, Chile, smennick@udec.cl): Quantitative determination of propranolol in human serum by high-performance thin-layer chromatography. J. Planar Chromatogr. 25, 54-59 (2012). HPTLC of propanolol in human serum on silica gel with chloroform-methanol 9:1 + 1 drop ammonia. Quantitative determination by absorbance measurement at 290 nm. Linearity was in the range of 5-100 ng/band. Intra-assay and inter-assay precision was in the range of 1.8-2.9 % (n = 3) and 3.5-3.9 % (n = 9), respectively. Limits of detection and quantification were 3.2 and 4.0 ng/band, respectively. Recovery was in the range of 96.4-98.8 % with an *%RSD* below 4.2 %.

clinical routine analysis, HPTLC, quantitative analysis 32c

110 112 P. MIGAS*, Magdalena HEYKA, Loretta POBLOCKA, M. KRAUZE (*Department of Pharmacognosy with Medicinal Plant Garden, Medical University of Gdañsk, Gen. J. Haller Al. 107, 80-416 Gdañsk, Poland, pmig@gumed.edu.pl): BMD-TLC - the useful technique for quantitative analysis of chelidonine, chelerithrine and berberine in herbal drugs. J. Planar Chromatogr. 25, 439-444 (2012). Bivariant multiple development (BMD)-TLC of chelidonine (1), chelerithrine (2) and berberine (3) in the herb and roots of *Chelidonium majus* on silica gel with toluene - ethyl acetate - methanol 1:1:1 for step I, ethyl acetate - formic acid - water 7:1:1 for step II and n-propanol - formic acid - water 21:2:2 for step III. Quantitative determination by absorbance measurement at 290 nm. Linearity was in the range of 200-1500 ng/zone for (1), 200-1000 ng/zone for (2) and 250-1500 ng/zone for (3). Limits of detection and quantification were 100 and 200 ng/zone for (1), 50 and 200 ng/zone for (2) and 50 and 250 ng/zone for (3), respectively. The intermediate/inter-day/intra-day precision was below 1.6 % (n=7). Recovery for (1) to (3) was in the range of 96-105 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 113 CH. MIN (Min Chunyang), L. FU (Fu Lingyan), Q. WANG (Wang Qi), J. LU (Lu Jing)* (*National Inst. for Food & Drug Contr., Beijing 100050, China): (Study of the method for differentiation of the dyes adulterated in Safflower) (Chinese). J. China Pharm. Univ. 25 (8), 772-775 (2011). Safflower, the dried flower of *Carthamus tinctorius* L. is a herbal TCM drug for invigorating the circulation of blood, stimulating the menstrual flow, dissipating blood stasis, relieving pain, and is prescribed clinically to cure amenorrhea, falling injuries and skin and external diseases. Due to the lack of the source some counterfeits have been found on the market in recent years. The methods were studied for differentiating the dyes used by the market for adulteration of safflower. For dyes, TLC of the extracts of the crude drugs on silica gel firstly with chloroform - methanol - glacial acetic acid 7:1:2, detection under daylight for identification of orange, then with ethyl acetate - *n*-butanol - ethanol - ammonia - water 1:3:3:1:1, detection under daylight for identification of acid red 73, lemon yellow and carminum respectively. Results obtained by HPLC were compatible with those obtained by TLC.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, comparison of methods 32e

110 114 M. MIRZA, S. TALEGAONKAR, Z. IQBAL* (*Department of Pharmaceutics, Jamia Hamdard, M B Road, New Delhi, India-110062, ziqbaljh@yahoo.co.in): Quantitative analysis of itraconazole in bulk, marketed, and nano formulation by validated, stability indicating high performance thin layer chromatography. J. Liq. Chromatogr. Relat. Technol. 35, 1459-1480 (2012). HPTLC of itraconazole on silica gel with toluene - ethyl acetate 1:5 + 1 drop ammonia. Quantitative determination by absorbance measurement at 266 nm. The hR_F value of itraconazole was 77 and selectivity regarding matrix was given. Linearity was in the range of 50-2000 ng/zone. The intra-day and inter-day accuracy were in the range of 81.9-97.8 and 89.2-97.1 %, respectively. The limits of detection and quantification were 14 and 43 ng/zone, respectively. Recovery (by standard addition) was between 99.6 and 100.2 %.

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

110 115 Kshipra MISRA, R. TULSAWANI, R. SHYAM, D. MEENA, Gertrud MORLOCK* (*Institute of Food Chemistry, University of Hohenheim, Garbenstrasse 28, 70599, Stuttgart, and Justus Liebig University of Giessen, Institute of Nutritional Science, IFZ, Heinrich-Buff-Ring 26, 35392 Gieben, Germany, gerda.morlock@uni-hohenheim.de): Hyphenated high-performance thin-layer chromatography for profiling of some indian natural efficiency enhancers. J. Liq. Chromatogr. Relat. Technol. 35, 1429-1443 (2012). Comprehensive HPTLC profiling of sugars, sugar alcohols, amino acids, flavonoids, and phenolic acids as well as further bioactive and antioxidative compounds in the Indian natural efficiency enhancer herbs *Hippophae rhamnoides, Valeriana wallichii, Triticum aestivum*, and fungus *Cordyceps sinensis*. HPTLC on silica gel with different mobile phases: for sugars, *n*-butanol-- isopropanol - acetic acid - boric acid solution 6:14:1:3, for amino acids, *n*-butanol - ammonia (25%) - pyridine - water 39:10:34:26, and for sugar alcohols, ethanol - ethyl acetate - water 7:2:1. Quantitative determination of sugars by absorbance measurement at 370 nm after derivatization with aniline diphenylamine *o*-phosphoric acid reagent (mixture of 70 mL aniline solution and 70 mL diphenylamine solution, 2 % in acetone each, and 10 mL *o*-phosphoric acid, 85%). Unknown marker compounds were characterized by HPTLC-attenuated total reflection fourier transform infrared spectroscopy (HPTLC-ATR FTIR) and HPTLC- electrospray ionization mass spectrometry (HPTLC-ESI-MS).

herbal, quality control, HPTLC, quantitative analysis 32e

110 116 N. MNCWANGI, W. CHEN (Wei Yang Chen), I. VERMAAK, A. VILJOEN*, N. GERICKE (*Department of Pharmaceutical Sciences, Faculty of Science, Tshwane University of Techno-

logy, Private Bag X680, Pretoria 0001, SouthAfrica, viljoenam@tut.ac.za): Devil's Claw - A review of the ethnobotany, phytochemistry and biological activity of *Harpagophytum procumbens.* J. Ethnopharmacol. 138, 755-771 (2012). HPTLC studies of *Harpagophytum procumbens* such as the quantification of harpagoside were reviewed. HPTLC of harpagoside in the roots of *Harpagophytum procumbens* on silica gel with dichloromethane - methanol - acetic acid 79:20:1. Detection by dipping into anisaldehyde - methanol - acetic acid - sulphuric acid 1:170:20:10, followed by heating at 120 °C for 5 min. Quantitative determination by absorbance measurement at 285 nm. HPTLC provides comparable results with HPLC but is less time consuming.

herbal, review, HPTLC, quantitative analysis, qualitative identification 32e

110 117 M. MO (Mo Mingxiu), X. ZHANG (Zhang Xiaoying)*, H. LIANG (Liang Huiming), SH. OUYANG (Ouyang Shujing) (*Dep. of Pharm., Xinhui People's Hosp., Guangdong, Jiangmen 529100, China): (Study of the method for the identification of chief ingredients and determination of volatile oil in compound Bibao tablet) (Chinese). Chinese J. of Herald of Med. 31 (10), 1344-1347 (2012). Compound Bibao tablet is a herbal TCM preparation prescribed clinically to treat chronic rhinitis, nasosinusitis and migraine. For quality control a method for the identification of the main ingredients and determination of volatile oil in the preparation has been established. TLC on silica gel 1) for Licorice, with ethyl acetate - acetic acid - water 18:2:1 with chamber saturation with the mobile phase for 15 min, detection by spraying with 10 % sulfuric acid in ethanol and heating mildly until the zones are visible, evaluation in daylight and under UV 366 nm; 2) for *Asarum sieboldi* Mig., with petroleum ether (60-90 ºC) - ethyl acetate 3:1 with chamber saturation with the mobile phase for 15 min, detection at UV 254 nm; 3) *Flos Magnoliae liliflorae* Buds, with petroleum ether (60-90 ºC) - ethyl acetate - formic acid 15:5:2 with chamber saturation with the mobile phase for 15 min, detection under UV 366 nm.

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

110 118 D. MODI*, B. PATEL (*Institute of Pharmaceutical Education and Research, Department of Pharmaceutical Chemistry, Kadi Sarva Vishwavidyalaya, Gandhinagar 382023, Gujarat, India, darshana_pharma@yahoo.co.in): Simultaneous determination of metformin hydrochloride and glipizide in tablet formulation by HPTLC. J. Liq. Chromatogr. Relat. Technol. 35, 28-39 (2012). HPTLC of metformin hydrochloride and glipizide in tablet formulation on silica gel with water - methanol 3:1 + 1 drop ammonia. Quantitative determination by absorbance measurement at 236 nm. The hR_F value of compounds (1) and (2) were 22 and 85 and selectivity regarding matrix was given. Linearity was in the range of 5000-25000 ng/band for (1) and 50-250 ng/band for (2). Limits of detection and quantification were 991 and 3003 ng/band for (1) and 10 and 29 ng/band for (2), respectively. Recovery (by standard addition) was between 98.1 and 101.5 % for both (1) and (2).

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

110 119 Miriam MONFORTE, Cecilia GUIZAR, J. RUBIO, Mildred CARRILLO, F. VAZQUEZ* (*Escuela de Nutrición, Departamento de Ciencias de la Salud, Universidad Anáhuac del Mayab, Km 15.5 Carretera Mérida-Progreso, Mérida Yucatán, Mexico, felipe@cicy.mx): Berberine and sanguinarine quantitation in *Argemone mexicana* L. (Papaveraceae) tissues by TLC-in situ fluorography. J. Planar Chromatogr. 25, 358-360 (2012). TLC of berberine and sanguinarine on silica gel with *n*-butanol - water - ammonia 8:1:1. Detection by densitometry in fluorescence mode at 325 nm. The hR_F of berberine and sanguinarine were 29 and 95, respectively.

herbal, quality control, qualitative identification 32e

110 120 P. NARAYANA, R. SEKAR* (*Analytical Chemistry Division, Indian Institute of Chemical Technology, Tarnaka, Hyderabad-500007, India, seker@iict.res.in): Development and validation of a stability-indicating HPTLC determination of zafirlukast in bulk drug and pharmaceutical dosage form. J. Planar Chromatogr. 25, 559-565 (2012). HPTLC of zafirlukast on silica gel with toluene - methanol - acetone 7:2:1. Quantitative determination by absorbance measurement at 240 nm. Linearity was in the range of 50-400 ng/zone. Limits of detection and quantification were 16 and 50 ng/zone, respectively.

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

110 121 J. NOWAKOWSKA, P. PIKUL* (*Faculty of Pharmacy, Department of Physical Chemistry, Medical University of Gdansk, Al. Gen. J. Hallera 107, 80-416, Gdansk, Poland, pikul.piotr@gumed.edu.pl): Thermodynamic study of thermal decomposition of ranitidine by HPLC. J. Liq. Chromatogr. Relat. Technol. 35, 1676-1685 (2012). HPTLC of ranitidine hydrochloride thermal decomposition products on silica gel with dimethyl sulfoxide (DMSO), acetonitrile, methanol, 25 % ammonia, 2-propanol, and 2-methoxyethanol in a concentration range of 0-100 %. Detection under UV light at 254 nm. Degradation products with the binary mobile phases are arranged in three groups, depending on the retention: acetonitrile/DMSO at 373K (product 3), acetonitrile/DMSO at 353 K (product 3), and 2-methoxyethanol/DMSO at 353 K and 373 K (product of 2 and 3).

pharmaceutical research, quality control, HPTLC, qualitative identification 32a

110 122 D. OLENNIKOV*, M. V. PARTILKHAEV (*Laboratory of Medical and Biological Researches, Department of Biologically Active Substances, Institute of General and Experimental Biology, Siberian Division, Russian Academy of Sciences, Sakhyanovoy st. 6, 670047 Ulan-Ude, Russian Federation, oldaniil@rambler.ru): Isolation and densitometric HPTLC analysis of rutin, narcissin, nicotiflorin, and isoquercitrin in *Caragana spinosa* shoots. J. Planar Chromatogr. 25, 30-35 (2012). HPTLC of rutin (1), narcissin (2), nicotiflorin (3) and isoquercitrin (4) on silica gel with ethyl acetate - 1,2-dichloroethane - acetic acid - 85 % formic acid - water 100:25:10:10:8. Quantitative determination by absorbance measurement at 360 and 387 nm. Intraday and interday precisions were in the range of 1.5-1.9 % and 1.6-1.9 %, respectively. Recovery was found to be 98.2-101.4 % for (1), 98.0-101.1 % for (2), 98.2-100.5 % for (3) and 98.1-101.9 % for (4).

