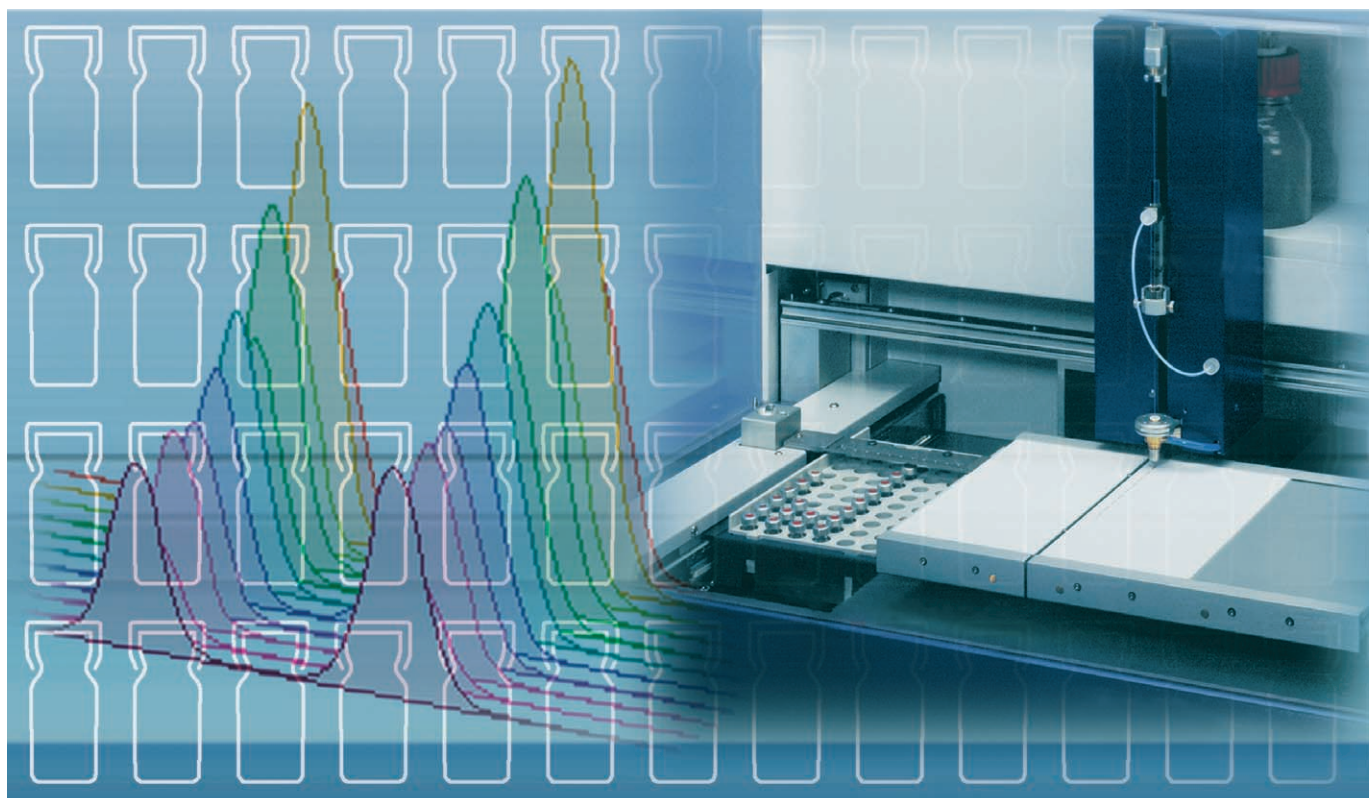


CBS

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**Planar Chromatography –
high sample throughput and unsurpassed
flexibility**

CAMAG

93

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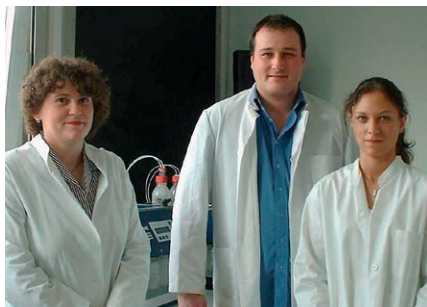


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Planar Chromatography in Practice

Quantification of signaling ceramides in primary keratinocytes



▲ From left to right:
Dr. Susanne Grether-Beck, Andreas Timmer,
Daniela Brammertz



▲ For high throughput analysis of ceramides and lipids in cell culture samples four AMD2 systems are connected via a serial port to a personal computer.

The main objective of the Environmental Health Research Institute is the analysis and evaluation of risks to human health that result from environmental factors in order to improve health care and to develop new preventive and therapeutic strategies.

The work of the Cell Biology group of Dr. Grether-Beck* focuses on the analysis of the effects of physiological doses of ultraviolet radiation on human skin cells with special regard to the induction of gene expression. Within the established signaling model, lipids and especially signaling ceramides play an essential role as secondary messengers. The latter differ from the so-called barrier ceramides, characteristic for keratinocytes within the Stratum corneum, with respect to structure and function.

Introduction

The formation of signaling ceramides with the common structural element N-acyl-D-erythro-sphingosine (acyl = C₁₂ to C₂₄) was studied for a long time using radioactive labeling techniques where the cells of interest were cultured in the presence of ¹⁴C or ³H labeled fatty acids e.g. palmitic acid (C₁₆). Quantification of isolated lipids was performed using conventional developing chambers for separation and by scraping off the spots identified by unspecific iodine vapor staining in the presence of a commercially available ceramide standard.

In view of safety of laboratory staff and due to the maintenance costs of an isotope laboratory (costs of disposal for radio-labeled solvents and for liquid scintillation counting) the presented alternative, non-radioactive quantification method is a cheaper and safer choice. The parallel chromatographic separation for 17 samples/standards lasts 42 min affording a total solvent consumption of 54 mL.

Sample preparation

Primary keratinocytes are scraped off the frozen culture and washed in phosphate buffered saline by centrifugation. Samples which have been normalized to 500 µg protein are extracted according to Folch [1] and subjected to mild alkaline hydrolysis [2,3,4].

Layer

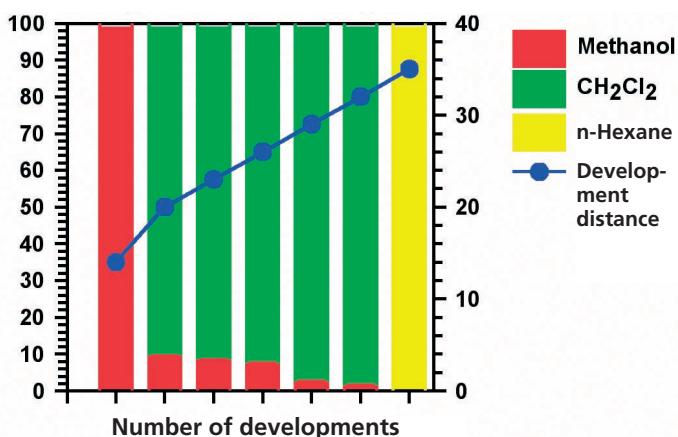
HPTLC plates silica gel 60 F_{254s} (Merck), 20 x 10 cm, pre-washed with 2-propanol (by immersion for at least 60 min) and dried at 120 °C for 20 min

Sample application

Bandwise with Linomat, 17 bands, band length 8 mm, application volumes of the standard mixture 1, 5, 10, 15, 20 µL and of samples 10 µL, distance from lower edge of plate 10 mm, distance from left edge 20 mm, track distance 10 mm

Chromatography

In AMD2 system with a 7 step gradient from methanol over dichloromethane to n-hexane using N₂ as inert gas, max. migration distance 35 mm, consumption of solvents is 54 mL per gradient (3 mL per determination), gradient time is 42 min (incl. 2 min drying time each), consequently chromatographic separation needs 2.5 min per determination for parallel analysis of 17 determinations on one plate



▲ AMD2 gradient based on 7 development steps from methanol over dichloromethane to n-hexane for separation of ceramides



CAMAG AMD System

(Automated Multiple Development)

The working group of Dr. Grether-Beck is employing 4 AMD2 systems in parallel to cope with the high sample throughput. That means chromatography of up to 68 determinations can be performed simultaneously in about 45 min.

AMD is used when the desired resolution is unattainable over the available separation distance by one step isocratic development. This is often the case for complex samples with high or differing matrix content, mixtures of components with a wide polarity range, or for multi-component mixtures.

For separation of samples with components covering a wide polarity range, a universal gradient reaching from high elution strength to very low elution strength is employed. The combination of multiple and gradient development leads to a focusing effect of the zones and peak sharpness is improved. This often leads to an increased sensitivity of detection which is also advantageous in this application for the quantification of signaling ceramides.

Post chromatographic derivatization

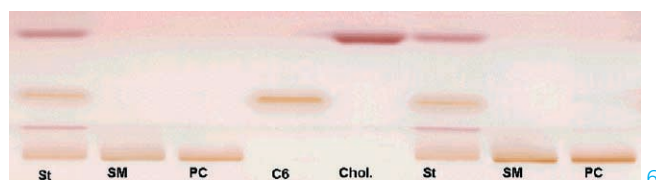
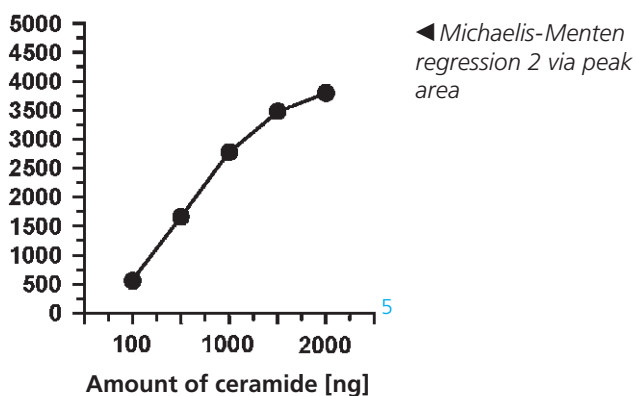
With Chromatogram Immersion Device the plate is dipped into manganese chloride reagent for 1 s. Subsequently, the plate is dried for 20 min at 120 °C [2]. Phospholipids, sphingolipids and cholesterol appear as yellow to brownish-violet zones on a white background.

Densitometric evaluation

TLC Scanner 3 with CATS software, absorption measurement at 550 nm, Michaelis-Menten regression 2 via peak area

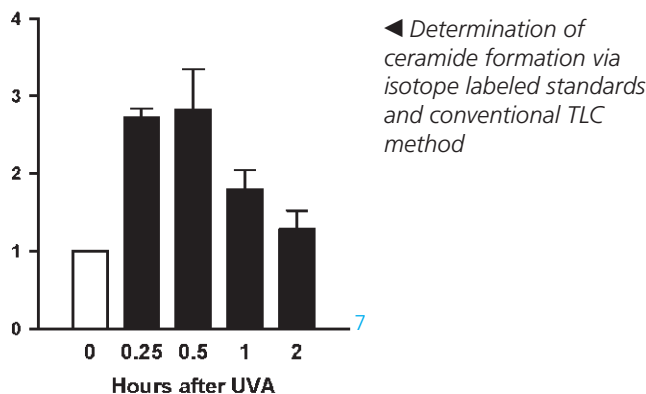
Results and discussion

Signaling ceramides are clearly separated from the other lipids contained within cellular lipid extracts e.g. sphingomyelin, phosphatidylcholine or cholesterol. Ceramides can be identified down to the picogram range. The results obtained are reproducible. The sensitivity of HPTLC-AMD2 determination even exceeds the sensitivity attained by in vivo labeling with ^{14}C or ^3H marked palmitic acid and conventional TLC method.

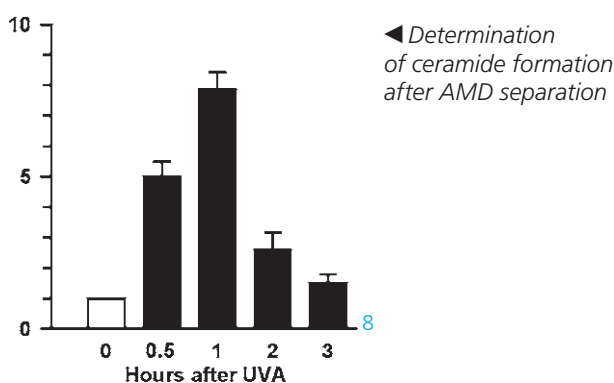


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Formation of signaling ceramides in primary keratinocytes after in vivo labeling using ^3H palmitic acid: The data are obtained as counts per minute and per 500 μg protein. Ceramide content of non-irradiated cells was set equal to one. The ceramide formation is given as n-fold induction [3].