quality control, herbal, quantitative analysis, HPTLC 32e

110 123 L. PAILLAT, C. PERICHET, S. LAVOINE, U. MEIERHENRICH, X. FERNANDEZ* (*Université de Nice-Sophia Antipolis, Parc Valrose, 06108 Nice Cedex 2, France, xavier.fernandez@unice.fr): Validated high-performance thin-layer chromatography (HPTLC) method for quantification of vanillin-ß-D-glucoside, and four major phenolic compounds in vanilla (*Vanilla planifolia*) fruits, beans, and extracts. J. Planar Chromatogr. 25, 295-300 (2012). HPTLC of vanillin-ß-D-glucoside (1), *p*-hydroxybenzaldehyde (2), vanillic acid (3), p-hydroxybenzoic acid (4) and vanillin (5) on silica gel with *n*-hexane - chloroform - methanol 5:36:4 + 1 drop glacial acetic acid as mobile phase. Quantitative determination by absorbance measurement at 254 nm for (3) and (4), 280 nm for (1) and (2) and 313 nm for (5). The hR_F values for compounds (1) to (5) were 9, 62, 57, 42 and 77, respectively. Linearity was in the range of 24-121 ng/band for (1), 7-33 ng/band for (2), 20-102 ng/band for (3), 21-106 ng/band for (4) and 14-70 ng/band for (5). Limits of detection and quantification were 8 and 20 ng/band for (1), 2 and 6 ng/band for (2), 14 and 20 ng/band for (3), 6 and 20 ng/band for (4) and 4 and 8 ng/band for (5), respectively. Intermediate/inter-day/intra-day precision was below 1.4 % (n=6). Mean recovery for the compounds was between 96.8 and 101.4 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 124 J. PATEL, P. TRIPATHI, V. SHARMA, N.CHAUHAN, V. DIXIT* (*Department of Pharmaceutical Sciences, Dr. Harisingh Gour Vishwavidyalaya, Sagar 470003, M.P., India, vkdixit2011@rediffmail.com): Phyllanthus amarus: ethnomedicinal uses, phytochemistry and pharmacology: a review. J. Ethnopharmacol. 138, 286-313 (2011). The review covers literature across from 1980 to 2011. HPTLC studies of *Phyllanthus amarus* such as fingerprint profiles and detection of phyllanthin and hypophyllanthin as marker components were reviewed. Comparative results with HPLC were also described.

herbal, review, HPTLC, quantitative analysis, qualitative identification, *Phyllanthus amarus* 32e, 1

110 125 M. PATEL*, A. PATEL, C. PATEL, R. BADMANABAN (*Quality Assurance Department, Shree Sarvajanik Pharmacy College, Nr. Arvind Baug, Mehsana, Gujarat 384001, India, mitpatel23@gmail.com): A simple and sensitive HPTLC method for simultaneous analysis of tolperisone hydrochloride and etodolac in combined fixed-dose oral solid formulation. J. Planar Chromatogr. 25, 85-88 (2012). HPTLC of tolperisone hydrochloride (1) and etodolac (2) in combined dosage form on silica gel with toluene - ethylacetate - ethanol 12:3:5. Quantitative determination by absorbance measurement at 260 nm. The hR_F of (1) and (2) were 40 and 58, respectively. Linearity was in the range of 75-450 ng/band for (1) and 200-1200 ng/band for (2). Limits of detection and quantification were 1 and 4 ng/band for (1) and 2 and 5 ng/band for (2), respectively. The intermediate/inter-day/intra-day precision was below 2 % (n=3). Recovery was between 98.1 and 100.9 %.

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

110 126 P. PATEL*, M. PATEL, B. VYAS, D. SHAH, T. GANDHI (*Department of Pharmacology, Maliba Pharmacy College, Bardoli-MhuvaRoad, Tarsadi, Bardoli, District-Surat, Gujarat 394350, India, paras.pharm@gmail.com): Antiurolithiatic activity of saponin rich fraction from the fruits of *Solanum xanthocarpum* Schrad. & Wendl. (Solanaceae) against ethylene glycol induced urolithiasis in rats. J. Ethnopharmacol. 138, 160-170 (2012). HPTLC of solasodine in the fruits of *Solanum xanthocarpum* on silica gel with toluene - ethyl acetate - diethylamine 12:1:1. Quantitative determination by absorbance measurement at 200 nm. The hR_F of solasodine was 52.

herbal, quality control, HPTLC, quantitative analysis 32e

110 127 A. PATIL, A. SHIRKHEDKAR*, S. SURANA, P. NAWALE (*R.C. Patel Institute Pharmaceutical Education and Research, Shirpur Dist., Dhule, India, atulshirkhedkar@rediffmail.com): Simultaneous determination of propanolol hydrochloride and flunarizine dihydrochloride in bulk and capsule using reversed-phase high-performance thin layer chromatography/densitometry. J. Chil. Chem. Soc. 57, 1033-1035 (2012). HPTLC of propanolol hydrochloride (1) and flunarizine dihydrochloride (2) in combined dosage form on silica gel with methanol - toluene - ammonia 14:6:1. Quantitative determination by absorbance measurement at 267 nm. The hR_F of compounds (1) and (2) were 63 and 48, respectively. Linearity was in the range of 800-4800 ng/zone for (1) and 200-1200 ng/zone for (2). Limits of detection and quantification were 25 and 75 ng/zone for (1) and 16 and 48 ng/zone for (2), respectively. Intermediate/intra-day/inter-day precision was below 2.0 % (n=6). Recovery for both (1) and (2) was between 99.3 and 100.9 %.

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

110 128 S. PAWAR, S. DHANESHWAR* (*Department of Pharmaceutical Chemistry, Bharati Vidyapeeth University, Poona College of Pharmacy, Pune, Maharashtra, India 411038, sunil.dhanesh-

war@gmail.com): Application of stability indicating high-performance thin-layer chromatographic method for quantitation of desvenlafaxine in pharmaceutical dosage form. J. Liq. Chromatogr. Relat. Technol. 35, 499-510 (2012). HPTLC of desvenlafaxine in dosage forms on silica gel with ethyl acetate - toluene - methanol 14:4:1 + 1 drop ammonia. Quantitative determination by absorbance measurement at 228 nm. The hR_F value of desvenlafaxine was 48 and selectivity regarding matrix was given. Linearity was between 100 and 1000 ng/spot. The intermediate/interday/intra-day precision was below 0.2 % (n=6). The limits of detection and quantification were 10 and 100 ng/spot, respectively. Recovery (by standard addition) was between 97.9 and 99. 1%.

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

110 130 L. POTALE, A. KHODKE, S. PATOLE, M. DAMLE* (*Department of Pharmaceutical Chemistry, AISSMS College of Pharmacy, Kennedy Road, Near RTO, Pune 411001, Maharashtra, India, mcdamle@rediffmail.com): Development and validation of a stability-indicating HPTLC method for the determination of mirtazapine as bulk drug and in pharmaceutical formulation. J. Planar Chromatogr. 25, 72-76 (2012). HPTLC of mirtazapine on silica gel with methanol - chloroform 9:1. Quantitative determination by absorbance measurement at 294 nm. The hR_F of mirtazapine was 56. Linearity was in the range of 400-2000 ng/band. The intermediate/interday/intra-day precision was below 2 %.

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

110 131 H. QI (Qi Hong)*, CH. LIU (Liu Chuantong) (*The People's Hosp. of Shimen, Hunan, Changde 415300, China): (Study on quality standard of Dingxuan granules) (Chinese). J. China Pharm. Univ. 21 (3), 16-18 (2012). Dingxuan granules are a herbal TCM preparation with nourishing effect on liver and kidney and curative effect on Meniere's syndrome. For quality control, TLC of the extracts of the medicine 1) for Radix Polygoni multiflori preparata, on silica gel first over 3.5 cm with chloroform - methanol 7:3, then over 7 cm with chloroform - methanol 20:1, detection at UV 366 nm; 2) for *Radix Paeoniae alba*, on silica gel with chloroform - methanol 4:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 4:1 and heating at 105 °C, viewing under daylight; 3) for the fruit of Chinese wolfberry, on silica gel developed with chloroform - ethyl acetate - formic acid 2:3:1, detection at UV 366 nm; 4) for *Angelica sinensis*, on silica gel with *n*-hexane - ethyl acetate 17:3, detection at UV 366 nm.

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

110 132 F. QIAN (Qian Fang)*, Q. DA (Da Qingguo) (*Jiangsu Provinc. Hosp. of TCM, Jiangsu, Nanjing 210029, China): (Study on the method for identication of Zhilou fumigating lotion by thin-layer chromatography) (Chinese). Chinese J. of Guide for Trad. Chinese Med. & Pharm. 18 (7), 78-79 (2012). Zhilou fumigating lotion is a herbal TCM preparation for treatment of external hemorrhoids and hemorrhoid postoperation hemorrhage. TLC of the extracts of the preparation 1) for *Rheum officinale*, on silica gel with the upper phase of petroleum ether (30-60 ºC) - ethyl formate - formic acid 15:5:1, detection at UV 366 nm; 2) for *Rhizoma Polygoni Cuspidati*, on silica gel with chloroform - acetone - formic acid 16:3:1, detection at UV 366 nm; 3) for Chinese gall, on silica gel with chloroform - formyl acetate - formic acid 5:5:1, detection at UV 254 nm; 4) for *Herba Houttuyniae*, on silica gel with toluene - ethyl acetate - formic acid - water 20:10:1:1, detection at UV 366 nm; 5) for *Salvia plebeia* R.Br., on polyamide phase with methanol - acetic acid - water 13:3:4, detection at UV 366 nm.

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

110 133 X. QIN (Qin Xiangyang) (Chongqing Municip. Hosp. Specialized for lungs, Chongqing 400020, China): (Identification of the main component drugs in Chuanxining pills by thin-layer chromatography) (Chinese). Chinese J. of Trad. Chinese Med. 21 (4), 585-586 (2012). Chuanxining pills are a herbal TCM preparation effective for tonifying kidney, relieving uneasiness of mind, body tranquilization, relieving a cough,and preventing asthma. They are prescribed clinically to cure bronchitis and pneumonectasis. For quality control, identification of the three main component drugs by TLC of the extracts of the preparations 1) for *Epimedium davidii* Franch, on silica gel with ethyl acetate - butanone - formic acid - water 10:1:1:1, detection by spraying with 2 % ferric chloride in ethanol and mild heating; 2) for *Fructus Ligustri Lucidi*, on silica gel with chloroform - methanol 40:1, detection by spraying with 10 % sulfuric acid in ethanol and heating mildly; 3) for *Angelica sinensis*, on silica gel with *n*-hexane - ethyl acetate 9:1, detection at UV 366 nm.

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

110 134 J. QIU (Qiu Jianyong)*, D. HONG (Hong Duyun), S. LI (Li Sumei), Y. LI (Li Yangxue) (*Guangdong Provinc. No. 2 Hosp. of Trad. Chinese Med., Guangzhou 510095, China): (Study on the quality standard of Sangma oral liquid) (Chinese). J. Jiangxi Univ. of TCM 23 (6), 49-52 (2011). Sangma oral liquid is a herbal TCM preparation for clearing heat, opening the inhibited lung-energy, reducing phlegm, relieving a cough, and curing children acute bronchitis. TLC of the extracts of the preparations 1) for Forsythia fruit, on silica gel with chloroform - methanol 7:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C until the zones are visible, viewing under daylight; 2) for Baical Skullcap root, on polyamide with toluene - ethyl acetate - methanol - formic acid 10:3:1:2, detection by spraying with 5 % ferric chloride in ethanol and viewing under daylight; 3) for Tatarian Aster root, on silica gel with chloroform - methanol - formic acid 90:10:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones are visible, evaluation under UV 366 nm.

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

110 135 A. RAJOPADHYE, T. NAMJOSHI, A. UPADHYE* (*Agharkar Research Institute, G. G. Agarkar Road, Pune, India, anuradhaupadhye@aripune.org): Rapid validated HPTLC method for estimation of piperine and piperlongumine in root of *Piper longum* extract and its commercial formulation. Brazilian Journal of Pharmacognosy 22, 1355-1361 (2012). HPTLC of piperine (1) and piperlongumine (2) on silica gel with toluene - ethyl acetate 3:2 as mobile phase. Quantitative determination by absorbance measurement at 342 and 325 nm for (1) and (2), respectively. The hR_F of compounds (1) and (2) were 51 and 74, respectively. Linearity was in the range of 20-100 ng/zone for (1) and 30-150 ng/zone for (2). Limits of detection and quantification were 7 and 20 ng/zone for (1) and 10 and 30 ng/zone for (2), respectively. Intermediate/intra-day/inter-day precision was below 1.1 % (n=6). Recovery for both (1) and (2) was between 93.7 and 96.7 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 136 I. REZIC*, Marina ZELENIC, T. REZIC (*Laboratory of Analytical Chemistry, Department of Applied Chemistry, Faculty of Textile Technology, University of Zagreb, Zagreb, Croatia, iva_rezic@net.hr): Monitoring of bioprocess by thin-layer chromatography in a horizontal rotating tubular bioreactor for removal of heavy metals. J. Planar Chromatogr. 25, 89-90 (2012). TLC of sample solutions from a horizontal rotating tubular bioreactor on cellulose-coated TLC plates. Detection of metal ions by spraying with solutions of quercetine (0.1 g/100 mL 2-propanol) and dimethylglyoxime (1.0 g/100 mL ethanol), and exposure to ammonia vapor. Linearity was found

to be in the range of 0.05-1.0 g/L.

pharmaceutical research, quality control, qualitative identification 32a

110 137 C. RUMALLA, B. AVULA, Y. WANG (Yan-Hong Wang), T. SMILLIE, I. KHAN* (*Department of Pharmacognosy, School of Pharmacy, The University of Mississippi, University, MS 38677, USA, ikhan@olemiss.edu): Densitometric HPTLC method development and analysis of anthocyanins from Acai (*Euterpe oleracea* Mart.) berries and commercial products. J. Planar Chromatogr. 25, 409-414 (2012). HPTLC of two anthocyanins, cyanidin-3-O-rutinoside (1) and cyanidin-3-O-glucoside (2), in the berries of *Euterpe oleracea* Mart. on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:26. Quantification by absorbance measurement at 520 nm. The hR_F of compounds (1) and (2) were 29 and 37, respectively. Linearity was in the range of 100-500 ng/zone. The limit of detection was 30 ng/zone for (1) and 40 ng/zone for (2) and the limit of quantification was 100 ng/zone for both (1) and (2). The intermediate/inter-day/intra-day precision was below 2.8 % (n=3). The average recovery was between 99.8-101.8 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 138 M. SAJEWICZ, D. STASZEK, M. HAJNOS, Teresa KOWALSKA* (*Institute of Chemistry, University of Silesia, 9 Szkolna Street, 40-006, Katowice, Poland, teresa.kowalska@vs.edu.pl): Comparison of TLC and HPLC fingerprints of phenolic acids and flavonoids fractions derived from selected Sage (Salvia) species. J. Liq. Chromatogr. Relat. Technol. 35, 1388-1403 (2012). HPTLC fingerprint of phenolic acids and flavonoids in 23 different sage species on silica gel with different mobile phases. When compared with spectrophotometric and HPLC/DAD methods, the HPTLC approach was a sufficient alternative by quickly providing a valuable complementary fingerprint material. HPTLC of 1) free phenolic acids and phenolic acids fractions derived from acid and basic hydrolysis, with benzene - ethyl acetate - formic acid 6:3:1; 2) Flavonoid aglycons with toluene - ethyl acetate -- formic acid 12:6:1; 3) basic flavonoid glycosides and acidic flavonoid glycosides fractions with ethyl acetate - water - formic acid - acetic acid 100:26:11:11. Detection under UV light at 366 nm.

herbal, quality control, HPTLC, qualitative identification 32e

110 139 A. SALAMA*, M. EL RIES, S. TOUBAR, M. HAMIDE, M. WALASH (*Pharmaceutical Chemistry Department, National Organization for Drug Control and Research, Giza, Egypt, salama_nahla2004@hotmail.com): Validated TLC and HPLC stability-indicating methods for the quantitative determination of dapsone. J. Planar Chromatogr. 25, 65-71 (2012). HPTLC of dapsone in presence of its oxidative degradants on silica gel with acetate - toluene 1:1. Quantitative determination by absorbance measurement at 289 nm. Linearity was in the range of 0.5-6.0 µg/zone. Recovery was 99.4 %. The method showed comparable results to a validated HPLC method.

pharmaceutical research, quality control, HPTLC, quantitative analysis, comparison of methods 32a

110 140 L. SAWANT*, Y. KACHWALA, P. SANGAVE, N. PANDITA (*School of Pharmacy and Technology Management, SVKM's NMIMS, Vile Parle (W), Mumbai-400056, India, laxmanpsawant@gmail.com): High-performance thin-layer chromatographic quantification of kaempferol and apigenin in the whole-plant powder of *Sida spinosa* Linn. J. Planar Chromatogr. 25, 301-305 (2012). HPTLC of kaempferol (1) and apigenin (2) in the whole-plant powder of *Sida spinosa* Linn. on silica gel with dichloromethane - methanol - formic acid 16:2:1. Quantitative determination by absorbance measurement at 340 nm. Linearity was in the range of 150-450 µg/mL for

(1) and 50-150 µg/mL for (2). The intermediate/inter-day/intra-day precision was below 2 %. Recovery was 99.5 % for (1) and 99.3 % for (2).

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

110 141 S. SHANKAR, A. SRIVASTAVA, K. RAWAT* (*Pharmacognosy & Ethnopharmacology Division, National Botanical Research Institute (CSIR), Rana Pratap Marg, Lucknow-226 001, India, pharmacognosy1@rediffmail.com): Isolation and quantification of vanillin through flash & HPTLC chromatographic techniques from *Decalepis hamiltonii* Wight and Arn root and their antioxidant studies. J. Liq. Chromatogr. Relat. Technol. 35, 2396-2407 (2012). HPTLC of vanillin in the roots of *Decalepis hamiltonii* Wight and Arn on silica gel with toluene - ethyl acetate 9:1. Quantitative determination by absorbance measurement at 254 nm. The hR_F value of vanillin was 42. Linearity was in the range of 2-12 µg/zone. Limits of detection and quantification were 1.3 and 3.9 µg/zone. The intermediate/inter-day/intra-day precision was 1.3 % (n=3), respectively. Recovery was between 99.4 and 100.3 %.

herbal, quality control, quantitative analysis, HPTLC 32e

110 142 E. SHIKANGA, I. VERMAAK, A. VILJOEN* (*Tshwane University of Technology, Private Bag X680, Pretoria, 0001, South Africa; viljoenam@tut.ac.za): An HPTLC-densitometry method for the quantification of pharmacologically active alkaloids in *Sceletium tortuosum* raw material and products. J. Planar Chromatogr. 25, 283-289 (2012). HPTLC of mesembranol (1), mesembrenol (2), mesembrine (3) and mesembrenone (4) in the aerial parts of *Sceletium tortuosum* on silica gel with dichloromethane - methanol 9:1 + 1 drop ammonia. Quantitative determination by absorbance measurement at 280 nm. The hR_F values for compounds (1) to (4) were 8, 28, 60 and 71, respectively. Linearity was in the range of 180-240 ng/band for (1) to (3) and 60-300 ng/band for (4). Limits of detection and quantification were 25 and 75 ng/band for (1), 31 and 95 ng/band for (2), 27 and 80 ng/band for (3) and 18 and 44 ng/band for (4), respectively. The intermediate/inter-day/intra-day precision was below 1.6 %. Mean recovery for the compounds was between 90.1 and 104.7 %.

herbal, quality control, HPTLC, quantitative analysis 32e

110 143 A. SHIRKHEDKAR*, D. DHUMAL, S. SURANA (*Department of Pharmaceutical Chemistry, R.C. Patel Institute of Pharmaceutical Education and Research, Shirpur, Dist: Dhule (M.S.) 425 405, India, atulshirkhedkar@rediffmail.com): A sensitive and specific high-performance thin-layer chromatography-densitometry method for determination of rebamipide in the bulk material and in pharmaceutical formulation. J. Planar Chromatogr. 25, 368-373 (2012). HPTLC of rebamipide on silica gel with toluene - methanol - triethylamine 16:9:1. Quantitative determination by absorbance measurement at 230 nm. The hR_F of rebamipide was 59. Linearity was in the range of 100-600 ng/band. Limits of detection and quantification were 6 and 18 ng/band, respectively. Intermediate intra-day/inter-day precision was below 1.7 % (n=3). Recovery (by standard addition) was between 100.1 and 100.9 %. The proposed HPTLC method is equivalent to a reported HPLC method.

pharmaceutical research, quality control, comparison of methods, quantitative analysis, HPTLC 32a

110 144 S. SONG (Song Sankong)*, X. SONG (Song Xia), X. YANG (Yang Xiaoyu), H. JIAO (Jiao Haisheng) (*Coll of Pharm., Lanzhou Univ., Gansu, Lanzhou 730000, China): (Qualitative identification of Rubingxiao tablet and quantitative determination of tetrahydropalmatine in the prepa-

ration) (Chinese). J. China Pharm. Univ. 21 (3), 22-23 (2012). Rubingxiao tablet is a herbal TCM preparation prescribed to treat mastitis and hyperplasia of mammary glands. Quality control by qualitative identification of its major component drugs and quantitative determination of tetrahydropalmatine in it. TLC of the extracts of the preparations on silica gel 1) for *Epimedium davidii* Franch, with ethyl acetate - butanone - formic acid - water 10:1:1:1, detection at UV 366 nm, followed by spraying with 1 % aluminium chloride in ethanol and viewing under UV 366 nm; 2) for *Astragalus mongholicus*, with chloroform - methanol 10:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones were visible, evaluation at UV 366 nm; 3) for *Poria cocos*, with toluene - ethyl acetate - formic acid 40:10:1, detection by spraying with 2 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C until the zones were visible, viewing under daylight.

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

110 145 D. SUN (Sun Dongmei)*, ZH. TAN (Tan Zhican), X. BI (Bi Xiaoli), W. LUO (Luo Wenhui) (*Guangdong Provincial Inst. of TCM, Guangzhou 510095, China): (Identification of Microcos paniculata Linn by thin-layer chromatography (TLC) and Fourier transform infrared spectroscopy (FTIR)) (Chinese). J. China Pharm. Univ. 21 (12), 15-16 (2012). The leaf of *Microcos paniculata* L. is a popular medicinal herbal drug used for cardiovascular protection, blood fat adjusting, and anti-aging. Presentation of methods for identifying the drug by TLC and FTIR. TLC of the extracts of the drug on silica gel 1) for kaempferide, with toluene - ethyl acetate - formic acid 9:3:1, detection by spraying with 3 % aluminiumchloride in ethanol followed by heating at 105 ºC for 5 min and viewing at UV 366 nm; 2) for vitexin and isovitexin, developed with ethyl acetate - methanol - water 100:17:13, detection by spraying with 3 % aluminiumchloride in ethanol followed by heating at 105 ºC for 5 min and viewing at UV 366 nm. The methods have been applied to 15 batches of real life sample available from different places of origin, and proved that TLC procedures as well as FTIR are simple, sensitive, reproducible and robust and suitable for the identification of the herbal drug.

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

110 146 H. TAN (Tan Huaimei)*, F. ZHOU (Zhou Fangyong), W. ZHANG (Zhang Wenxin) (*Zunyi Med. & Pharm. College, Guizhou, Zunyi 563000, China): (Study of the method for the quality control of Changyanning oral liquid) (Chinese). J. China Pharm. Univ. 21 (10), 37-39 (2012). Changyanning oral liquid is a herbal TCM preparation with *Euphorbia humifusa* Willd as the key component drug. For quality control identification of *Euphorbia humifusa* Willd by TLC and quantification of its active component gallic acid by HPLC. TLC on silica gel with toluene - ethyl acetate - formic acid 5:2:1, detection by spraying with 1 % aluminium chloride in ethanol, evaluation under UV 366 nm.

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

110 147 B. TITA, Olivia MARUTOIU, D. TITA, C. MARUTOIU*, Maria SORAN, Z. MOLDOVAN (*Babes-Bolyai University, Faculty of Orthodox Theology, 18 Avram Iancu Square, Cluj-Napoca, Romania, cmarutoiu@yahoo.com): Separation and identification of some non-steroidal anti-inflammatory drugs using TLC and HPLC-MS. J. Planar Chromatogr. 25, 523-527 (2012). TLC of a mixture of 12 non-steroidal anti-inflammatory drugs on silica gel with ethyl acetate - toluene - methanol - acetic acid 40:40:1:1. Quantitative determination by absorbance measurement at 254 nm. Limits of detection were in the range of 5-500 ng/zone. The method showed comparable

results to a validated HPLC method.

pharmaceutical research, qualitative identification, quantitative analysis 32e

110 148 S.S. TIWARI, M.M. PANDEY, S. SRIVASTAVA, A. RAWAT* (* Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute (CSIR), Lucknow, India): TLC densitometric quantification of picrosides (picroside-I and picroside-II) in *Picrorhiza kurroa* and its substitute *Picrorhiza scrophulariiflora* and their antioxidant studies Biomed. Chromatogr. 26 (1), 61-68 (2012) Picroside-I and picroside-II are known bioactive metabolites in Picrorhiza species. Presentation of a simple, precise method for the simultaneous determination of picrosides (picroside-I and picroside-II) in two different Picrorhiza species, *P. kurroa* and *P. scrophulariiflora.* TLC of the extracts of the medicinal herbal drugs on silica gel with chloroform - methanol 22:3. Quantification of picrosides (picroside-I and picroside-II) by absorbance measurement at UV 254 nm. Comparative study revealed that the content of picroside-I and picroside-II is higher in *P. scrophulariiflora* than *P. kurroa* the content of picroside-I was found to be 1.3 and 1.6 % in *P. kurroa* and P. *scrophulariiflora*, and of picroside-II 0.5 and 0.6 %, respectively. Study of the antioxidant potential of the two Picrorhiza species using DPPH* radical reagent. The scavenging activities of *P. kurroa* and *P. scrophulariiflora* were 37.7 % and 34.3 %, respectively, at a concentration of 0.1 mg/mL.

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

110 149 X. TONG (Tong Xin), W. LIU (Liu Wen), X. LIANG (Liang Xiaoxu), W. QI (Qi Wen), Y. PAN (Pan Yingni), H. HUA (Hua Huiming), X. LIU (Liu Xiaoqiu)* (*Coll of TCM, Shenyang Pharm. Univ., Shenyang 110016, China): (Qualitative and quantitative analysis of triperpenoids in Olibanum) (Chinese). J. of Modern Trad. Chinese Med. 14 (7), 11-13 (2012). Olibanum is the resin from the bark of *Boswellia carterii* Birdw. and *Boswellia bhaw*. dajiana Birdw. It containis triperpenoids such as 11-keto-beta-boswellic acid (KBA), and acetyl-11-keto-beta-boswellic acid (AKBA) which show antiinflammatory and antibiotic activity. In order to discern the false from the genuine drug in market, a method for the quality control of Olibanum produced in Indonesia, Ethiopia, Somalia and India has been presented. TLC on silica gel with cyclohexane - ethyl acetate - glacial acetic acid 50:10:1, detection at UV 254 nm.