Formation of signaling ceramides in primary keratinocytes: The separation of the lipids was done with extracts based on 500 μg protein using the AMD 2 system.



[1] Folch J, Lees M, Sloane-Stanley GH (1957), J Biol Chem 226: 497–509

[2] Grether-Beck S, Bonizzi G, Felsner I, Timmer A, Schmitt H, Sies H, Piette J, Krutmann J (2000) EMBO J 19: 5793–5800

[3] Grether-Beck S, Felsner I, Brenden H, Krutmann J (2003) J Biol Chem 278: 47498–47507

[4] Schieke S, von Montfort C, Buchczyk DP, Timmer A, Grether-Beck S, Krutmann J, Holbrook NJ, Klotz L-O (2004) Free Radic Res 38: 729–737

*A. Timmer, D. Brammertz, Dr. S. Grether-Beck, Environmental Health Research Institute of Heinrich-Heine-Universität Düsseldorf gGmbH, Auf'm Hennekamp 50, D-40225 Düsseldorf, Grether-Beck@uni-duesseldorf.de, Andreas.Timmer@ish.de, Tel. +49 -211 -3389303

From visual evaluation according to the pharmacopoeia to exact analytical results



▲ The analytical development group of STADA R&D GmbH: front row from left: Silke Nannen, Claudia Appel, Jana Kulle, Kerstin Armbrust, Brunhilde Zlattinger; back row from left Herbert Hofmann, Mike Kottbus, Dirk Willerscheid, Stefanie Rumpel.

The working group Analytical Development of the STADA R&D GmbH company of Bad Vilbel* is working on optimization and validation of analytical methods for assay of active pharmaceutical ingredients and determination of impurities. In special cases also analytical methods for drug release are developed. Another focus of the group is stability studies on finished products and analyses of raw materials.



▲ Kerstin Armbrust is quantifying N,N-diethylethane-1,2-diamine in metoclopramide finished products at the HPTLC working place

Introduction

The drug metoclopramide hydrochloride is used by the STADA R&D GmbH in liquid and solid pharmaceutical formulations. According to the monograph of Ph. Eur. 4, 2002, the impurity N,N-diethylethane-1,2-diamine is tested by conventional thin-layer chromatography using visual evaluation after post chromatographic derivatization. The TLC method based on the monograph was developed further and validated for exact quantitative planar chromatographic determination of N,N-diethylethane-1,2-diamine in metoclopramide finished products. The goals of the investigation were optimizing of chromatographic conditions and making analysis more objective by replacing visual evaluation of the impurity with an exact quantitative method applicable to metoclopramide containing solutions and tablets.

Planar chromatography (TLC/HPTLC) is established in almost all pharmacopoeias of the world for impurity checks on drugs. The following procedure is an example for a precise determination of impurities (related substances), which do not have (sufficient) UV absorbance and must be derivatized.

Sample preparation

Prior to application onto the HPTLC plate the aqueous sample solution is filtered through a membrane filter (PTFE, 0.45 μm). Aside of the drug metoclopramide it contains methyl- and propylhydroxybenzoate as preservatives.

Standard solutions

40 mg N,N-diethylethane-1,2-diamine are dissolved in water to a volume of 200 mL. 2 mL of this stock solution are diluted with water to 100, 20 and 10 mL respectively. The standard solutions correspond to 0.1%, 0.5% and 1% N,N-diethylethane-1,2-diamine, with respect to the metoclopramide concentration in test solution.

Layer

HPTLC plates silica gel 60 F₂₅₄ (Merck), 20 x 10 cm

Sample application

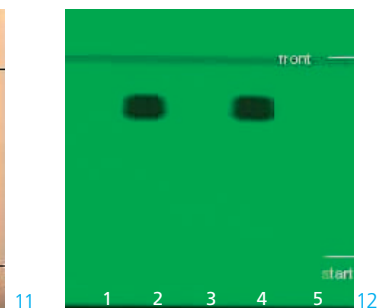
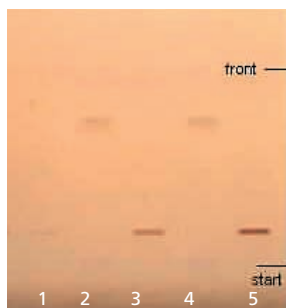
Bandwise with the CAMAG Automatic TLC Sampler 4, 18 tracks in data pair technique, band length 5 mm, track distance 8.5 mm, distance from the side 15 mm, distance from lower edge 10 mm, application volume 2.5 µL. Following the application the plate is dried for 10 min at room temperature.

Chromatography

In saturated CAMAG Twin Trough Chamber with concentrated ammonia solution (32 % w/w) – methanol – dichloromethane 3:15:80, migration distance 40 mm from lower edge. After chromatography the plate is dried in warm air for 5 min.

Post chromatographic derivatization

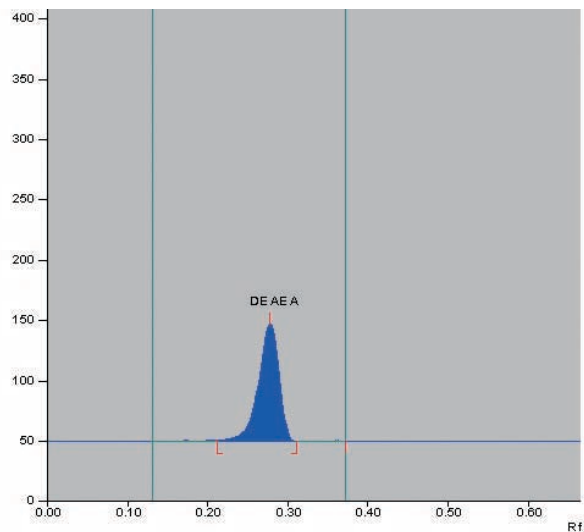
With CAMAG Chromatogram Immersion Device the plate is dipped into 0.2% ethanolic ninhydrin solution for 1 s, then dried in the oven for 5 min at 120 °C. N,N-diethylethane-1,2-diamine gives at hR_F-value of 28 brown-red zones on pink background. The preservatives do not react and therefore don't appear in the chromatogram.



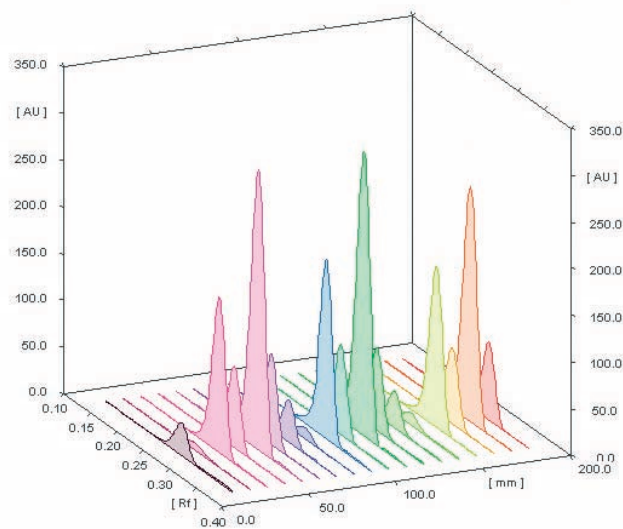
▲ Image obtained with CAMAG documentation system VideoStore at 254 nm prior to derivatization and in daylight after derivatization; track 1, 3, 5: standard solution 0.1 %, 0.5 %, and 1 %, respectively, track 2, 4: sample solutions

Densitometric evaluation

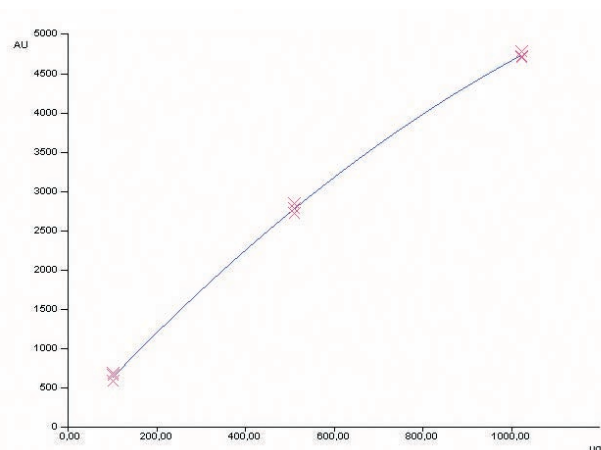
CAMAG TLC Scanner 3 with winCATS Software, absorption measurement with tungsten lamp at 480 nm, evaluation of peak area with polynomial regression.



▲ Sample solution metoclopramide spiked with 0.2% N,N-diethylethane-1,2-diamine (Peak DEAEA)



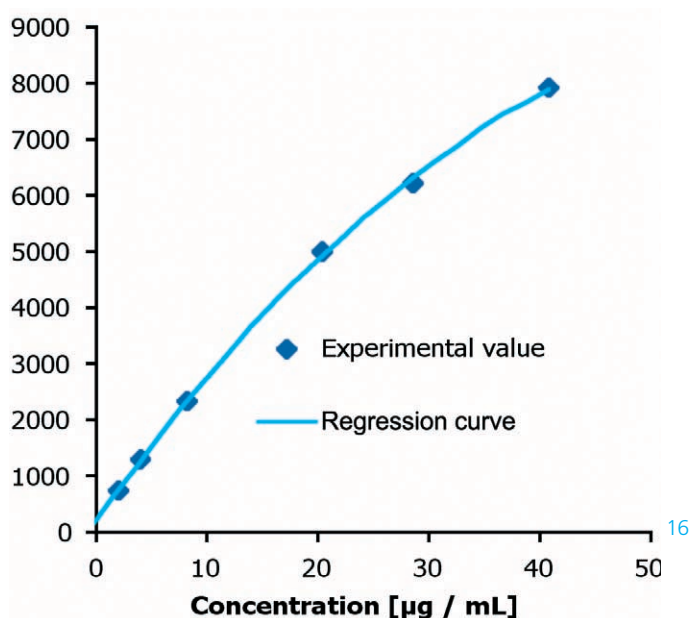
▲ 3D view of 18 tracks



▲ Polynomial calibration function for N,N-diethylethane-1,2-diamine with three standard levels ($r = 0,99969$, $sdv = 1,88\%$): the amount of impurity was smaller than the lowest calibration level ($< 0.1\%$)

Results and discussion

By moving from (conventional) TLC to HPTLC layers and optimizing the mobile phase composition a method for exact quantitation of N,N-diethylethane-1,2-diamine was developed. The method is validated. Intermediate precision is 1.65%. The recovery for 0.2–1.0% impurity is 100.5%. The correlation coefficient of the polynomial calibration function is 0.9995 with a residual standard deviation of 2.19%. Limit of quantitation of the impurity is at 0.05% N,N-diethylethane-1,2-diamine with respect to the concentration of metoclopramide.



▲ Validation: polynomial calibration function for N,N-diethylethane-1,2-diamine via peak area with a correlation coefficient of 0.9995 and a residual standard deviation of 2.19%

Further information and help – also for solid drug formulations – is available on request from the authors.

*Herbert Hofmann, STADA R&D GmbH, Analytical Development, Sladastrasse 2, D-61118 Bad Vilbel, Tel. +49-6101-603-0, herbert.hofmann@stada.de



CAMAG Automatic TLC Sampler 4 (ATS 4)

In several articles of this issue the ATS 4 is mentioned for fully automatic application of substances onto the HPTLC plate (see also pages 10 and 15). Samples can be applied as bands using the spray-on technique or as spots by contact transfer. Sample application in form of rectangles allows the application of large volumes.