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

110 150 S. WALODE*, A. KASTURE, S. WADODKAR (*Sinhgad Institute of Pharmaceutical Sciences, Kusgaon (Bk), Lonavala, Pune 410401, Maharashtra, India, sanjuwalode@rediffmail.com): Stability-indicating HPTLC method for the determination of atorvastatin and ezetimibe: application to pharmaceutical dosage forms. J. Planar Chromatogr. 25, 81-84 (2012). HPTLC of atorvastatin (1) and ezetimibe (2) on silica gel with methanol - toluene - chloroform - triethylamine 2:16:1:1. Quantitative determination by absorbance measurement at 259 nm. The hR_F values of compounds (1) and (2) were 7 and 37, respectively. Linearity was in the range of 500-1500 ng/band for both (1) and (2). Intermediate/inter-day/intra-day precision was below 2 % (n=3). Mean recovery was 99.7 % for both active agents.

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

110 151 A. WANG (Wang Aiying)*, ZH. WANG (Wang Zhemin), F. SHI (Shi Fuxiang), M. XIN (Xin Min), H. CUI (Cui Haixia) (*Tianshui Municip. Inst. of Drug Cont., Gansu, Tianshui 741018, China): (Detection of antidiabetics illegally mixed into health foods by thin-layer chromatogra-

phy combined with UV spectrophotometry) (Chinese). J. China Pharm. Univ. 26 (2), 150-154 (2012). Some health foods were illegally mixed with certain kinds of medicine which may harm the health of consumers. For example antidiabetics like glibenclamide, glipizide and gliclazide were illegaly added to health foods marketed for adjusting blood sugar. Presentation of TLC and UV methods for detection of these illegal additives. TLC on silica gel with chloroform - petroleum ether (60-90 ºC) - methanol - glacial acetic acid 8:14:1:1, detection at UV 254 nm. The methods were applied to three varieties of real life samples and the results showed no difference to those obtained by LC-MS.

food analysis, quality control, qualitative identification, comparison of methods 32e

110 152 Q. WANG (Wang Qi)*, Y. ZHANG (Zhang Yumei), ZH. DAI (Dai Zhong), J. LU (Lu Jing), R. LIN (Lin Ruichao) (*National Inst. for Food & Drug Contr., Beijing 100050, China): (Study on the method for the identification of amino acids and flavonoids in Astragalus by thin-layer chromatography) (Chinese) J. of China Pharm. 26 (1), 50-53 (2012) Astragalus, as traditional Chinese medicinal herbal crude drug, is the dried root of *Astragalus membranaceus* (Fisch.) Bge. Var. Monghoicus (Bge.) Hisao or *Astragalus membranaceus* (Fisch.) Bge. It is specially effective for nourishing vitality, invigorating splenic yang, inducing diuresis for removing edema and promoting tissue regeneration. TLC of the extracts of the crude drugs on silica gel 1) for amino acids (standards arginine, alanine, proline), with carbolic acid - water 5:3 (carbolic acid was double distilled just before mixing with water), detection by spraying with 0.5 % ninhydrin in ethanol and heating at 105 ºC until the zones are visible under daylight; 2) for flavonoids (standards formononetin, calycosin glucoside, calycosin), with chloroform - methanol 10:1, detection by exposure to ammonia vapors for a few minutes and viewing under UV 254 nm.

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

110 153 SH. WANG (Wang Shiqing) (Puyang Municip. Inst. of Drug Cont., Henan, Piyang 457000, China): (Qualitative analysis of rhaponiticin in Yankening tablets by thin-layer chromatography) (Chinese). J. Chinese Med. 25 (151), 1134-1135 (2010). Yankening tablet, specially effective for diminishing inflammation, is a herbal TCM preparation with *Rheum officinale* as key component drug. However, some counterfeits are used to replace the certified products, thus abating seriously the curative effect of the medicine. To control the quality of the medicine a method has been presented for inspection of rhaponiticin in Yankening tablets. TLC of the extracts of the medicine on silica gel with ethyl acetate - butanone - formic acid - water 10:7:1:1, detection under UV 366 nm. Identification 1) by fingerprint comparison with the standard rhaponiticin; 2) by comparing the online scanning spectra over the wavelength range from 200 - 700 nm.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, densitometry, comparison of methods 32e

110 154 X. WANG (Wang Xuefeng)*, J. CHEN (Chen Junming), Z. ZHENG (Zheng Ziyu), P. WANG (Wang Ping), X. JIA (Jia Xiaoguang), H, WEI (Wei Hongyan) (*The Group of TCM, Xinjiang Pharm. Co. Ltd., Xinjiang, Urumqi 830032, China): (Study of the method for the quality control of Compound Xuelian tablet) (Chinese). J. of Xinjiang Trad. Chinese Med. 30 (3), 70-72 (2012). Compound Xuelian tablet is a herbal TCM preparation prescribed clinically to cure rheumatism. TLC of the extracts of the medicine on silica gel 1) for *Saussurea involucrata*, with the upper phase of ethyl acetate - formic acid - water 10:1:2, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones are visible in daylight; 2) for *Rhizoma Corydalis*, with cyclohexane - chloroform - methanol 10:6:1, detection by exposure to iodine vapors until the zones are visible, evaluation in daylight and under UV 366 nm; 3) for *Radix Angelicae Pu-*

bescentis, with petroleum ether (30-60 ºC) - formyl acetate - formic acid 15:5:1, detection under UV 366 nm.

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

110 155 Y. WANG (Wang Ying) (Chongqing Municip. Trad. Chinese Med. Hosp., Chongqing 400021, China): (Study on the method for the quality control of Ningxin pills) (Chinese). Chinese J. of Trad. Chinese Med. for Emerg. 21 (5), 738-739 (2012). Ningxin pills are a TCM preparation with special efficiency on promoting blood circulation, removing blood stasis, relieving pain and are prescribed clinically to treat coronary disease and angina pectoris. For quality control, TLC of the extracts on silica gel 1) for *Panax Notoginseng* (Burk.) F.H.Chen, R*adix Ginseng rubra*, with the lower phase of chloroform - ethyl acetate - methanol - water 15:40:22:10, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C, viewing in daylight; 2) for *Rhizoma Corydalis*, with toluene - acetone 9:2, detection by exposure to iodine vapors and evaluation at UV 366 nm; 3) for borneol, with petroleum ether (30-60 ºC) - ethyl acetate - chloroform 11:1:3, detection by spraying with 1 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C, viewing in daylight.

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

110 156 Y. WANG (Wang Yinliang)*, H. YE (Ye Honghui), ZH. CHEN (Chen Zhongwu), ZH. TA (Ta Zhaoyin) (*Luoding Municip. People's Hosp., Inner Mongolia, Luoding 527200, China): (Study on the method for the quality of Jiangding Doukou Qufeng compound oral liquid by thin-layer chromatography) (Chinese). Inner Mongolian J. of Trad. Chinese Med. 10, 35-36 (2012). Jiangding Doukou Qufeng compound oral liquid is a herbal TCM preparation prescribed to effectively cure flatulence, loss of appetite, nausea, and vomiting. TLC of the extracts of the preparations on silica gel 1) for *Cortex Cinnamomi* and *Foeniculum vulgare*, with petroleum ether (60-90 ºC) - ethyl acetate 17:3, detection by spraying with 2,4-dinitrophenylhydrazine solution in 2N hydrochloric acid and viewing in daylight; 2) for *Rhizoma Zingiberis*, with petroleum ether (60-90 ºC) - chloroform - ethyl acetate 2:1:1, detection by spraying with 2 % vanillin in sulfuric acid - ethanol 1:200 and heating at 105 °C until the zones were visible in daylight.

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

110 157 Y. WANG (Wang Yu), M. GUO (Guo Maofeng), Y. XU (Xu YaN), K. QIN (Qin Kunmig) B. CAI (Cai Baochang)* (*Res. Center of Nat. Min. of Educ. for the Proj. of TCM Proc. Normal., Nanjing Univ. of TCM, Nanjing 210061, China): (Study of the factors influencing on the degradation of stachydrine in Yimucao ointment during its storage) (Chinese). J. of Global Trad. Chinese Med. 5 (5), 362-363 (2012). Yimucao ointment is a TCM preparation for curing irregular menstruation and postpartum blood stasis. It was found recently that degradation of stachydrine, the active component in the medicine appeared during its storage. In order to improve the quality control of the medicine the stability and influencing factors during its storage have been investigated. TLC on silica gel with *n*-butanol - ethyl acetate - hydrochloric acid 8:1:3, detection by heating at 105 °C for 15 min firstly and then spraying with 1 % ferric chloride in ethanol - 5 % potassium iodobismuthate solution 1:10 until the zones were visible in daylight. Quantitative determination of stachydrine by densitometry at 510 nm. Investigation of the influence of temperature and light on the content of stachydrine in Yimucao ointment showed that light is the primary reason causing degradation during its storage.

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

110 158 L. WULANDARI, M. YUMONO, G. INDRAYANTO* (*Faculty of Pharmacy, Airlangga University, Dharmawangsa Dalam, Surabaya 60286, Indonesia, gunawanindrayanto@yahoo.com): Densitometric determination of mebhydrolin napadisylate in tablets. J. Planar Chromatogr. 25, 60-64 (2012). HPTLC of mebhydrolin napadisylate in tablets on silica gel with methanol - ethyl acetate 1:1. Quantitative determination by absorbance measurement at 287 nm. Linearity was in the range of 600-1600 ng/zone. Limits of detection and quantification were found to be 19 and 56 ng/zone, respectively. The intermediate/inter-day/intra-day precision was below 2 %. Recovery (by standard addition) was in the range of 99.3-100.8 %.

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

110 159 CH. XIAN (Xian Chun), X. GONG (Gong Xiaojian)*, ZH. YANG (Yang Zhannan) (*Key Lab. for Inform. System of Mountainous Areas & Protection of Ecological Environment, Guizhou Norm. Univ., Guizhou, Guiyang, 550001, China): (Study on the method for the identification of *Saxifraga stolonifera* (L.) Meerb. by thin-layer chromatography) (Chinese). J. of Guizhou Normal Univ. (Natural Sci.) 30 (3), 7-8 (2012). *Saxifraga stolonifera* (L.) Meerb is a TCM herb for heat-clearing and detoxification and is often used as the key component in the preparations for curing otitis media, traumatic bleeding and hyperplasia of the prostate. TLC of the extracts of the whole medicinal herb and gallic acid as standard on polyamide phase with chloroform - methanol - formic acid 25:5:1, detection by spraying with a solution of ferric chloride - 2N hydrochloric acid - water 2:1:100 and heating until the zones are visible, viewing in daylight.

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

110 160 L. XIAO (Xiao Lixiang) (Xishuangbanna Autonomous Prefecture Hosp. of Dai Nationality Med., Yunnan, Jinghong 666100, China): (Approach of identification of different officinal positions of *Cassia fistula* by thin-layer chromatography) (Chinese). Chinese J. Ethnopharm.(4), 51-52 (2012). *Cassia fistula*, as a crude drug of Dai nationality medicine, is the mucus from the dried pod of *Cassia fistula* L. It has special efficiency in relieving internal heat or fever, apocatastasis and relieving pains. Its pods and leaves are both used as the component crude drug in preparations. To identify different officinal positions of the drugs available in different places of origin, TLC of the extracts of the drug on silica gel with petroleum ether (60-90 ºC) - ethyl acetate - formic acid 15:10:1, detection by spraying with 5 % aluminum chloride in ethanol and heating at 105 °C, evaluation at UV 366 nm.

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

110 161 R. XIE (Xie Ruonan)*, M. YANG (Yang Manqin), R. XU (Xu Ruowei), D. HU (Hu Die), W. LI (Lin Wenqi) (*The 2nd Affiliated Hosp. of Anhui TCM Coll., Anhui, Hefei 230061, China): (Study on the method for the quality control of Shiyiwei Huoxue tincture) (Chinese) J. Anhui Med. & Pharm. 16 (6), 768-769 (2012). Shiyiwei Huoxue tincture is a herbal TCM preparation for activation of blood circulation and is prescribed clinically to treat damages of soft tissues and diseases of the bone and joint system. For quality control, TLC of the extracts of the preparations on silica gel 1) for borneol, with *n*-hexane - ethyl acetate 17:3, detection by spraying with 1 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C until the zones are visible, evaluation in daylight; 2) for *Rhizoma Corydalis*, with *n*-hexane - chloroform - methanol 15:8:2, detection by exposure

to iodine vapors and viewing in daylight and under UV 366 nm.