The ATS 4 is easy to operate and controlled by winCATS. It applies substances under a cover to protect the object from environmental factors. It features a self-adjusting object support to accommodate objects of various thickness. The spray nozzle does not require adjustments. Aside of the standard rack also a special rack holding 96-well-plates can be installed. Automatic sample application increases precision and robustness during routine analysis in a GLP/GMP environment.

ATS 4 is available as option with a heated spray nozzle, especially useful for the application of aqueous solutions.

New head of Research and Development



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▲ Dr. Matthias Loppacher

In CBS 91 we reported that Mr. Peter Jänchen was promoted to the position of CEO after serving 10 years as manager of research and development. The new head of the R&D team is Dr. Matthias Loppacher who took over in June of 2003.



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▲ Members of the CAMAG Research and Development team (from left): Christoph Fankhauser, Hans-Ruedi Brugger, Klaus Schetter, Dr. Matthias Loppacher, Beatrice Kaeser, Andreas Durandi und Walter Reichenback

Dr. Loppacher studied Physics at the University of Basle and got his doctorate degree in connection with an international project on building a device for measurement of electron polarization (Møller Polarimetry) in Virginia, USA. After a short time as post doc at the Nuclear Research Institute at Newport News (VA, USA), which was sponsored by the University of Basle, he ventured into industry. As product manager of an internationally active company he optimized the know-how transfer between marketing and development, represented his company in a Standards Committee for High Voltage Testing and Measuring Technique, was scientifically active and successfully managed development projects. He gained experience in the area of strategies and future orientation while developing in collaboration with the ETH-Centre for Enterprise Sciences in Zurich a concept for Technology Management for a company of 600 people. As a result of an internship at a patent law firm Dr. Loppacher also has knowledge in patent law.

All of his skills will be utilized by CAMAG. We see his strengths particularly in interdisciplinary projects, for which diverse requests from mechanics, electronics, software and chemistry are the key for success. Dr. Loppacher brings in a broad knowledge and it is his goal to become actively involved in shaping the future of our company. Experienced colleagues from mechanics and electronics design as well as from the laboratory support him to meet the future needs of the TLC market with new ideas and developments.

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PLANAR CHROMATOGRAPHY**

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Liebe Freunde

In China weht ein frischer Wind für die Planar-Chromatographie. Ein mobiles Drogen-Überwachungs-Programm wurde von den Behörden ins Leben gerufen, um damit den sicheren Gebrauch von Arzneimitteln auch in ländlichen Gebieten zu fördern und die Einhaltung der gesetzlichen Bestimmungen zu sichern (S. 13). In dieses Programm ist die Planar-Chromatographie als fester Bestandteil integriert. Sie wird als schnelle Methode vor Ort in 344 mobilen Überwachungs-labors eingesetzt.

In der letzten CBS-Ausgabe wurde unter anderem die Firma Altana Pharma AG vorgestellt. Auch in einem anderen bedeutenden Pharma-Unternehmen, der Firma Stada R&D GmbH, wird die Planar-Chromatographie als schnelle und spezifische Methode eingesetzt (S. 5–7). Die von dem Stada-Team aktualisierte, vorgestellte Methode zeigt den dringlich notwendigen Zeitewandel weg von antiken DC-Methoden hin zur modernen Planar-Chromatographie.

Vier AMD-Kammern werden parallel in der Arbeitsgruppe von Frau Dr. Grether-Beck, Institut für Umweltmedizinische Forschung an der Heinrich-Heine-Universität Düsseldorf gGmbH, zur Lipid-analytik eingesetzt (S. 2–4).

Die simultane Trennung von 15 Doppelbestimmungen innerhalb von nur 15 min besticht bei der Anwendung von Dr. Jamshidi, Iran Polymer and Petrochemical Institute, Teheran (S. 10–12). Sie haben es bereits gemerkt, es geht um hohen Probendurchsatz als Themenschwerpunkt in dieser CBS-Ausgabe.

In diesem Jahr fand ein reger planar-chromatographischer Meinungsaustausch statt. Einige europäische Events finden Sie auf Seite 9 – sind Sie im nächsten Jahr dabei?

Herzlichst Ihre

Gerda Morlock

Gerda Morlock

Dear friends

A fresh wind for TLC is blowing in China. A "Mobile Drug Testing Program" was created by the authorities to ensure safe use of drugs also in rural areas of the country and to enforce the corresponding law (p. 13). Planar chromatography is integral part of this program. The technique will be employed on site by 344 mobile laboratories.



In the last CBS issue we featured Altana Pharma AG. Also another important pharmaceutical company, Stada R&D GmbH, utilizes planar chromatography as a rapid and specific method (p. 5–7). The updated method presented by the Stada team illustrates the necessity of changing from antique TLC to modern Planar Chromatography.

Four AMD chambers are used in parallel for analysis of lipids by the team of Dr. Grether-Beck of the Institute of Environmental-Health Research at Heinrich Heine University Düsseldorf.

Simultaneous determination of 15 samples in duplicate within only 15 min stands out in the application by Dr. Jamshidi, Iran Polymer and Petrochemical Institute, Teheran (p.10–12). You have already noticed, the thematic focus of this CBS issue is high sample throughput.

This year a broad exchange on Planar Chromatography took place. Some of the European events are mentioned on page 9. Will you participate next year?

Sincerely yours,

Gerda Morlock

Gerda Morlock

CAMAG

**SEPTEMBER
2004**

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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
 - f) Clinico-chemical applications and profiling body fluids
 - g) Herbal and traditional medicines
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

2. Fundamentals, theory and general

- 93 001 D. JÄNCHEN* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jaenchen@camag.com): Quantitative TLC/HPTLC in the bulk drug industry. *CBS* **81**, 10-12 (1998). A number of applications of HPTLC in the bulk drug industry are presented: process optimization, fermentation process monitoring, impurity profile, vessel residue certification and bulk drug analysis. The technique is advantageous when a large number of similar samples are to be analyzed and time and cost considerations are of importance.
Pharmaceutical research, quantitative analysis, bulk drug 2a, 32a
- 93 031 B. RENGER et al., see section 11c
- 93 003 M. SAJEWICZ, A. PIENIAK, R. PIETKA, K. KACZMARSKI, T. KOWALSKA* (*Inst. Chem., Silesian Univ., 9, Szkolna Street, 40-006 Katowice, Poland): Densitometric acquisition of concentration profiles in planar chromatography and its possible shortcomings, Part 1: 4-Phenylbutyric acid as analyte. *Acta Chromatographica* **14**, 5-15 (2004). Investigation of lateral interactions of 4-phenylbutyric acid in mild planar chromatographic systems comprising low-activity stationary phases, such as microcrystalline cellulose or chromatography paper, and low-polarity decalin as mobile phase. Acquisition of densitograms of the bands of interest was carried out in order to judge the presence or absence of lateral interactions of the analyte in the chromatographic system by comparison of the shape of the concentration profile. Discussion of the observation that the concentration profile of 4-phenylbutyric acid on cellulose powder could be easily measured on a freshly developed chromatogram only after drying whereas on chromatography paper developed at room temperature it was hardly detectable, and became fully shaped only two days later. Presentation of a tentative explanation on this phenomenon.
Traditional medicine, densitometric acquisition, 4-phenylbutyric acid 2a, 11a
- 93 007 U. WIPPO et al., see section 3d
- 93 002 A. PIENIAK, K. KACZMARSKI, M. SAJEWICZ, W. ZAPALA, A. GOLEBIOWSKA, R. TOMOLA, T. KOWALSKA* (*Inst. Chem., Silesian Univ., 9, Szkolna Street, 40-006 Katowice, Poland): A densitometric study of co-elution in thin-layer chromatography, and its physicochemical modeling. *Acta Chromatographica* **14**, 16-36 (2004). Study on co-elution by using two binary mixtures which can form mixed associative structures by hydrogen bonding: 1) a carboxylic acid (2-phenylbutyric acid) and a ketone (benzophenone), and 2) an aliphatic alcohol (5-phenylpentanol, and the same ketone (benzophenone). By using mild chromatographic conditions and working in the non-linear region of the adsorption isotherm, the co-elution of the two analytes from each pair in the form of a single chromatographic band was demonstrated, three different physicochemical explanations of their densitometrically measured concentration profiles were suggested, and semi-quantitative simulations of these profiles were performed. Discussion and comparison of the experimental results obtained by planar chromatography and HPLC using fully analogous working conditions.
Co-elution, binary mixtures 2c

3. General techniques

- 93 005 H. HAUCK*, M. SCHULZ, C. LORENZ, A. KOCH (*Merck KGaA, A&R/R&D Synthesis and Derivatization, Frankfurter Str. 250, D-64293 Darmstadt, Germany, Heinz-

Emil.Hauck@merck.de): Does the use of spherical adsorbents for HPTLC pay off? CBS **92**, 5-7 (2004). Comparison of irregular HPTLC and spherical lichrospher phases relating to development time, retention, selectivity, separation power, and sensitivity of detection.

Quantitative analysis, spherical adsorbent

3b

- 93 007 U. WIPPO* (*Federal Institute for Drugs and Medical Devices, Kurt-Georg-Kiesinger-Allee 3, D-53175 Bonn, Germany, wippo@bfarm.de): Conversion of a gradient from an AMD1 to an AMD2 system. CBS **92**, 10-12 (2004). Conversion of AMD1 gradients into AMD2 gradients with a mathematical system using Excel 97.

AMD, mathematical system, conversion of gradients

3d, 2a

- 93 004 R. BARTZATT* (*Medicinal Chem. Lab., Dept. of Chem., Durham Sci. Center, College of Arts and Sci., Univ. of Nebraska, 6001 Dodge Street, Omaha, NE 68182 USA; bartzatt@mail.uno.maha.edu): Dansylation of aromatic, aliphatic, and medicinal carboxylic acid compounds in 1 M Na₂CO₃ buffer. Anal. Chim. Acta **488**, 203-209 (2003). Dansylation of aromatic carboxyl compounds (i.e. aspirin), aromatic primary amines, and aliphatic carboxyl compounds in 1 M Na₂CO₃ buffer at pH 11. TLC on silica gel of fluorescent labeled analytes using methylene chloride, ethyl acetate, acetone, or desired mixture of the solvents. Methylene chloride was superior to ethyl acetate or acetone. Fluorescent analytes were observed under UV lamp. Limits of detection for dansylated carboxyl compounds was 1-5 µg.

Carboxylic acid, dansyl chloride, prechromatographic derivatization, fluorescence detection

3e, 11a, 17a

- 93 006 W. KREISS,* G. EBERZ, C. WEISEMANN (*Central Research/Structural Research, Bayer AG, D-51368 Leverkusen, Germany): Bioluminescence detection for planar chromatography. CBS **88**, 12-13 (2002). TLC of waste water samples or spinach extract on silica gel. Selective detection of bioactive compounds by dipping in suspension of luminescent microorganisms (*Vibrio fischeri*). Visual detection of substance zones by reduced luminescence.

Herbal, food analysis, environmental, toxicology, qualitative identification, bioluminescence

3e, 29, 37a

4. Special techniques

- 93 008 D. JÄNCHEN* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jaenchen@camag.com): CAMAG DuoChrom, interface between HPLC and Planar Chromatography. CBS **83**, 2-3 (1999). HPLC of carotenoids on a RP-30 column with a gradient of 90 min from methanol - MTBE - water 80:15:2 to methanol - MTBE - water 4:45:1. HPTLC-AMD on silica gel with a 30-step gradient from methanol containing ammonia via dichloromethane to n-pentane.