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

110 162 Y. XIE (Xie Yan), T. ZHU (Zhu Taiyong), N. QIN (Qin Na) (Henan Provinc. Osteopathic Res. Inst., Henan, Luoyang 471002, China): (Study of the method for the quality control of Yiqi Shenggu granules) (Chinese). J. of World Trad. Chinese Med. 7 (1), 77-78 (2012). Yiqi Shenggu granule is a newly developed herbal TCM preparation for treating wounded catagma. TLC of the extracts of the preparation on silica gel 1) for Aucklandia root, with petroleum ether (60-90 ºC) - ethyl acetate 9:1, detection by spraying with 5 % vanillin in ethanol - sulfuric acid 200:1 and mild heating, viewing in daylight; 2) for Carthamus flower, twice with chloroform - toluene - methanol 15:5:1, detection by spraying with 10 % sulfuric acid in ethanol and viewing at UV 366 nm; 3) for red Peony root, with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in ethanol - sulfuric acid 200:1 and mild heating, viewing in daylight. Determination of astragaloside A in the extract of the medicine by TLC on silica gel with chloroform - methanol - water 13:7:2, detection by spraying with 10 % sulfuric acid in ethanol and viewing at UV 366 nm. Quantification by densitometry at 520 nm with a linearity range of the calibration curve of 1.13-6.75 µg/zone (r = 0.995, $n = 6$), a precision of $\%RSD = 2.0$ ($n = 6$) within plate and plate-to-plate, and standard addition recovery of 99.4 % ($\%RSD = 2.3$, $n = 6$).

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

110 163 F. XU (Xu Feng)*, D. WU (Wu Dongpeng), CH. WANG (Wang Chun) (*Guangdong Vocational Coll. of Food & Drug, Guangdong, Guangzhou 510520, China): (Approach of the identification of Weijianning capsules by thin-layer chromatography) (Chinese). Chinese J. of Guide for Trad. Chinese Med. & Pharm. 18 (5), 80-81 (2012). Weijianning capsules, a herbal TCM preparation for tonifying spleen and invigorating the stomach, is prescribed clinically to treat superficial, erosive and atrophic gastritis. Presentation of a TLC method for the quality control of the preparations optimized by investigating the sample extraction procedure and the mobile phases employed. TLC of the extracts on silica gel 1) for *Radix Scrophulariae* with ethyl acetate - ethanol - water 20:5:3, detection by spraying with a solution of 3 g vanillin - 100 mL ethanol - 0.5 mL sulfuric acid and heating at 105 °C until the zones were clearly visualized, evaluation in daylight; 2) for *Fructus Amomi* with *n*-hexane - ethyl acetate 3:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones were clearly visualized, evaluation in daylight. Identification by fingerprint comparison in parallel with the individual standard components.

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

110 164 Y. XU (Xu Yong), Y. CHU (Chu Yanrong), ZH. Jia (Jia Zhengwei), K. WANG (Wang Ke), SH. JI (Ji Shen)* (*Shanghai Inst. for Food & Drug Contr.Shanghai 201203, China): (Study on the method for the quality standard of Zhachong Shisanwei pill) (Chinese. J. of Qilu Med. & Pharm. 31 (6), 327-330 (2012) Zhachong Shisanwei Pill is a herbal TCM preparation for invigorating the circulation of blood. TLC on silica gel 1) for *Fructus Chebulae* and the standard gallic acid, with chloroform - ethyl acetate - formic acid 3:2:1, detection by spraying with 2 % ferric chloride in ethanol and heating at 105 ºC until the zones are visible in daylight; 2) for *Radix Aucklandiae* and the standards costunolide and dehydrocostus lactone, with chloroform - cyclohexane 5:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 1:200 and heating at 105 ºC until the zones are visible in daylight; 3) for *Aquilaria agallocha* (Lour.) Roxb, with toluene - ethyl acetate 4:1, detection under UV 366 nm; 4) for *Rhizoma Acori Tatarinowii*, with petroleum ether

(60-90 ºC) - ethyl acetate 8:3, detection under UV 366 nm.

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

110 165 H. YADAV, P. MUNGARA, M. JIVRAJANI, M. NIVSARKAR, S. ANANDJIWALA* (*Department of Natural Products, National Institute of Pharmaceutical Education and Research (NIPER) - Ahmedabad, B. V. Patel Pharmaceutical Education and Research Development (PERD) Centre, Sarkhej-Gandhinagar Highway, Thaltej, Ahmedabad-380054, Gujarat, India, drsheetalanand@yahoo.co.in): TLC-densitometric quantification of negundoside, ursolic acid, eugenol, lupeol, and beta-sitosterol using HPTLC from *Vitex Negundo* leaves. J. Liq. Chromatogr. Relat. Technol. 35, 1565-1584 (2012). HPTLC of negundoside (1), ursolic acid (2), eugenol (3), lupeol (4), and beta-sitosterol (5) in the leaves of *Vitex Negundo* on silica gel with toluene - methanol 9:1. Detection by dipping in anisaldehyde sulfuric acid reagent for approximately 1 min, followed by heating at 100 °C for 5 min. Quantitative determination by absorbance measurement at 525 nm. The hR_F value of compounds (1) to (5) were 47, 61, 56, 54 and 38, and selectivity regarding matrix was given. Linearity was in the range of 200-800 ng/zone for (1), 72-576 ng/zone for (2), 200-1000 ng/zone for (3), 150-900 ng/zone for (4) and 80-480 ng/zone for (5). Limits of detection and quantification were 80 and 200 ng/zone for (1), 18 and 72 ng/zone for (2), 60 and 200 ng/zone for (3), 50 and 100 ng/zone for (4) and 20 and 60 ng/zone for (5), respectively. The method provides acceptable intra-day and inter-day precision for (1) to (5). The average recoveries for compounds (1) to (5) were found to be 99.9, 100.1, 99.7, 100.0, and 99.9 %, respectively.

herbal, quality control, HPTLC, quantitative analysis 32e

110 166 L. YANG (Yang Li), Y. ZUO (Zuo Yajie), H. LU (Lu Hong), J. ZENG (Zeng Jianguo)* (*Changsha Central Hosp., Hunan, Changsha 410004, China): (Study on the method for the quality control of Yifeitongluo granules) (Chinese). J. China Pharm. Univ. 21 (10), 26-27 (2012). Yifeitongluo granules are a herbal TCM preparation for the combined treatment of deficiency of both qi and yin. TLC of the extracts of the preparations on silica gel 1) for *Rhizoma Polygonati*, with chloroform - methanol - acetic acid 5:4:1, detection by spraying with 2.5 % phosphomolybdic acid in ethanol and heating at 105 °C until the zones are visible; 2) for *Viola yedonensis* Makino, with toluene - ethyl acetate - formic acid 5:3:1, detection under UV 366 nm; 3) for *Stemona sessilifolia* (Miq.) Miq, with toluene - propanone - methanol 16:6:1 after preconditioning with ammonia vapors, detection by spraying with 5 % potassium iodobismuthate and viewing in daylight.

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

110 167 L. YANG (Yang Li)*, CH. HU (Hu Changjiang), W. ZHOU (Zhou Wei), X. TAN (Tan Xiao) (*Sichuan Neo-Green Pharm. Co., Ltd., Sichuan, Chengdu 610081, China): (Study on the quality standard for *Ligusticum wallichii* dispensing granules) (Chinese). J. China Pharm. Univ. 21 (10), 28-29 (2012). Dispensing granules of TCM are prepared herbal medicines in small pieces ready for decoction used for making up a clinical prescription. *Ligusticum wallichii*, as crude herbal drug, is the root of *Ligusticum chuanxiong* Hort. Its effects are invigorating the circulation of blood, promoting the circulation of qi, relieving rheumatic pains, and it is used clinically to cure chest discomfort, cardialgia, irregular menses, algomenorrhea, abdominal pain and headache. For quality control, TLC on silica gel with toluene - ethyl acetate - formic acid 40:10:1, detection at UV 366 nm and under daylight after spraying with 1 % ferric chloride - 1 % potassium ferricyanide 1:1.

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

110 168 X. YANG (Yang Xiaoying) (Linying County People's Hosp. No.2, Henan, Linying 462600, China): (An improved method for the quality control of Langchuang pills) (Chinese). Chinese J. of Clin. Res. on Trad. Chinese Med. 4 (9), 34-35 (2012). Langchuang pills are a herbal TCM preparation for clearing heat, removing toxicity and invigorating the circulation of blood. For quality control, TLC of the extracts of the preparations on silica gel 1) for the root of red-rooted salvia, with chloroform - benzene 5:1, detection by viewing in daylight; 2) for *Radices Rehmanniae*, with chloroform - methanol 8:1, detection by spraying with a saturated solution of 2,4-dinitrophenylhydrazine in ethanol and viewing in daylight; 3) for the rhizome of Chinese Goldthread, with benzene - chloroform - ammonia 5:3:1, detection at UV 366 nm.

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

110 169 R. YOUSSEF*, E. KHAMIS, M. EL-SAYED, Mona MONEIM (*Faculty of Pharmacy, Department of Pharmaceutical Analytical Chemistry, University of Alexandria, El-Messalah, Alexandria 21521, Egypt, rmmy1973@yahoo.com): Validated HPTLC method for simultaneous determination of loratadine and desloratadine in presence of co-formulated drug. J. Planar Chromatogr. 25, 456-462 (2012). HPTLC of loratadine (1) and desloratadine (2) on silica gel with methanol + 1 drop ammonia. Quantitative determination by absorbance measurement at 254 nm. The hR_F values of (1) and (2) were 76 and 20, respectively. Linearity was in the range of 250-850 ng/band for (1) and 100-1000 ng/band for (2). Limits of detection and quantification were 90 and 250 µg/band for (1) and 30 and 100 µg/band for (2), respectively. Intra- and inter-day precisions were found to be less than 2 %. Recoveries were between 97.8 and 101.5 % for both (1) and (2).

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

10 170 Maria ZERAIK, Janete YARIWAKE*, J. WAUTERS, Monique TITS, L. ANGENOT (*São Carlos Institute of Chemistry, The São Paulo University, CP 780, 13560-970 São Carlos, Brazil, janete@iqsc.usp.br): Analysis of passion fruit rinds (*Passiflora edulis*): isoorientin quantification by HPTLC and evaluation of antioxidant (radical scavenging) capacity. Quim. Nova. 35, 541-545 (2012). HPTLC of isoorientin on silica gel with ethyl acetate - formic acid - water 9:1:1. Detection by dipping into a solution of diphenylboric acid-2-aminoethyl ester (100 mg) and PEG 400 (500 mg) in methanol (10 mL). Quantitative determination by fluorescence measurement at 366 nm. The HPTLC method (120 min) was almost seven times faster than the HPLC method (700 min). The amount of solvent consumed in the HPTLC method (12 mL) was almost eightfold less than that used in the HPLC method (98 mL), indicating that HPTLC is an alternative technique for analyzing large numbers of samples.

herbal, quality control, quantitative analysis, HPTLC, comparison of methods 32e

110 171 D. ZHANG (Zhang Depei), H. HUANG (Huang Haibo)*, L. ZHENG (Zheng Lidan) (*Guangzhou Univ. of Chinese Med. & Pharm., Guangdong, Guangzhou 510006, China): (Study of the method for the differentiation of *Achyranthes aspera* and Guangdong *Achyranthes aspera*) (Chinese). Chinese J. of Health Industry (4), 99-101 (2012). Guangdong *Achyranthes aspera*, a herbal TCM drug, is the dried root of *Eupatorium chinense* L. which is used for the treatment of laryngeal diseases. *Achyranthes aspera*, another traditional Chinese herbal crude drug, is the dried root of *Achyranthes aspera* L. It has a similar shape and properties as Guangdong *Achyranthes aspera*, but contains different active constituents and is used for the treatment of colds and fever. The drugs should not be confounded. Development of a method for the differentiation of both drugs. TLC of the extracts of the crude drugs on silica gel with diethyl ether - *n*-hexane 2:1, detection by spraying with 5 % phosphomolybdic acid in ethanol and heating at 105 °C until the zones are visible, evaluation at UV 366 nm. In addition to TLC the drugs are differentiated by

microscopy of the dried drug powders and the cross sections of the fresh samples.

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

110 172 D. ZHANG (Zhang Dihua), X. LI (Li Xiaowei)*, SH. HAN (Han Shuang) (*Section of Chinese materia medica preparation, Taihe Trad. Chinese Med. Hosp., Anhui, Taihe County 236600, China): (Study of the method for the quality control of Shisiwei Dangui Huoxue mixture by thin-layer chromatography) (Chinese). Chinese J. Mod. Drug Appl. 6 (12), 131-132 (2012). Shisiwei Dangui Huoxue mixture is a herbal TCM for promoting blood circulation and relieving pain, and is prescribed clinically to treat blood stasis, local swelling caused by lesion of main and collateral channels of the injured limb after catagma. For quality control, TLC on silica gel 1) for *Angelica sinensis*, with *n*-hexane - ethyl acetate 9:1, detection at UV 366 nm; 2) for *Radix Paeoniae alba*, with chloroform - ethyl acetate - methanol - formic acid 200:25:50:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 1:4 followed by mild heating and evaluation in daylight.

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

110 173 H. ZHANG (Zhang Haiying), SH. ZHAO (Zhao Shengjun), J. XIE (Xie Jie)* (*Affiliated Hosp. of Trad. Chinese Med., Xinjiang Univ. of Med., Xinjiang, Urumuqi 830000, China): (Study of the method for the quality control of Jinshishenyan pills) (Chinese). Chinese J. of Inform. on TCM 17 (12), 45-48 (2010). Jinshishenyan pills are a herbal TCM preparation effective for nourishing kidney, activating spleen-energy, relieving internal heat or fever, and removing blood stasis. For quality control, TLC of the extracts on silica gel for 1) *Lonicera japonica*, with the upper phase of ethyl acetate - formic acid - water 14:5:5, detection at UV 366 nm; 2) for *Cortex Phellodendri chinensis*, developed with benzene - ethyl acetate - methanol - isopropanol - concentrated ammonia 12:6:3:3:1, detection by exposure to ammonia vapors for a few minutes and evaluation at UV 366 nm.

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

110 174 L. ZHANG (Zhang Ling), D. WANG (Wang Dequn)*, RI ZHA (Zha Riwei), ZH. ZONG (Zha Zhen), K. ZHANG (Zhang Ke) (*Anhui Key Lab. of Modern Trad. Chinese Med., Dep. of Pharmacy, Anhui College of Trad. Chinese Med., Anhui, Hefei 230031, China): (Determination of kaempferol in *Rubus idaeus*) (Chinese). J. of Modern Trad. Chinese Med. 14 (2), 12-14 (2012). *Rubus idaeus*, the dried immature fruits of *Rubus chingii* Hu, is a herbal TCM drug specially effective for tonifying the kidney, controlling nocturnal emission, nourishing the liver, and improving eyesight. It is prescribed clinically to cure spermatorrhea, night emission, enuresis, frequent micturition, sexual impotence and premature ejaculation. TLC of the extracts of the crude drug on silica gel with toluene - ethyl acetate - formic acid 9:3:1, detection by spraying with 2 % aluminium chloride in ethanol and viewing under UV 366 nm.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, comparison of methods 32e

110 175 M. ZHANG (Zhang Mingzi)*, SH. JU (Ju Shanji), Y. HAN (Han Yingchen) (*Yanbian Autonomous Prefecture Test Inst. of Food & Pharm., Jilin, Yanbian 133001, China): (The quality assay of Shenqixinjikang compound oral liquid by thin-layer chromatography) (Chinese). J. China Pharm. Univ. 21 (4), 30-31 (2012). Shenqixinjikang compound oral liquid, as a TCM preparation

is specially effective for invigorating the circulation of blood, inducing resuscitation, relieving pain, and is prescribed clinically to cure cardiopalmus and angina pectoris. For quality control, TLC of the extracts of the medicine 1) for *Rhizoma Polygoni Cuspidati*, on silica gel with petroleum ether (60-90 ºC) - formyl acetate - formic acid 15:5:1, detection at UV 366 nm; 2) for *Leguminosae*, on silica gel with *n*-butanol - acetic acid - water 5:1:4, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C for 5 min followed by evaluation under daylight or UV 366 nm; 3) for Rehmannia, on silica gel with chloroform - methanol - water 14:6:1, detection by spraying with 3 % vanillin in ethanol - sulfuric acid 100:1 and heating at 105 °C followed by evaluation under daylight.

pharmaceutical research, traditional medicine, quality control, qualitative identification, comparison of methods 32e

110 176 Q. ZHAO (Zhao Qizhong)*, Q. ZHAO (Zhao Qian), Q. LI (Li Qinfang), Y. LI (Li Yanju) (*Puer Municip. Inst. for Food & Drug, Yuannan, Puer 665000, China): (Study of the method for the quality control of Tengzi tea) (Chinese). Chinese J. Ethnopharm. (12), 41-42 (2011). Tengzi tea, *Ampelopsiscan oniensis* (Hook. et Am.) Planch, is an age-old herbal TCM drug for invigorating the circulation of blood, diminishing inflammation, relieving pains and stoping bleeding, and is prescribed clinically to cure colds and rheumatism, and as adjunctive therapy for hypertension and coronary disease. TLC of the extracts of the crude drugs on polyamide phase with chloroform - ethyl acetate - formic acid 10:10:1, detection under UV 366 nm. In addition microscopic differentiation, and determination of the water content by oven drying. Determination of the extract content by cold-maceration and hot-maceration with water, ethanol or diluted ethanol.

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

110 177 Y. ZHAO (Zhao Yanpu)*, L. FENG (Feng Li), D. LI (Li Dongmei), ZH. ZHAO (Zhao Zhenxia) (*Hebei Provinc. Inst. for Drug Contr., Hebei, Shijiazhuang 050011, China): (Study on the method for the quality control of Niuhuangjingnao tablets) (Chinese). J. China Pharm. Univ. 26 (2), 167-171 (2012). Niuhuangjingnao tablets are a herbal TCM effective specially in clearing heat, removing toxicity, relieving uneasiness of mind and body tranquilization, and are prescribed clinically to cure dizziness, swelling and pain in the throat. In order to perfect the procedure for the quality control of the medicine a method has been presented. TLC of the extracts of the medicine 1) for borneol, on silica gel with toluene - ethyl acetate 10:1, detection by spraying with 5 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C until the zones were visualized, viewing in daylight; 2) for Rheum officinale, on silica gel with the upper phase of petroleum ether (60-90 ºC) - ethyl acetate - formic acid 15:5:1, detection by viewing at UV 366 nm, followed by exposure to ammonia vapors and viewing at UV 366 nm; 3) for the rhizome of Chinese Goldthread, on silica gel with toluene - isopropanol - ethyl acetate - methanol - water 20:5:10:5:1, detection by exposure to ammonia vapors and viewing at UV 366 nm; 4) for *Fructus Forsythiae*, on silica gel with chloroform - methanol - glacial acetic acid 70:10:1, detection by spraying with 10 % sulfuric acid in ethanol and heating at 105 °C until the zones were visualized, viewing in daylight; 5) for honeysuckle, on polyamide phase with ethyl acetate - methanol - formic acid 20:2:3, detection by viewing at UV 366 nm; 6) for the root of Kudzu vine and Cape jasmine, on silica gel with chloroform - methanol - water 28:10:1, detection by viewing at UV 366 nm, followed by spraying with 5 % vanillin in sulfuric acid - ethanol 1:4 and heating at 105 °C until the zones were visualized, viewing in daylight.

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

33. Inorganic substances

110 178 S. BHAWANI*, S. HENA, M. IBRAHIM, O. SULAIMAN, R. HASHIM, K. SAN (*School of Chemical Sciences, Universiti Sains Malaysia, 11800, Pulau Pinang, Malaysia, sabhawani@gmail.com): Identification and separation of lead (II), nickel (II), and cobalt (II) on silica gel 60 F254 high-performance thin-layer chromatographic plates with mixed aqueous sodium dodecyl sulfate-oxalic acid solvent system. J. Planar Chromatogr. 25, 355-357 (2012). HPTLC of lead (II), nickel (II), and cobalt (II) on silica gel with 0.2 M aqueous sodium dodecyl sulphate - 0.08 M oxalic acid 1:9. Lead(II) was detected by spraying with 0.5 % dithizone in carbon tetrachloride, where as nickel (II) and cobalt (II) were detected by spraying with 1.0 % solution of alcoholic (ethanol) dimethyglyoxime. The hR_F values of lead (II), nickel (II), and cobalt (II) were 1, 85 and 49, respectively.

environmental, quality control, qualitative identification 33a

38. Chiral separation

110 179 R. BHUSHAN, J. MARTENS*, Charu AGARWAL, S. DIXIT (*Institute of Pure and Applied Chemistry, CvO Universitaet Oldenburg, 26129 Oldenburg i. O./Germany, juergen.martens@uni-oldenburg.de): Enantioresolution of some beta-blockers and a beta-2-agonist using ligand exchange TLC. J. Planar Chromatogr. 25, 463-467 (2012). HPTLC of the enantiomers of the beta-blockers (bisoprolol, metoprolol, and propranolol) and a beta-2-agonist (salbutamol) on silica gel impregnated with Cu(II) complexes of L-threonine, L-serine, and L-tartaric acid, with different mobile phases. Limits of detection were within the range 0.19-0.26 µg for enantiomers of the selected compounds. L-Ser proved to be a very good ligand for exchange and successful enantioresolution using common mobile phases.

pharmaceutical research, quality control, HPTLC, quantitative analysis, qualitative identification 38

HPTLC-MS analysis using a novel compact single quadrupole mass spectrometer

Frank Porbeck and Dr. Andreas Wiesner, Advion, Harlow, UK, presented a new and very compact single quadrupole mass spectrometer named expressionCMS, which can optionally be used with an ESI or APCI ion source. Via the TLC-MS Interface it was coupled to HPTLC. Its performance was exemplarily shown for isopropylthioxanthone (ITX) and caffeine in cooperation with the working group of Professor Gertrud Morlock, Justus Liebig University Giessen, Germany.

HPTLC analysis

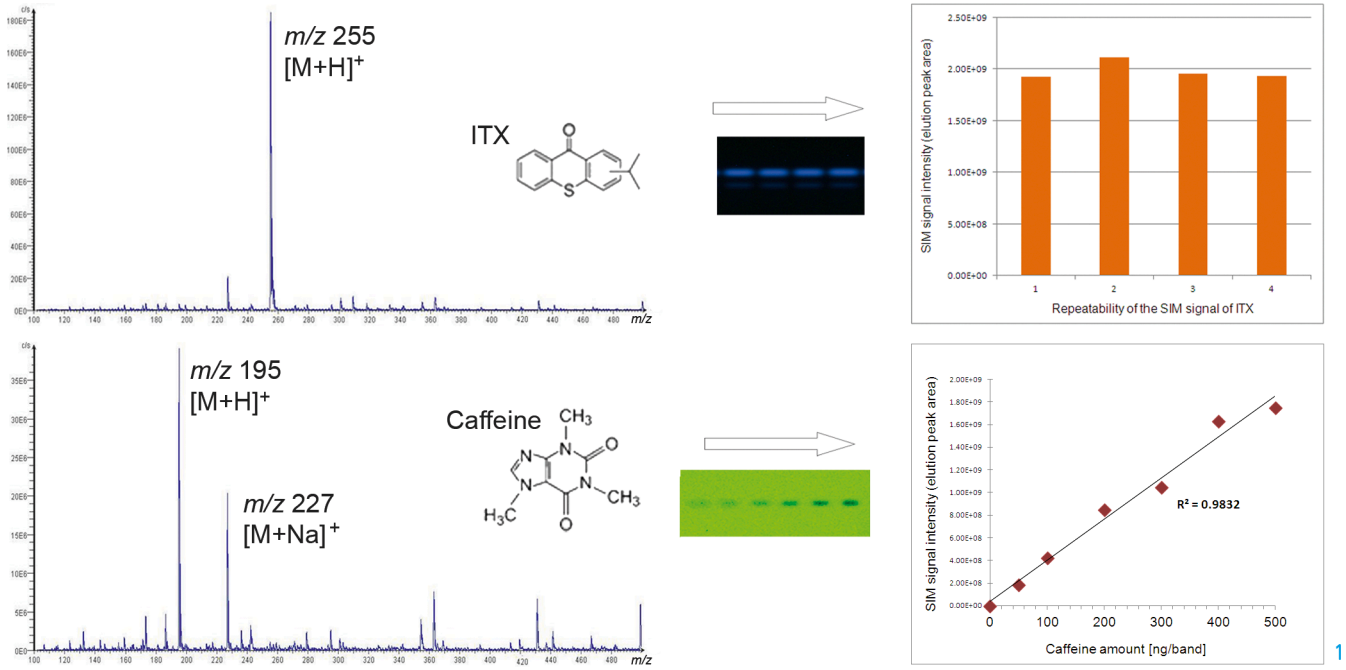
Layer: HPTLC silica gel 60 F_{254} plates (Merck) pre-washed with methanol – water 4:1; application as 6 mm bands with ATS 4; development with toluene – *n*-hexane 4:1 for ITX and acetonitrile containing 2.5 % ammonia for caffeine; evaluation with TLC Scanner 4 by fluorescence measurement at 366/>400 nm (ITX) and absorbance measurement at 275 nm (caffeine) to check the HPTLC performance.

MS analysis

Elution with TLC-MS Interface (circular elution head) with methanol – ammonium formate buffer (10 mM, pH 4) 19:1 at a flow rate of 0.1 mL/min, online connected to the MS, which was run in ESI^+ mode for selected ion monitoring (SIM) and full scan.

Results and Discussion

The analytical response showed a determination coefficient (R^2) of 0.9966 (ITX, 5–100 ng/band) and 0.9832 (caffeine, 50–500 ng/band). The repeatability (*%RSD*) was determined to be 3.9 % (ITX, 50 ng, *n* = 4) and 8.8 % (caffeine, 300 ng, *n* = 6). Based on this good performance data, the compact MS enabled mass-over-charge (*m/z*) signal intensities of HPTLC zones in a concentration-dependent (quantitative) and reliable manner. As a very compact MS, it will help to establish MS in the workflow of TLC/HPTLC laboratories.



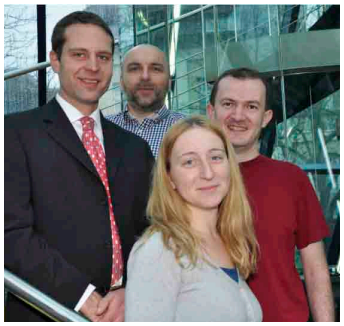
Up: Mass spectrum of ITX and its repeatability in the SIM mode (%RSD = 4 %)
Down: Mass spectrum of caffeine and its analytical response in the SIM mode (R^2 = 0.9832)

Further information is available from:

Prof. Dr. Getrud Morlock, Justus Liebig University Giessen, Germany, Gertrud.Morlock@ernaehrung.uni-giessen.de

*Advion, Kao Hockham Building, Harlow, Essex CM20 2NQ, UK and 10 Brown Rd, Ithaca, NY 14850, USA

Introduction of special HPTLC and TLC plates for coupling with mass spectrometry



Michael Schulz, Bernhard Schubach, Susanne Minarik, Hans Griesinger (from left to right)

Coupling thin-layer chromatography with mass spectrometry and NMR, is becoming increasingly important. To support this development, Merck Millipore has developed special plates, HPTLC plates for coupling with MS and TLC plates for coupling with methods requiring larger amounts of a substance, *e.g.* NMR. There are a number of ways to couple thin-layer chromatography with mass spectrometry [1]. Applications using the TLC-MS Interface, which can be coupled in a straightforward manner to an existing HPLC-MS system, have been described several times in the CBS.