AMD, comparison of methods, HPLC, DuoChrom, carotenoids, online coupling of HPLC with HPTLC

4d, 15a, 30b

- 93 018 K. K. ATINDEHOU et al., see section 8a

- 93 091 S. CAI et al., see section 32c

- 93 101 L. DONG et al, see section 32c

- 93 108 N. HUANG (Huang Nojia) et al., see section 32c

- 93 119 X. LIU (Liu Xin) et al., see section 32c
- 93 120 H LU (Lu Hua) et al., see section 32c
- 93 134 A. SIEVERS et al., see section 32e
- 93 138 H. TIAN (Tian Hong) et al., see section 32c
- 93 143 Y. WANG (Wang Yuan) et al., see section 32c
- 93 055 W. XIA (Xia Wei) et al., see section 15a
- 93 147 Y. XIA (Xia Yufeng) et al., see section 32c
- 93 150 ZH. XIE (Xie Zhimin) et al., see section 32c
- 93 152 L. XU (Xu Liting) et al., see section 32c
- 93 153 R. XU (Xu Renliu) et al., see section 32c
- 93 154 A. YANG (Yang Andong) et al., see section 32c
- 93 058 Y. YANG (Yang Yun) et al., see section 17a
- 93 158 X. YANG (Yang Xixong) et al., see section 32c
- 93 075 X. ZHANG (Zhang Xuelan) et al., see section 22
- 93 164 Y. ZHENG (Zheng Yi) et al., see section 32c

5. Hydrocarbons and halogen derivatives

- 93 010 D. JÄNCHEN* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jaenchen@camag.com): Analysis of polycyclic aromatic hydrocarbons. CBS **83**, 6-7 (1999). HPTLC-AMD of PAHs in liver and lung of animals exposed to contaminated soil on RP-18 with a 14-step gradient from acetonitrile to methanol - water 9:1. Quantification by fluorescence measurement at 313/>400 nm and 254/>400 nm, respectively. Strict linearity ($r^2 > 0.99$ on 5 measuring points) was proven. Coefficient of variation was found to be < 5 %, and recoveries between 55 and 70 % were given. Limits of detection was determined to be 0.2 to 2 ng/g sample.
- Environmental, agricultural, AMD, quantitative analysis 5b, 37d
- 93 009 D. JÄNCHEN* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jae

nchen@camag.com): Detection and quantitation of additives in mineral oil. CBS **81**, 6-7 (1998). HPTLC-AMD on silica gel with a gradient based on methanol – dichloromethane – n-hexane. Quantification by densitometry with absorbance measurement at 220 nm and fluorescence measurement at 366/>400 nm.

Quantitative analysis, postchromatographic derivatization, AMD, lubricants 5d, 35c

8. Substances containing heterocyclic oxygen

- 93 018 K. K. ATINDEHOU, E. F. QUEIROZ, C. TERREAUX, D. TRAORE, K. HOSTETTMANN* (*Institute of Pharmacognosy and Phytochemistry, CH-1015 Lausanne, Switzerland): Three new prenylated isoflavonoids from the root bark of *Erythrina vogelii*. *Planta med.* **68**, 181-182 (2002). TLC of 1-methoxyphaseollidin, isowighteone, and vogelin A, B, C on silica gel. Detection under UV light at 254 nm and with vanillin-sulfuric acid reagent. Detection of 1-methoxyphaseollidin and isowighteone by a direct TLC bioautographic assay with LB (Luria-Bertani) medium.
Pharmaceutical research, qualitative identification, *Erythrina vogelii*, isoflavonoids 8a, 4e
- 93 013 G. BOJASE, R. R. T. MAJINDA*, B. A. GASHE, C. C. W. WANJALA (*Department of Chemistry, University of Botswana, Private Bag 00704, Gaborone, Botswana): Antimicrobial flavonoids from *Bolusanthus speciosus*. *Planta med.* **68**, 615-620 (2002). Preparative TLC of 5,7,3'-trihydroxy-4'-methoxy-5'-prenylisoflavone, 5,7,3'-trihydroxy-4'-methoxy-6,5'-d iprenylisoflavone, 5,7,2',4'-tetrahydroxy-8,3'-diprenylisoflavone, bolusanthin II, bolucarpan A, B, C, D on silica gel with n-hexane - acetone 3:1 by 4-fold development. Testing for antimicrobial activity was done by TLC bioautographic technique using the standard procedures by Rahalison et al. against 5 microorganisms.
Pharmaceutical research, herbal, preparative TLC, *bolusanthus speciosus*, flavonoids 8a
- 93 019 W.-L. LO, F.-R. CHANG, C.-C. LIAW, Y.-C. WU* (*Graduate Institute of Natural Products, Kaohsiung Medical University, Kaohsiung 807, Taiwan, Republic of China): Cytotoxic coumaronochromones from the roots of *Euchresta formosana*. *Planta med.* **68**, 146-151 (2002). Analytical and preparative TLC of trifolirhizin, quercetin, euchretin K, L, M, and N on silica gel using i. a. chloroform - methanol - 29 % ammonia 100:10:1, n-hexane - chloroform 20:1 and 30:1, chloroform ≠methanol 10:1, 20:1 and 30:1 as well as n-hexane - acetone 4:1, 7:1, 10:1 and 60:1. Detection by spraying with Dragendorff's reagent or 50 % sulfuric acid followed by heating on a plate heater.
Pharmaceutical research, preparative TLC qualitative identification, *Euchresta formosana*, coumaronochromones 8a
- 93 020 N. P. D. NANAYAKKARA*, C. L. BURANDT JR., M. R. JACOB (*National Center for Natural Products Research, School of Pharmacy, The University of Mississippi, University, MS 38677, USA): Flavonoids with activity against methicillin-resistant *Staphylococcus aureus* from *Dalea scandens* var. *paucifolia*. *Planta med.* **68**, 519-522 (2002). Preparative TLC of 5²-(1^{'''},1^{'''}-dimethylallyl)-8-(3^{''},3^{''}-dimethylallyl)-2',4',5,7-tetrahydroxyflavone and 2(S)-5²-(1^{'''},1^{'''}-dimethylallyl)-8-(3^{''},3^{''}-dimethylallyl)-2'-methoxy-4',5,7-trihydroxyflavone on silica gel with dichloromethane - methanol 49:1 and n-hexane - ethyl acetate 1:1.
Pharmaceutical research, preparative TLC, qualitative identification, *Dalea scandens* var. *paucifolia*, flavonoids 8a
- 93 063 C. C. W. WANJALA et al., see section 22

- 93 011 A. A. AHMED*, A. E.-H. H. MOHAMED, O. TZAKOU, A. GANI, A. YANNITSAROS, M. A. EL-MAGHRABY, M. E. HASSAN, K.-P. ZELLER (*Department of Chemistry, Faculty of Science, El-Minia University, El-Minia 61519, Egypt): Three pyrone glucoside derivatives from *Conyza albida*. *Planta med.* **68**, 664-666 (2002). TLC of Z-lachnophyllum ester, E-lachnophyllum lactone, and Z-cumulene on silica gel with diethyl ether - petroleum ether 5:1.
Pharmaceutical research, herbal, qualitative identification,
Conyza albida, pyrone glucoside derivatives 8b
- 93 012 S. APERS*, K. CIMANGA, D. VANDEN BERGHE, E. VAN MEENEN, A. O. LONGANGA, A. FORIERS, A. VLIETINCK, L. PIETERS (*Department of Pharmaceutical Sciences, University of Antwerp, Universiteitsplein 1, 2610 Antwerp, Belgium): Antiviral activity of simalikalactone D, a quassinoid from *Quassia africana*. *Planta med.* **68**, 20-24 (2002). Analytical and preparative TLC of quassinoids (quassin and simalikalactone) on silica gel with diethylether - methanol 9:1. Detection under UV light at 254 and 366 nm and after spraying subsequently with 5 % phosphomolybdic acid and 60 % sulfuric acid in methanol, followed by heating at 120 °C for 10 min.
Pharmaceutical research, qualitative identification,
simalikalactone D, *Quassia africana* 8b
- 93 084 M. BEAUFOUR et al., see section 29f
- 93 014 C. CABALLERO-GEORGE, P. M. L. VANDERHEYDEN, T. DE BRUYNE, A.-A. SHAHAT, H. VAN DEN HEUVEL, P. N. SOLIS, M. P. GUPHA, M. CLAEYS, L. PIETERS, G. VAN-QUELIN, A. J. VLIETINCK* (*Department of Pharmaceutical Sciences, University of Antwerp (UIA), Universiteitsplein 1, 2610, Antwerpen, Belgium): In vitro inhibition of [³H]-angiotensin II binding on the human AT1 receptor by proanthocyanidins from *Guazuma ulmifolia* bark. *Planta med.* **68**, 1066-1071 (2002). TLC of (-)-epicatechin, (+)-catechin, procyanidin-B2, and procyanidin-C1 on silica gel with ethyl acetate - acetic acid - formic acid - water 75:2:3:20. Detection by UV light at 254 and 366 nm and by spraying with vanillin-sulfuric acid reagent (1 % vanillin in methanol and 5 % sulfuric acid in ethanol).
Pharmaceutical research, herbal, qualitative identification,
Guazuma ulmifolia bark 8b
- 93 015 M. I. CHOUDHARY*, AZIZUDDIN, A. KHALID, S. Z. SULTANI, ATTA-UR-RAHMAN (*International Center for Chemical Sciences, H. E. J. Research Institute of Chemistry, University of Karachi, Karachi 752760, Pakistan): A new coumarin from *Murraya paniculata*. *Planta med.* **68**, 81-83 (2002). Preparative TLC of 2'-O-ethylmurrangatin and 2'-O-ethylmurrangatin acetate on silica gel with chloroform - methanol 19:1. Detection under UV light at 254 nm.
Pharmaceutical research, qualitative identification, preparative TLC 8b
- 93 016 J.-R. IOSET, G. E. RAOELISON, K. HOSTETTMANN* (*Institute de Pharmacognosie et Phytochimie, B. E. P., CH-1015 Lausanne, Switzerland): An LC/DAD-UV/MS method for the rapid detection of aristolochic acid in plant preparations. *Planta med.* **68**, 856-858 (2002). TLC of aristolochic acid on silica gel with chloroform - methanol - acetic acid 13:4:1 before submitting to a second migration on RP-18 with methanol - water 7:13. Detection by spraying with diphenylamine reagent (0.5 % diphenylamine in 60 % sulfuric acid) followed by heating for 10 min at 100 °C. Detection limit 1 µg in visible light and 0.2 µg under UV light at 366 nm.
Quality control, qualitative identification, aristolochic acid 8b, 11a
- 93 017 C. ITO, M. ITOIGAWA*, N. KOJIMA, H. T.-W. TAN, J. TAKAYASU, H. TOKUDA, H. NISHINO, H. FURUKAWA (*Tokai Gakuen University, Ukigai, Miyoshi-cho, Nishikamo-gun,

- Aichi 470-0207, Japan): Cancer chemopreventive activity of rotenoids from *Derris trifoliata*. *Planta med.* **70**, 8-11 (2004). Preparative TLC of 6aa,12aa-12a-hydroxyelliptone, deguelin, a-toxicarol, rotenone, elliptone on silica gel using dichloromethane, benzene - methanol 47:3, n-hexane - ethyl acetate 4:1, and benzene - acetone 47:3. Detection under UV light at 254 nm.
- Pharmaceutical research, herbal, preparative TLC,
Derris trifoliata, rotenoids 8b
- 93 021 I.-H. PARK, S.-Y. JEON, H.-J. LEE, S.-I. KIM, K.-S. SONG* (*Division of Applied Biology and Chemistry, College of Agriculture and Life Sciences, Kyungpook National University, 1370 Sankyuk-Dong, Daegu 702-701, Korea): A β -secretase (BACE 1) inhibitor hispidin from the mycelial cultures of *Phellinus lintens*. *Planta med.* **70**, 143-146 (2004). Analytical and preparative TLC of hispidin (6-(3,4-dihydroxystyryl)-4-hydroxy-2-pyrone) on silica gel with benzene - ethyl acetate - acetic acid 10:1:1. Detection by spraying with iron(III) chloride reagent.
- Pharmaceutical research, herbal, preparative TLC qualitative identification,
Phellinus lintens 8b
- 93 022 Z. ZHANG, H. N. ELSOHLY*, M. R. JACOB, D. S. PASCO, L. A. WALKER, A. M. CLARK (*National Center for Natural Products Research, School of Pharmacy, University of Mississippi, University, MS 38677, USA): Natural products inhibiting *Candida albicans* secreted aspartic proteases from *Tovomita krukovi*. *Planta med.* **68**, 49-54 (2002). TLC of 3,5-dihydroxy-4-methoxyxanthone, 1,3,5,7-tetrahydroxy-8-isoprenylxanthone, 1,3,5-trihydroxy-8-isoprenylxanthone, 1,5,7-trihydroxy-8-isoprenylxanthone, 1,3,7-trihydroxy-2-isoprenylxanthone, 1,5-dihydroxyxanthone, 1,6-dihydroxy-5-methoxyxanthone, 1,3,5-trihydroxyxanthone, 1,3,6-trihydroxy-5-methoxyxanthone, 1,6-dihydroxy-3,5-dimethoxyxanthone, gentisein, 3-geranyl-2,4,6-trihydroxybenzophenone, betulinic acid, 3,4-dihydroxybenzoic acid on silica gel with chloroform - methanol 9:1 and benzene - ethyl acetate 4:1, or on silica gel RP-18 with methanol - water 7:3.
- Pharmaceutical research, qualitative identification, *Tovomita krukovi* 8b

10. Carbohydrates

- 93 023 W. JALYSCHKO, M. RUSSEV, R. BRANDENBURGER, L. W. Kroh* (*Institute of Food Chemistry, Technical University Berlin, Gustav-Meyer-Allee 25, D-13355 Berlin, Germany): Analysis of carbohydrates with planar chromatography. *CBS* **82**, 9-11 (1999). HPTLC-AMD of glucose, malt oligosaccharides, anhydrosugars, transglycosidation products, fructose and its derivatives, maltulose, Amadori-compounds and Heyns-compounds on silica gel. Caramelization: isocratic 23-step gradient based on chloroform - methanol - 0.05 % aqueous boric acid 20:11:2 with 3 mm increments of the developing distance. Maillard-reaction: isocratic 3-step gradient chloroform - dichloromethane - methanol - water 35:15:35:6 over 25, 50, 75 mm developing distance. Detection by dipping in aniline-diphenylamine-phosphoric acid reagent, followed by heating at 120 °C for 10 min. Quantification by absorbance measurement at 385 nm.
- Qualitative identification quantitative analysis AMD 10a
- 93 024 B. P. DA SILVA, J. P. PARENTE* (*Laboratório de Química de Plantas Medicinais, Núcleo de Pesquisas de Produtos Naturais, Centro de Ciências da Saúde, Universidade Federal do Rio de Janeiro, PO Box 68045, CEP 21949-970, Rio de Janeiro, RJ Brasil): Chemical properties and biological activity of a polysaccharide from *Melocactus depressus*. *Planta med.* **68**, 74-76 (2002). TLC of monosaccharides (galactose, arabinose) as TMSi methyl glycosides on silica gel with butanol - pyridine - water 6:4:3. Detection by spraying with resorcinol sulfuric acid.
- Pharmaceutical research, qualitative identification,
Melocactus depressus, galactose, arabinose 10a

- 93 025 G. VACCARI*, G. LODI (*Department of Chemistry, University of Ferrara, Via Borsari 45, I-44100 Italy): Identification and quantitative determination of oligosaccharides in beet molasses. *CBS* **84**, 4-7 (2000). HPTLC-AMD of raffinose, sucrose, 1+6-kestoses, neo-kestose, nystose, and fructosyl-nystose on diol layer with a 9-step gradient from acetonitrile/acetone 1:1 - water 85:15 to acetonitrile/acetone 1:1 - water 95:5. Detection by dipping in 4-aminobenzoic acid reagent, followed by heating at 115 °C for 15 min. Quantitative determination by fluorescence measurement at 366/>400 nm and absorbance measurement at 400 nm.
- Quality control, AMD densitometry quantitative analysis, oligosaccharides, beet molasses, sugar production 10a

11. Organic acids and lipids

- 93 004 R. BARTZATT et al., see section 3e
- 93 026 S. ERAZO*, R. NEGRETE, M. ZALDIVAR, N. BACKHOUSE, C. DELPORTE, I. SILVA, E. BELMONTE, J. L. LÓPEZ-PÉREZ, A. S. FELICIANO (*Department of Pharmacological and Toxicological Chemistry, School of Chemical and Pharmaceutical Sciences, University of Chile, P. O. Box 233, Santiago-1, Chile): Methyl psilate: A new antimicrobial metabolite from *Psila bovensis*. *Planta med.* **68**, 66-67 (2002). TLC of methyl psilate on silica gel with dichloromethane - ethyl acetate 9:1. Visualization under UV light at 366 nm, by spraying with anisaldehyde - sulfuric acid and with Liebermann-Burchard reagent. Bioautography after sterilization by UV light at 254 nm: the plate was overlaid with Plate Count Agar, inoculated with an overnight culture, placed in a humid chamber and incubated overnight at 37 °C. The bioautograms were sprayed with an aqueous solution of thiazoyl blue (MTT).
- Pharmaceutical research, qualitative identification, *Psila bovensis*, methyl psilate, bioautography 11a
- 93 016 J.-R. IOSET et al., see section 8b
- 93 029 D. JÄNCHEN* (Ed.) (CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jaenchen@camag.com): Planar chromatography for the analysis of malolactic fermentation. *CBS* **84**, 14-15 (2000). HPTLC of wine samples on silica gel with diisopropyl ether - formic acid - water 16:3:1 with chamber saturation. Detection by heating at 110 °C for 15 min, followed by cooling to room temperature and dipping in bromophenol blue reagent. Quantitative determination of malic, lactic, and succinic acid by absorbance measurement at 430 nm.
- Food analysis, quality control, densitometry, quantitative analysis, malolactic fermentation, wine, malic acid, lactic acid, succinic acid 11a
- 93 003 M. SAJEWICZ et al., see section 2a
- 93 085 J.A. THAKARDA et al., see section 30a
- 93 033 R.F. WILSON*, T.C. MARQUARDT (*USDA-ARS, Raleigh, North Carolina 27695-7620, USA): An improved reversed-phase thin-layer chromatography method for separation of fatty acid methyl esters. *JAOCS* **75**, 1889-1892 (1998). TLC of fatty acid methyl esters from plant tissues on RP-18 phase with acetonitrile - 1,4-dioxane - acetic acid 80:20:1 containing 10 % silver nitrate.

Qualitative identification, argentation chromatography, fatty acid methyl esters 11a

93 149 Y. XIE (Xie Yumei) et al., see section 32c

93 027 H. FARWANAH, K. RAITH*, R. NEUBERT, S. ZELLMER, (*Institute for Pharmaceutical Technology and Biopharmacy, Martin Luther University, W.-Langenbeck-Str. 4, D-06120 Halle, Germany, raith@pharmazie.uni-halle.de): Improved analysis of skin lipids by AMD. CBS **90**, 2-4 (2003). HPTLC-AMD on silica gel with an 11-step gradient with chloroform - ethanol - acetone followed by 3 isocratic steps with chloroform for separation of cholesterol, cholesterol sulfate and various ceramide classes. For separation of cholesterol, fatty acids, triacylglycerol, cholesterol esters, and squalene a 2-step gradient with n-hexane - ethyl acetate followed by an isocratic step with n-hexane. Conditioning between single runs with 4 M acetic acid. Detection by dipping in copper sulfate reagent followed by heating at 150 °C for 20 min. Quantitative determination by absorbance measurement at 546 nm.

Pharmaceutical research, HPTLC AMD quantitative analysis densitometry, skin lipids, ceramides, fatty acids, cholesterol

11c, 13c, 15a, 17c

93 028 C. HEIFT*, K. SCHIPMANN, R. LANGE (*Degussa Texturant Systems Germany GmbH & Co. KG, Ausschläger Elbdeich 62, D-20539 Hamburg, Germany, claudia.heift@degussa.com): Effective analysis of phospho- and glycolipids in plant lecithins. CBS **90**, 6-7 (2003). HPTLC phospholipids and glycolipids from rape seed on lichrospher silica gel with chloroform - methanol - acetone - water 18:15:2:1 for phospholipid separation and with acetone - chloroform - water 30:15:2 for glycolipid separation, developing distance 70 mm each. Detection by dipping in molybdato-phosphoric acid reagent (5 % in ethanol) followed by heating at 120 °C for 15 min. Quantitative determination by absorbance measurement at 720 nm.

Herbal, quality control traditional medicine, HPTLC densitometry

11c

93 030 E. REICH* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland): Planar chromatography for the detection of lipids in pot fragments and other artifacts. CBS **85**, 9 (2000). HPTLC of pot fragment extracts on RP-18 plates developed twice at 4 °C with first methanol - acetonitrile - THF 8:2:1 over 20 mm and second methanol - acetonitrile - THF 18: 2:1 over 80 mm after pre-chromatographic derivatization with 1 % N,N'-dicyclohexylcarbodiimide in dichloromethane followed by dansyl semicadaverine in dichloromethane. After drying the plate was dipped in 4 % Triton X100 in hexane. Evaluation at 366/>400 nm.

Qualitative identification, archaeological artifacts, trace analysis, lipids, pot fragments

11c

93 031 B. RENGER* (*Byk Gulden, Robert-Bosch-Str. 8, D-78224 Singen, bernd.renger@byk.de): Analysis of phospholipids/lecithin - cost comparison planar chromatography/HPLC. CBS **81**, 2-5 (1998). The performance and reliability of result of both procedures is comparable - HPTLC being slightly better than HPLC. For the assay of phospholipids in pure substances, pharmaceutical products and lecithin HPTLC is more cost efficient than HPLC by a factor of 2.5.

Quantitative analysis, benchmarking

11c, 2a

93 032 C. SCHAERER* (*Fachhochschule beider Basel FHBB, Department of Chemistry, CH-4132 Muttenz, Switzerland): Comparison of HPLC-ELSD and modern planar chromatography in today's quality control of phospholipids. CBS **89**, 12-15 (2002). HPTLC on silica gel with chloroform - methanol - water - ammonia 24% 65:30:4:2 over 90 mm with chamber saturation for 30 min. Detection by dipping in CuSO₄/H₃PO₄ reagent for 8 s followed by heating at 170 °C for 10 min. Quantitative determination by absorbance measurement at 500 nm. Comparising operating

costs the HPTLC analysis appears preferable for quality control of samples with known content. HPLC is more cost effective for the analysis of samples with unknown content.

Quality control, comparison of methods, quantitative analysis, densitometry, phospholipids, HPLC

11c

13. Steroids

93 027 H. FARWANAH et al., see section 11c

93 094 J.-J. CHEN et al., see section 32e

14. Steroid glycosides, saponins and other terpenoid glycosides

93 034 F. HU, K. SCHMIDT, S. STOYANOVA, L. ZENGZHI, U. GRÄFE, M. HAMBURGER* (*Institute of Pharmacy, Friedrich-Schiller-University Jena, Semmelweisstr. 10, 07743 Jena, Germany): Radical scavengers from the entomogenous deuteromycete *Beauveria amorpha*. *Planta med.* **68**, 64-65 (2002). TLC of 6-methyl-1,2,4-trihydroxybenzene-1-O- β -D-4'-methylglucopyranoside and (-)-terredionol on silica gel with chloroform - methanol - water 13:7:1. Detection with Godin's reagent or DPPH radical (1 mg/mL in ethanol).

Pharmaceutical research, qualitative identification

14

93 035 T.-H. LEE, G.-J. WANG, C.-K. LEE Y.-H. KUO, C.-H. CHOU* (*Department of Biological Sciences, National Sun Yat-sen University, Kaohsiung, Taiwan 804, Republic of China): Inhibitory effects of glycosides from the leaves of *Melaleuca quinquenervia* on vascular contraction of rats. *Planta med.* **68**, 492-496 (2002). TLC of glycosides (3-hydroxy-5-methoxy-4-methylphenyl β -D-glucopyranoside, 4-benzoyl-2-C- β -glucopyranosyl-3,5-dihydroxy-6-methylphenyl β -D-glucopyranoside, 2-endo- β -D-glucopyranosyloxy-1,8-cineole, and 2-exo- β -D-glucopyranosyloxy-1,8-cineole, roseoside, citroside) on silica gel with ethyl acetate - formic acid - water 17:2:3. Detection under UV light at 254 nm and by dipping in vanillin - sulfuric acid reagent.