In order to meet the high requirements of mass spectrometry in terms of purity and detectability Merck Millipore has developed HPTLC plates silica gel 60 F_{254} for MS*. These plates are very low in impurities and show less background noise, resulting in a better signal-to-noise ratio during measurements.

The plates are wrapped in aluminum foil to prevent the adsorption of impurities from the ambient laboratory atmosphere as well as additives from packaging materials. The layer thickness is 100 µm, resulting in a higher concentration of the analyte in the eluted volume and thereby better detection. As a part of their quality assurance program, which guarantees a constant high standard of quality, they perform regular procedures for HPTLC plates, including the additional determination of the signal-to-noise ratio for each batch as is described below. The performance of the layers is demonstrated by the separation and identification of insulin and desamido insulin. Other interesting applications in the field of insulin analysis could be for example purity tests and in-process control.

Sample preparation

To obtain desamido insulin, human insulin recombinant, PAN Biotech, 9.66 mg/mL was dissolved in 0.1 % trifluoroacetic acid and kept at room temperature for 24 h [2].

Stationary phase

HPTLC plates silica gel 60 F_{254} MS-grade (1.00934.0001)

Mobile Phase

For insulin application: 2-butanol – pyridine – ammonia (25 %) – water 39:34:10:26

Mass spectrometry

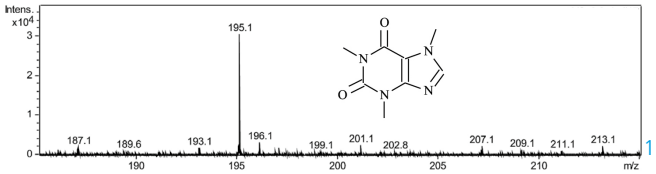
Elution with the TLC-MS Interface and analysis with a Bruker MaXis high resolution Q-TOF mass spectrometer in ESI positve mode. Eluent: acetonitrile (Merck hypergrade for LC-MS) – water (Milli-Q ultrapure water) 19:1

Performance of the HPTLC MS-grade plate

To ensure a consistently high quality for use in mass spectrometry, the signal-to-noise ratio for 5 ng of caffeine is shown as follows. Using a 0.5 µL capillary, 5 ng caffeine in methanol was applied onto the plate. Elution was performed with the TLC-MS Interface directly connected to the ion source of the mass spectrometer. The mass signals, as detected during the elution process, were used to evaluate the mass trace of caffeine. At the maximum of the measured signal the intensity was determined and the signal-to-noise ratio was calculated.

**HPTLC plates silica gel 60 F_{254} MS-grade, 20×10 cm, layer thickness 100 µm (Merck 1.00934.0001, CAMAG 034.0934)*

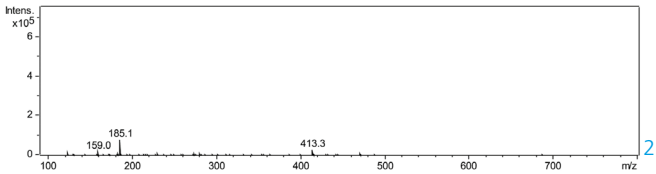
TLC plates silica gel 60 F_{254} MS-grade, 20×20 cm, layer thickness 200 µm (Merck 1.00933.0001, CAMAG 034.0933). These plates are especially suited for TLC-NMR coupling; see also the article on page 2 of this edition.



1

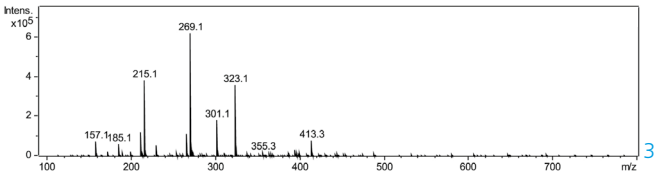
Mass spectrum of 5 ng caffeine at m/z 195.1 $[M+H]^+$

The comparison of the background signals of the new HPTLC MS-grade plate with a conventional HPTLC plate shows the differences in the purity of the layers. By using acid-free eluents for HPTLC-MS analysis, there is no need to pre-wash the plates. However, high purity solvents specified for coupling of liquid chromatography with mass spectrometry should be used.



2

Background spectrum of an HPTLC MS-grade plate with acetonitrile – water 19:1



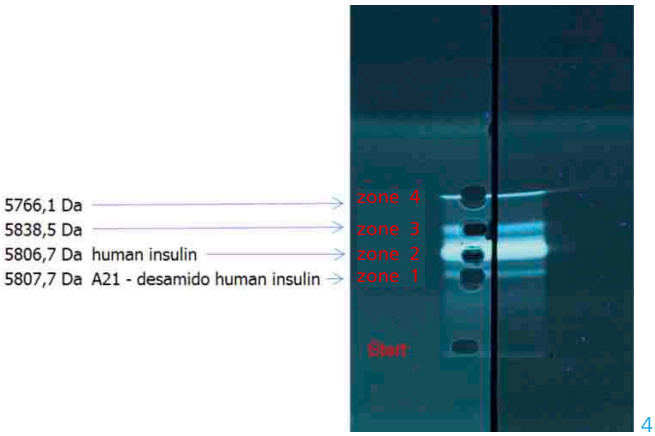
3

Background spectrum of a regular HPTLC plate with acetonitrile – water 19:1

Example: Identification of insulin and desamido insulin

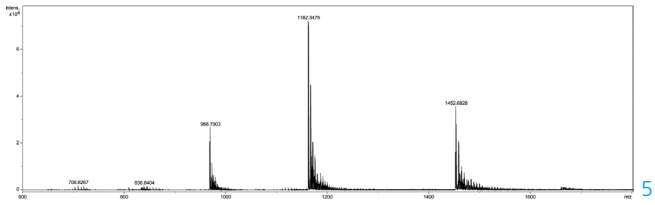
The separation and identification of human insulin and its secondary components play a major role in the production of human insulin. A crucial subcomponent that can occur under acidic conditions is A21 desamido human insulin. This is caused by deamination of the terminal amino acid asparagine [2]. The resulting mass difference between human insulin and A21 desamido human insulin is only 1 Da.

For demonstration, insulin solution was applied as a long band. After development the plate was cut in the middle of the band and one half stained with fluorescamine. By reference to the stained half, the measurement positions for the second half were marked. These positions were eluted with the TLC-MS Interface. For cross-check, the measured half was also stained with fluorescamine and photographed as depicted. Although there is only 1 Da difference between insulin (5806.7 Da) and desamido insulin (5807.7 Da) separation and identification were achieved. Two other bands with the masses 5766.1 and 5838.5 Da are detected.



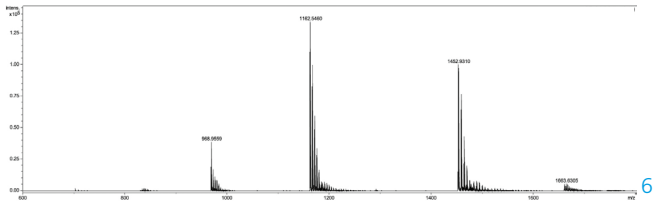
4

Separation of human insulin from desamido human insulin on HPTLC silica gel F_{254} MS-grade



5

Mass spectrum of human insulin



6

Mass spectrum of desamido human insulin

[1] G. Morlock, W. Schwack TrAc 29/10 (2010) 1157

[2] C. Yomota *et al.*, J Chromatogr A 721 (1996) 89

Further information is available from the authors on request.

Contact: Michael Schulz, Merck KGaA, MM-LER-CP, Frankfurter Str. 250, 64293 Darmstadt, michael.schulz@merckgroup.com

Solid phase extraction as clean-up for pesticide residue analysis of tea samples using planar chromatographic developing techniques

Prof. Dr. Wolfgang Schwack and Claudia Oellig

One of the salient research topics of Professor Schwack, University of Hohenheim, is method development in the analysis of pesticide residues. In addition to the development and optimization of methods for the special class of dithiocarbamate fungicides and automatization of the extraction process, the development of efficient clean-up methods in residue analysis is an important issue.

Introduction

In the European Union, maximum residue limits are regulated for over 500 pesticides in food and feed. Sensitive, selective, and robust analytical methods for pesticide residue analysis are required to ensure these limits for consumer protection. Extracts for residue analysis of plant foods contain a significant amount of interfering matrix compounds, which contribute to matrix effects in GC-MS and LC-MS analysis of pesticides, thereby suppressing signal, *i.e.* limits of detection. An efficient clean-up of the extracts is the most reliable way to prevent this matrix effect. For extracts of fruits and vegetables a new clean-up concept, using planar solid phase extraction (high-throughput planar solid phase extraction, HTpSPE) was recently introduced and successfully validated [1, 2]. Tea samples are a particular challenge in residue analysis because they provide very matrix-rich extracts. Besides chlorophyll and polyphenols, especially high amounts of co-extracted caffeine contribute to matrix effects, in both LC-MS and GC-MS analyses. Therefore, the aim of the work presented here is to adapt the successfully developed HTpSPE [1, 2] to tea samples.

In contrast to common clean-up methods like dispersive or cartridge SPE and size exclusion chromatography for multi-residue analysis, HTpSPE is a cost-effective, reliable, and rapid alternative with an efficient clean-up of fruit and vegetable extracts [1]. Even for tea samples, the modified HTpSPE provides very clean sample extracts and reproducibly good recoveries.

Spiking of samples

Dry tea samples were spiked with a mixture of 7 representative pesticides at a level of 0.01, 0.1 and 1 mg/kg: acetamiprid, azoxystrobin, chlorpyrifos, fenarimol, mepanipyrim, penconazole and pirimicarb. As internal standards, tris(1,3-dichloroisopropyl) phosphate (TDCPP) was used for quantitation and Sudan II as visible marker for the target analyte zone.

Sample extraction

Organic black and green tea samples (2 g) were extracted with 10 mL acetonitrile with a homogenizer (27000 rpm, 1 min), followed by centrifugation (4000 g, 5 min) [3]. A pre-cleaning of the extracts was performed by dispersive SPE [4] (2 mL extract, 100 mg weak anion exchanger (primary secondary amine), 200 mg C18, 300 mg $MgSO_4$).

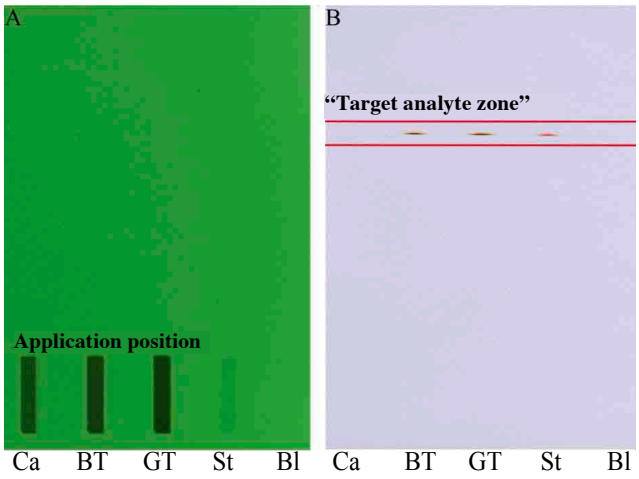
Layer type

TLC plates silica gel 60 F_{254} (Merck), 20 × 20 cm, after prewashing with acetonitrile to 15 cm; the lower 20 × 10 cm were used after cutting with the TLC Plate Cutter.

Sample application

Area application with Automatic TLC Sampler 4 (ATS4), length 3 mm, height 16 mm, track distance 14 mm, distance from the side 23 mm, distance from lower edge 14 mm, application volume 50 µL.

Editor's Note: The value entered for the lower edge (14 mm) corresponds to an absolute distance from the lower edge of about 6 mm, as the 16-mm high area is applied around the entered 14-mm distance value during area application.



1

Application zones before development under 254 nm (A) and target analyte zone after two-fold development under white light (B) - extracts of black (BT) and green (GT) tea; pesticide standard mixture (St), caffeine (Ca), extraction blank (Bl)

Planar solid phase clean-up

In the Automatic Developing Chamber ADC 2 with 5 mL acetonitrile – water 19:1 (v/v), migration distance 85 mm (developing time 10 min), drying time 5 min; 2nd development in the opposite direction with 5 mL acetone – water 7:1 (v/v), migration distance 31 mm (developing time 3 min), drying time 5 min. The plate was equilibrated for 5 min before each development with $MgCl_2$ (33% relative humidity).