Pharmaceutical research, qualitative identification, *Melaleuca quinquenervia*, glycosides

14

93 036 M. SANTOUR, A.-C. MITAINE-OFFER, T. MIYAMOTO, A. DONGMO, M.-A. LACAILLE-DUBOIS* (*Laboratoire de Pharmacognosie, Unité UMIB, EA 3660, Faculté de Pharmacie, Université de Bourgogne, 7, Bd. Jeanne d'Arc, BP 879 00, F-21079 Dijon Cedex, France): Antifungal steroid saponins from *Dioscorea cayensis*. *Planta med.* **70**, 90-92 (2004). HPTLC and TLC of saponins (26-O- β -D-glucopyranosyl-22-methoxy-3 β ,26-dihydroxy-25(R)-furost-5-en-3-O-a-L-rhamnopyranosyl-(1-4)-a-L-rhamnopyranosyl-(1-4)-[a-L-rhamnopyranosyl-(1-2)]- β -D-glucopyranoside, dioscin, and diosgenin 3-O-a-L-rhamnopyranosyl-(1-4)-a-L-rhamnopyranosyl-(1-4)-[a-L-rhamnopyranosyl-(1-2)]- β -D-glucopyranoside) on silica gel with chloroform - methanol - water 13:7:2 (lower phase), of saponin with chloroform - methanol 9:1 and of sugars (e. g. glucose, rhamnose) with chloroform - methanol - water 8:5:1. Detection i. a. under UV light at 254 nm.

Pharmaceutical research, herbal, qualitative identification, *Dioscorea cayensis*

14

93 053 J. WANDJI et al., see section 15a

15. Terpenes and other volatile plant ingredients

- 93 038 S. BLOCK*, C. STÉVIGNY, M.-C. DE PAUW-GILLET, E. DE HOFFMANN, G. LLABRÈS, V. ADJAKIDJÉ, J. QUETIN-LECLERCQ (*Laboratoire de Pharmacognosie, Unité CHAM, UCL 72.30, Avenue E. Mounier 72, 1200 Bruxelles, Belgium): Ent-Trachyloban-3 β -ol, a new cytotoxic diterpene from *Croton zambesicus*. *Planta med.* **68**, 647-649 (2002). TLC of ent-trachyloban-3 β -ol on silica gel with toluene - ethyl acetate - methanol 40:9:1. Detection by derivatization with anisaldehyde - sulfuric acid reagent.
Pharmaceutical research, herbal, qualitative identification 15a
- 93 039 Y.-X. CHENG, J. ZHOU*, S.-M. DENG, N.-H. TAN (*Laboratory of Phytochemistry, Kunming Institute of Botany, The Chinese Academy of Sciences, Kunming 659204, Yunnan, P. R. China): New norsesquiterpenoids from *Cucubulus baccifer*. *Planta med.* **68**, 101-103 (2002). TLC of drummondol on silica gel with chloroform - acetone 3:1 or chloroform - methanol - water 30:20:3.
Pharmaceutical research, qualitative identification, *Cucubulus baccifer*, norsesquiterpenoids 15a
- 93 037 J. A. DOUGLAS*, B. M. SMALLFIELD, E. J. BURGESS, N. B. PERRY, R. A. ANDERSON, M. H. DOUGLAS, V. LEANNE GLENNIE (*Crop and Food Research, Ruakura Research Centre, Private Bag 3123, Hamilton, New Zealand): Sesquiterpene lactones in *Arnica montana*: A rapid analytical method and the effects of flower maturity and simulated mechanical harvesting on quality and yield. *Planta med.* **70**, 166-170 (2004). Analytical TLC of acetyldihydrohelenalin, methacryloyldihydrohelenalin, acetyl-, methacryloyl-, isobutyryl-, tigloyl-, 2-methylbutyryl-, and isovalerylhelenalin on silica gel with n-pentane - diethyl ether 1:3. Detection under UV light at 254 nm.
Pharmaceutical research, herbal, qualitative identification, *Arnica montana* 15a
- 93 027 H. FARWANAH et al., see section 11c
- 93 047 D. N. GUEDES, D. F. SILVA, J. M. BARBOSA-FILHO, I. A. MEDEIROS* (*Laboratório de Tecnologia Farmacêutica, Universidade de Paraíba, Caixa Postal 5009, 58051-970 Joao Pessoa, PB, Brazil): Muscarinic agonist properties involved in the hypotensive and vasorelaxant response of rotundifolone in rats. *Planta med.* **68**, 700-704 (2002). Preparative TLC of rotundifolone on silica gel with hexane by 3-fold development. Visualization under UV light.
Pharmaceutical research, preparative TLC, rotundifolone 15a
- 93 040 J.-T. HAN, H.-Y. KIM, Y.-D. PARK, Y.-H. LEE, K.-R. LEE, B.-M. KWON, N.-I. BAEK* (*Graduate School of Biotechnology, Kyun Hee University, Seochun-Ri 1, Kiheung-Eup, Yongin-Si, Kyunggi-Do 449-701, Korea): Aceriphyllic acid A, a new ACAT inhibitory triterpenoid, from *Aceriphyllum rossii*. *Planta med.* **68**, 558-561 (2002). TLC of 3 new triterpenoids 3-oxoolean-12-en-27-oic acid, 3a-hydroxyolean-12-en-27-oic acid, and β -peltoboykinolic acid on silica gel with n-hexane - ethyl acetate - ethanol 10:1:2.
Pharmaceutical research, qualitative identification, *aceriphyllum rossii*, aceriphyllic acid A 15a
- 93 041 Q.-B. HAN, W. XIANG, R.-T. LI, M.-L. LI, S.-W. LI, H.-D. SUN* (*State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Heilongtan, Kunming 650 204, P. R. China): Cytotoxic ent-kaurane diterpenoids from *Isodon rubescens* var. *rubescens*. *Planta med.* **70**, 269-272 (2004). Analytical and

preparative TLC of i. a. xindonguin D and F, and melissoidesin G on silica gel with chloroform - methanol 7:1; 9:1; 10:1; 20:1 or cyclohexane - isopropanol 4:1 by 3-fold development, 8:1 or cyclohexane - diethyl ether 1:2. Visualization by spraying with 10 % sulfuric acid in ethanol followed by heating.

Pharmaceutical research, herbal, preparative TLC qualitative identification 15a

93 008 D. JÄNCHEN* (Ed.), see section 4d

93 056 J. Y. KIM, K.-W. LEE, S.-H. KIM, J. J. WEE, Y. S. KIM, H. J. LEE* (*Department of Food Science and Technology and School of Agricultural Biotechnology, Seoul National University, Suwon, 441-744, Korea): Inhibitory effect of tumor cell proliferation and induction of G2/M cell cycle arrest by panaxytriol. *Planta med.* **68**, 119-122 (2002). TLC of the polyacetylenic compound panaxytriol on silica gel with hexane - ethyl acetate 3:1. Visualization under UV light at 254 nm.

Pharmaceutical research, qualitative identification, panaxytriol 15a

93 042 L.-Y. KONG*, M.-J. QIN, M. NIWA (*Department of Natural Medicinal Chemistry, China Pharmaceutical University, Nanjing 210009, P. R. China): New cytotoxic bis-labdanic diterpenoids from *Alpina calcarata*. *Planta med.* **68**, 813-817 (2002). Analytical and preparative TLC of calcatararin D and E on silica gel with n-hexane - ethyl acetate 23:2 and 43:7 by 2-fold development. Visualization under UV light at 254 nm and by spraying with 10 % molybdcic acid reagent followed by heating.

Pharmaceutical research, herbal, preparative TLC qualitative identification, *Alpina calcarata*, diterpenoids 15a

93 043 J. LEE, B. MIN, S. LEE, M. NA, B. KWON, C. LEE, Y. KIM, K.-H. BAE* (*College of Pharmacy, Chungnam National University, Taejeou 305-764, Korea): Cytotoxic sesquiterpene lactones from *Carpesium abrotanoides*. *Planta med.* **68**, 745-747 (2002). Preparative TLC of carpesiolin, carabrone, carabrol, telekin, ivalin, and 11,13-didehydroivaxillin on silica gel with n-hexane - ethyl acetate - acetone 3:1:1 and 8:1:1 as well as n-hexane - acetone 2:1.

Pharmaceutical research herbal, preparative TLC, *Carpesium abrotanoides*, sesquiterpene lactones 15a

93 044 R.-W. LIN, I.-L. TSAI, C.-Y. DUH, K.-H. LEE, I.-S. CHEN* (*Graduate Institute of Natural Products, Kaohsiung Medical University, Kaohsiung, Taiwan 807, Republic of china): New lignans and cytotoxic constituents from *Wikstroemia lanceolata*. *Planta med.* **70**, 234-238 (2004). Analytical and preparative TLC of (-)-aptosimon, (-)-diasesamin-di-y-lactone, (-)-sesamin, (+/-)-syringaresinol, (+)-wikstromol, (+)-hinokinin, palmitic acid, stearic acid, 3,6-dihydroxy-2-methoxy-4-methylacetophenone, 2,6-dimethoxy-p-benzoquinone, and lichenxanthone on silica gel with n-hexane - ethyl acetate 5:1, 10:1 and chloroform - acetone 4:1, 20:1.

Pharmaceutical research, herbal, preparative TLC, qualitative identification 15a

93 045 A. M. MADUREIRA, M.-J. U. FERREIRA*, N. GYÉMÁNT, K. UGOCSAI, J. R. ASCENSO, P. M. ABREU, J. HOHMANN, J. MOLNÁR (*Centro de Estudos de Ciências Farmacêuticas, Faculdade de Farmácia da Universidade de Lisboa, Av. das Forças Armadas, 1600-083 Lisboa, Portugal): Rearranged jatrophane-type diterpenes from *Euphorbia* species. Evaluation of their effects on the reversal of multidrug resistance. *Planta med.* **70**, 45-49 (2004). TLC of i. a. portlandicine (2a, 5a, 14a, 17a-tetraacetoxy-3 β -benzoyloxy-15 β -hydroxy-9-oxoparaliane), 1 β , 5a, 14a, 17a-tetraacetoxy-3 β -benzoyloxy-15- β -hydroxy-9-oxo-paraliane, and oleanolic acid on silica gel with dichloromethane - methanol 99:1 by 3-fold development and 49:1 as well as

chloroform by 5-fold development. Visualization under UV light at 254 nm and by spraying with sulfuric acid - acetic acid - water 1:20:4 followed by heating.

Pharmaceutical research, qualitative identification, Euphorbia species 15a

- 93 046 M. MAO, Z. JIA* (*Department of Chemistry, Lanzhou University, Lanzhou 730000, P. R. China): Eremophilane sesquiterpenes from *Cacalia ainsliaeflora*. *Planta med.* **68**, 55-59 (2002). Analytical and preparative TLC of 3 β -angeloyloxy-8 α -hydroxy-6 β -methoxyeremophil-7(11),9(10)-dien-8,12-olide, 2:3 β -angeloyloxy-6 β ,8 α -dihydroxyeremophil-7(11),9(10)dien-8,12-olide, 5:3,8-oxo-eremophila-6,9-dien-12-oic acid, 3:3 β -angeloyloxy-8 α -hydroxy-6 β -ethoxyeremophil-7(11),9(10)-dien-8,12-olide, 4:3 β -angeloyloxy-8-oxo-eremophila-6,9-dien-12-oic acid on silica gel with chloroform - methanol 12:3:2 and dichloromethane - methanol 10:1.

Pharmaceutical research, qualitative identification, *Cacalia ainsliaeflora* 15a

- 93 048 Y. REN, P. J. HOUGHTON*, R. C. HIDER, M.-J. R. HOWES (*Department of Pharmacy, King's College London, Franklin-Wilkins Building, 150 Stamford Street, London SE1 9NN, United Kingdom) : Novel diterpenoid acetylcholine-esterase inhibitors from *Salvia miltiorhiza*. *Planta med.* **70**, 201-204 (2004). Analytical and preparative TLC of dihydrotanshinone, cryptotanshinone, and tanshinone I, IIA on silica gel with dichloromethane and dichloromethane - ethyl acetate 4:1.

Pharmaceutical research, herbal, preparative TLC qualitative identification 15a

- 93 049 N. SULTANOVA, T. MAKHMOOR, A. YASIN, Z. A. ABILOV, V. B. OMURKAMZINOVA, ATTA-UR-RAHMAN, M. I. CHOUDHARY* (*HEJ Research Institute of Chemical Sciences, University of Karachi, Karachi, Pakistan): Isotamarixen - a new antioxidant and propylendopeptidase-inhibiting triterpenoid from *Tamarix hispida*. *Planta med.* **70**, 65-67 (2004). Analytical and preparative TLC of 3 α -(3'',4''-dihydroxy-trans-cinnamoyloxy)-D-friedoolean-14-en-28-oic acid and isorhamnetin on silica gel with dichloromethane - methanol 46:1 and methanol - chloroform 3:7. Visualization under UV light at 254 nm.

Pharmaceutical research, herbal traditional medicine, preparative TLC, qualitative identification, *Tamarix hispida* 15a

- 93 165 F.M. TRIOLO et al., see section 35b

- 93 050 A. ULUBELEN*, H. BIRMAN, S. ÖKSÜZ, G. TOPEN, U. KOLAK, A. BARLA, W. VOELTER (*Faculty of Pharmacy, University of Istanbul, 34452 Beyazit-Istanbul, Turkey): Cardioactive diterpenes from the roots of *Salvia eriophora*. *Planta med.* **68**, 818-821 (2002). Preparative TLC of i. a. ferruginol, horminone, acetylhorminone, 4,14-dihydroxy-saprorthoquinone, aethiopinone, salvipisone, and salvilimbino on silica gel with petroleum ether, toluene - dichloromethane 17:3, and dichloromethane - methanol 49:1.

Pharmaceutical research, herbal, preparative TLC, *Salvia eriophora*, diterpenes 15a

- 93 051 C. VALENTE, M. J. U. FERREIRA*, P. M. ABREU, N. GYÉMÁNT, K. UGOCSAI, J. HOHMANN, J. MOLNÁR (*Centro de Estudos de Ciências Farmacêuticas, Faculdade de Farmácia da Universidade de Lisboa, Av. das Forças Armadas, 1 600-083 Lisboa, Portugal): Pubescenes, jatrophone diterpenes, from *Euphorbia pubescens*, with multidrug resistance reversing activity on mouse lymphoma cells. *Planta med.* **70**, 81-84 (2004). Preparative TLC of pubescene D (3 β -,9 α -diacetoxy-7 β -benzoyloxy-15 β -hydroxy-14-oxo-2 β H-jatropha-5E,12E-diene) on silica gel with chloroform - acetone 9:1. Detection under UV light at 254 nm.

Pharmaceutical research, herbal, preparative TLC, *Euphorbia pubescens* 15a

- 93 052 C. VALENTE, M. PEDRO, J. R. ASCENSO, P. M. ABREU, M. S. J. NACIMENTO, M.-J. U. FERREIRA*, (*Centro de Estudos de Ciências Farmacêuticas, Faculdade de Farmácia da Universidade de Lisboa, Av. das Forças Armadas, 1600-08 3 Lisboa, Portugal): Euphobubescenol and euphobubescene, two new jatrophone polyesters, and lathyrane-type diterpenes from *Euphorbia pubescens*. *Planta med.* **70**, 244-249 (2004). TLC of euphobubescenol (5a,8a,15β-triacetoxy-3a-benzoyloxy-4a-hydroxy-9,14-dioxo-13βH-jatropha-6(17),11E-diene, jolkinaol A, and A' on silica gel with chloroform - methanol 39:1 by 3-fold development, chloroform - methanol 19:1 by 2-fold development, secondly chloroform - methanol 9:1. Visualization under UV light and by spraying with sulfuric acid - acetic acid - water 1:20:4 followed by heating.
Pharmaceutical research, herbal, qualitative identification 15a
- 93 053 J. WANDJI*, F. TILLEQUIN, D. A. MULHOLLAND, J.-D. WANSI, T. Z. FOMUM, V. FUENDIJEI, F. LIBOT, N. T. SABANG (*Department of Organic Chemistry, University of Yaounde-1, Faculty of Science, P. O. Box 812, Yaounde, Cameroon): Fatty acid esters of triterpenoids and steroid glycosides from *Gambeya africana*. *Planta med.* **68**, 822-826 (2002). TLC of β-amyrin stearate, β-amyrin eicosanoate, β-amyrin docosanoate, and erythrodiol fatty acid esters on silica gel with n-hexane - dichloromethane 4:1, and dichloromethane - methanol 19:1. Visualization under UV light at 254 and 366 nm and by spraying with 1 % vanillin - 5 % sulfuric acid reagent followed by heating at 100 °C. Also TLC of xylose and glucose with i-propanol - water 17:3 and detection with p-anisidine phthalic acid reagent.
Pharmaceutical research herbal, qualitative identification, *Gambeya africana* 15a, 14
- 93 054 M. WERTHER* (*Institut für Tierarzneimittel GmbH, Berliner Allee 317-321, D-13088 Berlin, Germany, margit.werther @camag-berlin.de): Testing of feed mixers by HPTLC. *CBS* **89**, 9 (2002). HPTLC of feedstuff on silica gel with 1) chloroform - methanol 1:1 over 30 mm followed by drying and 2) n-hexane - acetone 4:1 over 50 mm in horizontal development chamber with sandwich configuration. Quantitative determination by absorbance measurement at 466 nm.
Quality control, densitometry, quantitative analysis, feed mixers, accuracy of mixing, canthaxanthine 15a
- 93 055 W. XIA (Xia Wei)*, X. ZHANG (Zhang Xinzhi) (*Coll. Materials Sci. & Chem. Eng., Zhejiang Univ., Hangzhou 31002 7, P. R. China): (Isolation of β-carotene from discarded tobacco leaves.) (Chinese). *Chinese J. Chromatogr. (Sepu)* **22** (1), 54-56 (2004). TLC of the leave extract or eluate (on silica gel column eluted with petroleum ether) on silica gel with benzene - acetone 4:1. Detection by iodine vapour. Identification by comparison with the standard and NMR. Quantitation by HPLC.
Agricultural, quality control, qualitative identification, β-Carotene 15a, 4e, 30
- 93 159 C. YANG et al., see section 32e

17. Amines, amides and related nitrogen compounds

- 93 004 R. BARTZATT et al., see section 3e
- 93 057 M. STOYKE* (*Institute for Veterinary Pharmacology and Toxicology GmbH, Weissenseer Str. 36, D-16321 Bernau, Germany): Determination of histamine in fish and fish products by HPTLC. *CBS* **83**, 9-11 (1999). HPTLC of histamine in fish on RP-18 W with n-propanol - 25 % ammonia 4:1 without chamber saturation. Detection by dipping in Pauly's reagent. Evaluation with video

technology and quantitative determination by absorption measurement at 530 nm.

Food analysis, quality control, quantitative analysis, densitometry,
histamine, fish, video technology 17a

- 93 058 Y. YANG (Yang Yun)*, Y. FENG (Feng Yinman), T SHI (Shi Tianjun), Y. WANG (Wang Yuming) (*Henan Coll. TCM, Zhengzhou, 450008, P. R. China): (Study of the quality standard for Wuziyanzong pills.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (7), 538-540 (2004). TLC on silica gel with 1) petroleum ether - ethyl acetate - butanone ≠methanol - water 15:25:3:7:1; 2) petroleum ether - ethyl acetate - formic acid 15:5:1. Detection 1) under UV light, 2) by spraying with 5 % potassium iodobismuthate solution and heating at 105 °C for 5 min. Identification by fingerprint technique. Quantification of betaine by densitometry at 514 nm. Pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis 17a, 4d, 32c
- 93 094 J.-J. CHEN et al., see section 32e
- 93 027 H. FARWANAH et al., see section 11c

18. Amino acids and peptides, chemical structure of proteins

- 93 059 B. CLASSEN*, W. BLASCHEK (*Pharmazeutisches Institut der Christian Albrechts-Universität Kiel, Abteilung Pharmazeutische Biologie, Gutenbergstr. 76, 24118 Kiel, Germany): An arabinogalactan-protein from cell culture of *Malva sylvestris*. *Planta med.* **68**, 232-236 (2002). HPTLC of Arg, Asx, Glx, Ser, Thr, Ala on silica gel with acetonitrile - water - acetic acid 85:14:1, 3-fold horizontal development followed by heating at 80 °C for 10 min after each development. Detection of amino acids by derivatization with ninhydrin reagent, followed by heating at 120 °C for 10 min. Visualization of Hyp by derivatization with NBD-chloride reagent (sodium acetate in aqueous methanol and 0.2 % 7-chloro-4-nitrobenzo-2-oxa-1,3-diazole in ethanol), followed by heating (110 °C, 2 min) and treatment with fumes of hydrochloric acid. Pharmaceutical research, qualitative identification, amino acids 18a
- 93 060 B. HESS, J. SHERMA* (*Dep. Chem., Lafayette College, Easton, P.A. 18042, USA): Quantification of arginine in dietary supplement tablets and capsules by silica gel high performance thin-layer chromatography with visible mode densitometry. *Acta Chromatographica* **14**, 60-69 (2004). TLC on silica gel with 1-butanol - glacial acetic acid - water 3:1:1 with chamber saturation. Detection by spraying with ninhydrin reagent (0.3 g ninhydrin in 100 mL 1-butanol plus 2 mL glacial acetic acid), followed by heating at 110 °C for several min. Quantification by densitometry at 495 nm. Validation of the accuracy by analysis of spiked blank and standard addition samples and precision by performing replicate analysis on a single day and on different days. Recoveries and RSD for spiked blank and standard addition samples were 98.8 % and 98.5 %, 1.92 % and 0.67 %, respectively. Discussion of use of the method for the routine quality control of nutritional supplements. Food analysis, quality control, traditional medicine, quantitative analysis 18a
- 93 061 E. REICH* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland): Cleaning validation - an ideal task for the automatic TLC sampler ATS4. *CBS* **85**, 4-5 (2000). HPTLC of peptide fragment A on lichrospher silica gel with dichloromethane over 50 mm. Quantitative

determination by absorbance measurement at 250 nm. Precision is 0.23 % (n=10 at 50 ng) and repeatability 2.9 % (n=30 over 5 plates at 50 ng), recovery is found to be 104 % at 50 ng.

Quality control, quantitative analysis, densitometry, cleaning validation 18b

22. Alkaloids

- 93 066 A. H. ABOU-DONIA, M. E. AMER, F. A. DARWISH, F. F. KASSEM, H. M. HAMMODA, M. S. ABDEL-KADER*, B.-N. ZHOU, D. I. G. KINGSTON (*Department of Pharmacognosy, Faculty of Pharmacy, Alexandria University, Alexandria, Egypt): Two new alkaloids of the crinane series from *Pancreatium Sickenbergeri*. *Planta med.* **68**, 377-379 (2002). Analytical and preparative TLC of ent-6a/6 β -hydroxybuphanisine, haemanthamine, norgalathamine, vittatine, 11-hydroxyvittatine, and (-)-8-demethylmaritidine on silica gel using chloroform - methanol 19:3 and 9:1.

Pharmaceutical research, qualitative identification, preparative TLC, *Pancreatium Sickenbergeri*, crinane series 22

- 93 062 B. AKENDENGUE, E. NGOU-MILANA, F. ROBLLOT, A. LAURENS*, R. HOCQUEMILLER, P. GRELLIER, F. FRAPPIER (*Laboratoire de Pharmacognosie, UPRES-A CNRS 8076 (BioCIS), Faculté de Pharmacie, F-92296 Châtenay-Malabry, France): Antiplasmodial activity of *Uvaria klaineana*. *Planta med.* **68**, 167-169 (2002). Preparative TLC of crotosparine, crotonosine, and zenkerine on silica gel with ethyl acetate - methanol 6:4. Visualization under UV light.

Pharmaceutical research, preparative TLC, *Uvaria klaineana*, crotosparine, crotonosine, zenkerine 22

- 93 064 C. CABALLERO-GEORGE, A. J. VLIETINCK* (*Department of Pharmaceutical Sciences, University of Antwerp (UIA), Universiteitsplein-1, 2610, Antwerpen, Belgium): Inhibitory activity on binding of specific ligands to the human angiotensinII AT1 and endothelin 1 ETA receptors: Bioactive benzophenanthridine alkaloids from the root of *Bocconia frutescens*. *Planta med.* **68**, 770-775 (2002). Preparative and analytical TLC of chelirubine, sanguinarine, macarpine, and chelerythrine on silica gel and aluminum oxide with chloroform - methanol 49:1. Detection under UV light.