Derivatization

With Chromatogram Immersion Device, dipping the plate in primuline reagent (0.2 % in acetone – water 4:1 (v/v)); vertical speed 4 cm/s; immersion time 0 s

Documentation of the clean-up

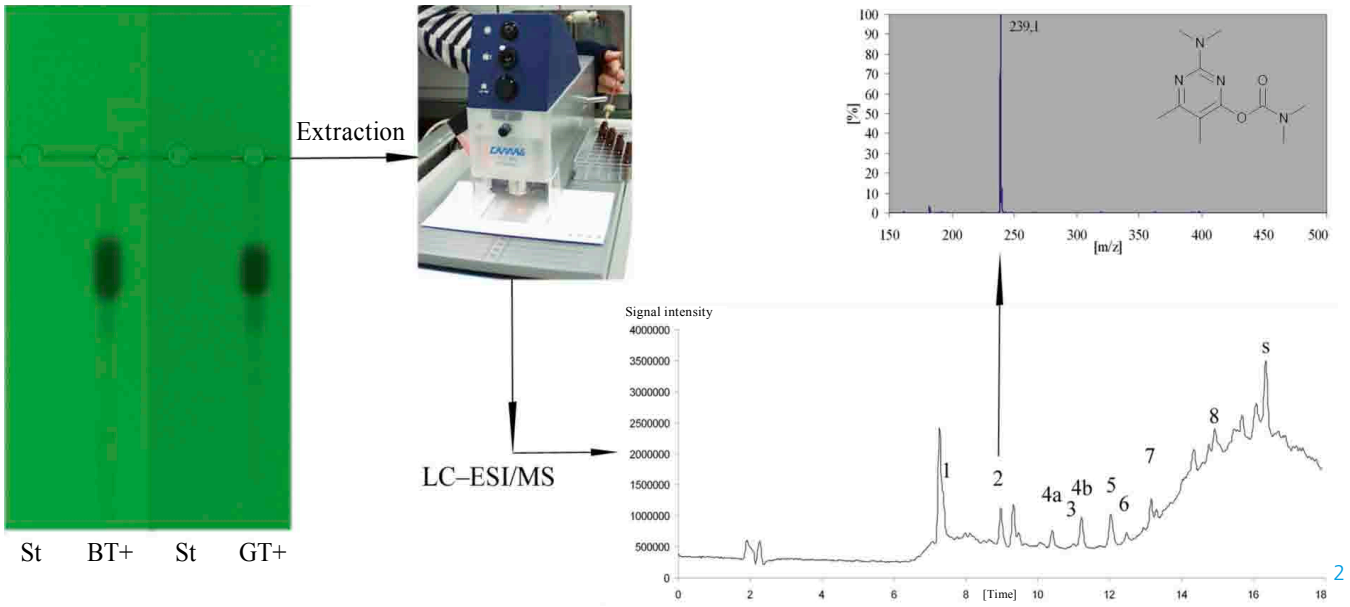
With TLC Visualizer under 254 nm, 366 nm and white light illumination and, after derivatization under 366 nm

TLC-MS elution

Elution of the target analyte zone with TLC-MS Interface for 1 min into autosampler vials with acetonitrile – 10 mM ammonium formate buffer 1:1 (v/v), flow rate 0.2 mL/min

Results and discussion

The planar chromatographic developing technique was used to separate pesticides from co-extracted



2

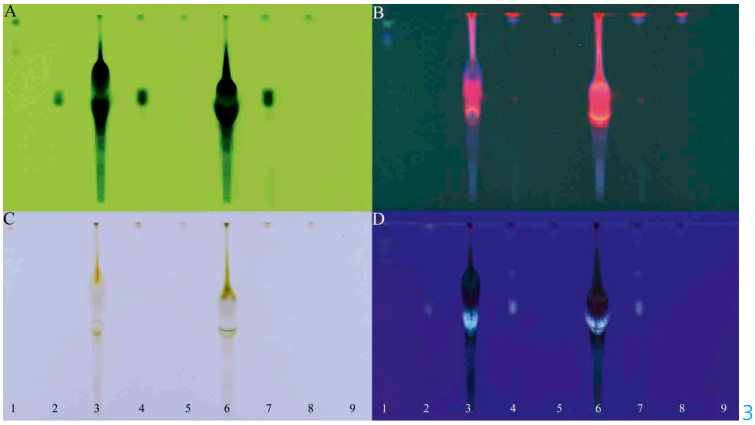
Elution of the target zone by the TLC-MS Interface and following LC-ESI/MS analysis: total ion current chromatogram of a spiked extract of green tea (GT+) and the mass spectrum of pirimicarb (m/z 239.1, $[M+H]^+$) at 9 min (reprinted from [3] with permission)

matrix and to collect the pesticides within one zone (target analyte zone). By experimenting with different layer types as well as adjusting the mobile phase to the specific matrix components in tea (especially caffeine and chlorophyll), a two-fold development on TLC silica gel was most successful. Following this HTpSPE clean-up, the target analyte zone (pesticides) – visible by the addition of Sudan II – could easily be eluted by the TLC-MS Interface into autosampler vials or directly transferred online into the LC-MS system. The extracts were absolutely colorless and free of caffeine.

The new clean-up method could successfully be proven with pesticides from various substance classes for black and green tea samples. Extracts after HTpSPE are much cleaner than extracts obtained by the commonly applied QuEChERS method including dSPE [4]. The successful clean-up could easily be verified by imaging devices.

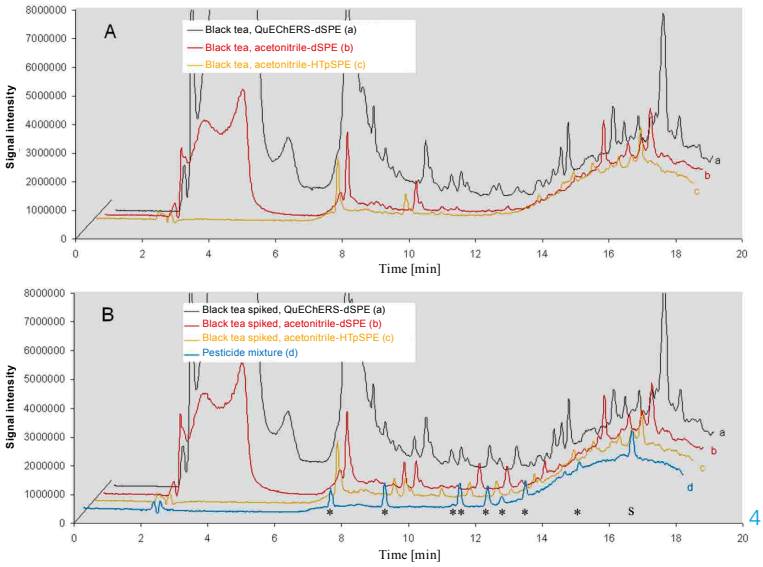
LC-MS chromatograms demonstrated the successful HTpSPE clean-up as well: extracts of spiked tea samples provided almost matrix-free baselines (background signal) without interferences, very similar to pure solvent standards. This is especially apparent with the anticipated caffeine interference, camouflaging or concealing early eluting pesticides, thus causing the heretofore difficulties in GC-MS analysis.

Mean recoveries by LC-MS/MS (72–111 % over all pesticides at two spiking levels, $n = 4$) with excellent relative standard deviations of 0.7–4.7 % confirm the powerful HTpSPE clean-up as reproducible and without loss. Solvent standards could simply be used for calibration instead of the commonly used matrix-matched standards, as HTpPSE extracts were nearly matrix-free and calibration curves were almost identical with solutions of pure standards.



3

Comparison of clean-up for extracts of black (3, 4, 5) and green tea (6, 7, 8) after single development at 254 nm (A), 366 nm (B), white light illumination (C), and at 366 nm after derivatization (D): QuEChERS-dSPE extracts (3, 6), acetonitrile-dSPE extracts before (4, 7) and after HTpSPE (5, 8), pesticide mixture (each approx. 500 ng/zone) (1), caffeine (10 µg/zone) (2) and extraction blank (9) (reprinted from [3] with permission)



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LC-MS total ion current chromatograms of black tea extracts: QuEChERS-dSPE extracts (a), acetonitrile-dSPE extracts before (b) and after HTpSPE (c); (A) blank extracts, (B) extracts spiked with 1 mg/kg pesticide mixture (*), Sudan II (s) (reprinted from [3] with permission)

Spiking level	0.1 mg/kg				0.01 mg/kg			
LC-MS/MS (n = 4)	Recovery [%]		*%RSD*		Recovery [%]		*%RSD*	
Tea type	black	green	black	green	black	green	black	green
Acetamiprid	73	72	4.7	2.3	73	73	3.8	3.4
Azoxystrobin	111	108	3.8	3.7	114	105	3.7	3.2
Chlorpyrifos	87	106	1.8	3.1	101	102	3.7	2.7
Fenarimol	95	94	3.6	2.4	106	94	0.8	1.6
Mepanipyrim	103	99	2.4	2.9	104	100	2.5	1.7
Penconazole	78	88	1.1	3.0	87	87	2.7	0.7
Pirimicarb	95	96	4.3	3.2	102	103	2.1	4.0

On a 20 x 10 cm plate, the clean-up of 12 sample extracts was performed in parallel at one go with a developing time of 30 min. Including sample application the total clean-up takes about 70 min (= 6 min/sample) at the very low solvent consumption of 1 mL/sample. Hence HTpSPE is a cost-effective and rapid alternative to common clean-up techniques for multi-residue analysis of pesticides in food – even for difficult to analyze sample matrices such as tea.

[1] Oellig, C., Schwack, W. J Chromatogr A 1218 (2011) 6540

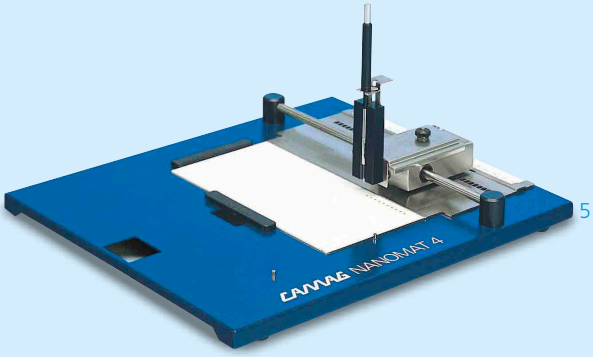
[2] Oellig, C., Schwack, W. CBS 107 (2011) 9

[3] Oellig, C., Schwack, W. J Chromatogr A 1260 (2012) 42

[4] www.quechers.com

Further information is available on request from the authors.

Contact: Claudia Oellig, Institute of Food Chemistry, University of Hohenheim, 70599 Stuttgart, Claudia.Oellig@uni-hohenheim.de



5

CAMAG Nanomat 4

Sample application in the form of spots using capillary pipettes is still routine in TLC/HPTLC. With the Nanomat 4 samples are precisely positioned without damage to the layer. The actual sample dosage is performed with a fixed volume capillary pipette precisely guided by the Universal Capillary Holder, so that the separation zones can be densitometrically scanned according to a programmed scanning pattern.

Der Nanomat is suitable for

- Conventional TLC plates including self coated plates up to 20 x 20 cm
- HPTLC plates 10 x 10 and 20 x 10 cm
- TLC and HPTLC sheets up to 20 x 20 cm


6

Capillary Dispenser

The capillary pipettes are loaded into the dispenser from rechargeable magazines. Capillaries of 0.5, 1.0, 2.0 and 5.0 µL are available. With the Universal Capillary Holder a capillary pipette is taken up from the dispenser, filled with sample solution and placed against the magnetized application head of the Nanomat. Upon manual lowering the head, the capillary touches the layer with a constant pressure transferring the solution to the layer by capillary forces.

For further information viz. catalog "Instrumental Thin-Layer Chromatography 2013" and **www.camag.com/nanomat**

Sample application with CAMAG Linomat 5

1

Precision of the applied volume, exact positioning and compactness of the application zones are decisive for the quality of the analysis.

With the Linomat samples are sprayed onto TLC/HPTLC plates in the form of bands with nitrogen or compressed air. Sample application is automatic, only changing the sample (filling, inserting and rinsing the syringe) is manual. The Linomat is suitable for routine use.

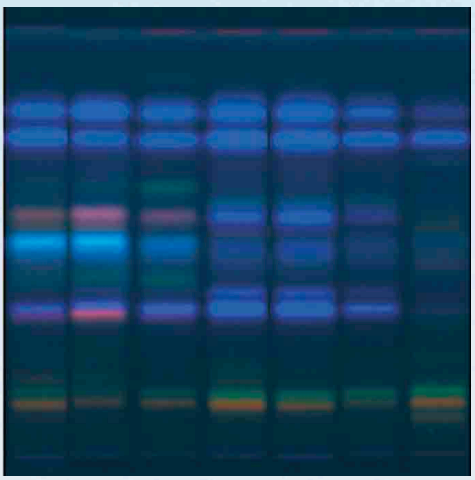
Starting zones sprayed on as narrow bands ensure the highest resolution attainable with the planar chromatographic system selected.

Since the sample is uniformly distributed over the length of the band, densitometric evaluation can be performed by aliquot scanning, *i.e.* only the center portion of the zone is measured, ensuring highest precision.

The Linomat can be operated by software control (winCATS or VisionCATS) or in stand-alone mode. It is a cost attractive alternative to the CAMAG Automatic TLC Sampler ATS 4 in cases where sample throughput does not justify a fully automatic solution.

Advantages of sample application with the Linomat 5

- With the spray-on technique larger sample volumes can be applied than by contact transfer.
- For multi-level calibration different volumes of one standard solution can be applied without affecting precision.
- Also spiking of samples is possible, simply by over-spraying.
- Over-spraying pre-chromatographic derivatization reagent can be done as well with suitable reagents.


2

HPTLC fingerprint chromatogram of flavonoids from green tea

Further information can be found in the special brochure "CAMAG Linomat 5" or under **www.camag.com/linomat5**

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