Pharmaceutical research, herbal, qualitative identification, preparative TLC, *Bocconia frutescens*, benzophenanthridine alkaloids 22

- 93 065 J.-J. CHEN, Y.-L. CHANG, C.-M. TENG, C.-C. SU, I.-S. CHEN* (*Graduate Institute of Pharmaceutical Sciences, Kaohsiung Medical University, Kaohsiung, Taiwan, Republic of China): Quinoline alkaloids and anti-platelet aggregation constituents from the leaves of *Melicope semecarpifolia*. *Planta med.* **68**, 790-793 (2002). Preparative and analytical TLC of melisemine, confusadine, melicarpinone, edulinine, (S)-(-)-7,8-dimethoxyplatydesmine, isoplatydesmine, skimmianine, confusaneline, kokusaginine, and haplopine on silica gel with chloroform - acetone 5:1 and 10:1, chloroform - methanol 25:1 and 3:1, benzene - ethyl acetate 1:1, benzene - methanol 10:1, and ethyl acetate - methanol 10:1.

Pharmaceutical research, herbal, preparative TLC, qualitative identification, *Melicope semecarpifolia* 22

- 93 103 N. F. DE MOURA et al., see section 32e

- 93 067 J. HOHMANN*, P. FORGO, J. MOLNÁR, K. WOLFARD, A. MOLNÁR, T. THALHAMMER, I. MÁTHÉ, D. SHARP LES (*Department of Pharmacognosy, University of Szeged, P. O. Box

- 121, 6701 Szeged, Hungary): Antiproliferative amaryllidaceae alkaloids isolated from the bulbs of *Sprekelia formosissima* and *Hymenocallis x festalis*. *Planta med.* **68**, 454-457 (2002). Preparative TLC of (+)-tazettine, (+)-pretazettine, (+)-3-epimacronine, (-)-lycorine, (-)-haemanthidine, (+)-haemanthamine, (+)-8-O-demethylmaritidine, ismine, trisphaeridine on silica gel with benzene - chloroform - methanol 7:2:1 in ammonia vapour, chloroform - methanol 9:1 and cyclohexane - ethyl acetate - ethanol 15:10:1.
- Pharmaceutical research, qualitative identification, preparative TLC, *Sprekelia formosissima*, amaryllidaceae alkaloids 22
- 93 068 S.-H. JIANG, P.-M. YANG*, H. ZHOU, D.-Y. ZHU (*Shanghai Institute of Pharmaceutical Industry, 1320 Beijing Road (w.), Shanghai 200040, P. R. China): Two norditerpenoid ester alkaloids from *Aconitum bulleyanum*. *Planta med.* **68**, 1147-1149 (2002). Analytical and preparative TLC of talatisamine, 8-O-acetyl-14-p-methoxybenzoate and 14-p-methoxybenzoate of talatisamine on silica gel with chloroform - methanol 20:1. Visualization by derivatization with Dragendorff's reagent.
- Pharmaceutical research, herbal, qualitative identification, preparative TLC, *Aconitum bulleyanum* 22
- 93 069 S. KOUL, T. K. RAZDAN*, C. S. ANDOTRA, A. K. KALLA, S. KOUL, S. C. TANEJA (*Department of Chemistry, University of Jammu, Ambedkar Road, Jammu-180006, India): Benzophenanthridine alkaloids from *Corydalis flabellata*. *Planta med.* **68**, 262-265 (2002). TLC of 6-(2-hydroxyethyl)-5,6-dihydrosanguinarine, 6-acetyl-5,6-dihydrosanguinarine, N-methyl-2,3,7,8-tetramethoxy-5,6-dihydrobenzophenanthridine-6-ethanoic acid, N-methyl-2,3,7,8-tetramethoxy-6-oxo-5,6-dihydrobenzophenanthridine, oxosanguinarine, spallidamine, 6-acetyl-5,6-dihydrochelerythrine, 6-oxochelerythrine, and sanguidimerine on silica gel with benzene - ethyl acetate 9:1, 8:2 and 7:3.
- Pharmaceutical research, qualitative identification, benzophenanthridine alkaloids, *Corydalis flabellata* 22
- 93 070 A. PACI*, L. MERCIER, P. BOURGET (*Dept. of Clinical Pharm., Inst. Gustave Roussy, 39 Rue Camille Desmoulins, F-94805 Villejuif Cedex, France. apaci@igr.fr): Rapid analysis of vincal alkaloids in infusion bags. *CBS* **91**, 2-4 (2003). HPTLC on silica gel with dichloromethane - methanol 93:7 over 50 mm after preconditioning for 10 min. Quantitative determination by absorbance measurement at 274 nm. Repeatability is better than 3.5 % and intermediate precision better than 5.1 %.
- Pharmaceutical research, quality control, herbal, densitometry 22
- 93 071 Y. SHELUDKO, I. GERASIMENKO, H. KOLSHORN, J. STÖCKIGT* (*Department of Pharmaceutical Biology, Institute of Pharmacy, Johannes Gutenberg-University, Staudinger Weg 5, D-55099 Mainz, Germany): Isolation and structure elucidation of a new indole alkaloid from *Rauwolfia serpentina* hairy root culture: The first naturally occurring alkaloid of the raumacline group. *Planta med.* **68**, 435-439 (2002). TLC of 10-hydroxy-N(a)-demethyl-19,20-dehydro-raumacline on silica gel with ethyl acetate - methanol - water - ammonia 70:20:10:1, chloroform - methanol - water - ammonia 50:45:5:1 and 50:40:10:1. Detection under UV light at 254 nm and after spraying with ceric ammonium sulfate (CAS) reagent.
- Pharmaceutical research, qualitative identification, *Rauwolfia serpentina*, indole alkaloid 22
- 93 072 C.-H. TAN, B.-D. WANG, S.-H. JIANG, D.-Y. ZHU* (*State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Shanghai Institute of Biological Sciences, Chinese Acade-

my of Sciences, 294 Taiyuan Road, Shanghai 200 031, P. R. China): New Lycopodium alkaloids from *Huperzia serrata*. *Planta med.* **68**, 188-190 (2002). TLC of 11a-hydroxyphlegmariurine B, 7a-hydroxyphlegmariurine B, 7a,11a-dihydroxyphlegmariurine on silica gel with chloroform - methanol 12:1. Detection by iodine vapor.

Pharmaceutical research, qualitative identification,
Huperzia serrata, lycopodium alkaloids

22

93 073 W. TERNES* (*Center for Food Sciences, Veterinary School of Hannover, Postfach 71 11 80, D-30545 Hannover, Germany, waldemar.ternes@tiho-hannover.de): HPTLC method for the determination of ergot alkaloids in grain, flour, and bread. *CBS* **82**, 12-15 (1999). HPTLC of ergometrine, ergotamine, and ergocryptine on silica gel with dichloromethane - methanol 9:1 with chamber saturation. Quantification by fluorescence measurement at 245/>310 nm.

Quantitative analysis, densitometry, ergometrine, ergotamine, ergocryptine 22

93 074 M. VANHAELEN*, J. DUCHATEAU, R. VANHAELEN-FASTRÉ, H. JAZIRI (*Laboratory of Pharmacognosy and Bromatology, Pharmaceutical Institute, Université Libre de Bruxelles, CP 205-4, Bd. du Triomphe, 1050 Bruxelles, Belgium): Taxanes in *Taxus baccata* pollen: Cardiotoxicity and/or allergenicity? *Planta med.* **68**, 36-40 (2002). Analytical and preparative TLC of taxine B, paclitaxel, baccatin III, 10-deacetyl baccatin III on silica gel with chloroform - methanol 24:1. Visualization by spraying with Dragendorff and iodoplatinate reagent or a 3 % sulfuric acid ethanolic solution and heating at 120 °C for 5 min.

Pharmaceutical research, qualitative identification, *Taxus baccata*, taxanes

22

93 063 C. C. W. WANJALA, B. F. JUMA, G. BOJASE, B. A. GASHE, R. R. T. MAJINDA* (*Department of Chemistry, University of Botswana, Private Bag 00704, Gaborone, Botswana): Erythrina alkaloids and antimicrobial flavonoids from *Erythrina latissima*. *Planta med.* **68**, 640-642 (2002). Analytical and preparative TLC of i. a. (+)-10,11-dioxoerysotrine, erysotrine, syringaresinol, erybraedin, neobavaisoflavone, abyssinone IV, neorautenol, isoneorautenol, shinpatercarpin, and erythrinin B on silica gel with chloroform - ethyl acetate 6:1 by 3-fold development. Visualization under UV light at 254 and 366 nm and by spraying with vanillin - sulfuric acid reagent. TLC bioautography for antibacterial and antifungal assays.

Pharmaceutical research, herbal, preparative TLC, qualitative identification,
Erythrina latissima, flavonoids, alkaloids

22, 8a

93 075 X. ZHANG (Zhang Xuelan)*, ZH. ZHANG (Zhang Zhaowang) (*Shangdong Univ. TCM, Jinan 250014, P. R. China): (Comparative study of extraction combination isolated from ingredients of Jiaotai pills by semi-bionic extraction.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (7), 526-529 (2004). TLC on silica gel with benzene - ethyl acetate - methanol - isopropanol - conc. ammonia 20:10:5:5:1. Detection of berberine chloride under UV 365 nm. Identification by standard comparison. Quantification by densitometry at 342 nm. GC of volatile oil and HPLC of cinnamyl aldehyde. Discussion of the optimization of the extraction methods of combined ingredients of the medicine.

Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, semi-bionic extraction,
berberine chloride, volatile oil, cinnamyl aldehyde

22, 4d, 23e

23. Other substances containing heterocyclic nitrogen

93 084 M. BEAUFOUR et al., see section 29f

93 110 H. JEHLE et al., see section 32a

93 075 X. ZHANG (Zhang Xuelan) et al., see section 22

27. Vitamins and various growth regulators

93 076 E. I. PONDER, B. FRIED, J. SHERMA* (*Dep. Chem., Lafayette College, Easton, P.A. 18042, USA): Thin-layer chromatographic analysis of hydrophilic vitamins in standards and *Helisoma trivolvis* snails. *Acta Chromatographica* **14**, 70-81 (2004). TLC of thiamine (B1), riboflavin (B2), niacin (B3), pyridoxine (B6), cobalamin (B12) ascorbic acid (C) and folic acid on silica gel and chemical bonded silica gel with 14 mobile phases, three of which giving the best separation: 1) 1-butanol - chloroform - acetic acid - ammonia - water 7:4:5:1:1, 2) benzene - methanol - acetone - acetic acid 14:4:1:1, 3) chloroform - ethanol - acetone - ammonia 2:2:1:1. Detection in visible light, under UV light at 254 nm and 366 nm. Evaluation of other detection methods such as spraying with 0.5 % ether solution of iodine - Dragendorff reagent for B1, 1 % methanol solution of 1-chloro-2,4-dinitrobenzene followed by 3 M NaOH for B3 and B6, 0.4 % methanol solution of 2,6-dichloroquinone-4-chloroimide for B6, 1:1 mixture of 2 % H₂SO₄ - ethanol and 0.2 % ethanol solution of p-dimethylaminocinnaldehyde for biotin. Quantification by video densitometry. Discussion of identification of vitamins in biological samples by using different visualization method in combination with R_f values.

Traditional medicine, food analysis quality control, densitometry

27

28. Antibiotics, mycotoxins

93 079 L. VICARD*, P. CHARBINAT, F. DUBOST (*Aventis Pharma S.A, LABS, 31-33 Quai Armand Barbès, F-69583 Neuville-sur-Saone, France. louise.vicard@aventis.com): Determination of antibiotics in waste water after biological treatment. *CBS* **90**, 14 (2003.) HPTLC of waste water on silica gel with dichloromethane - methanol - ammonia 90:10:1. Quantitative determination by absorbance measurement at 270 nm.

Environmental, densitometry, quantitative analysis,
qualitative identification, antibiotics

28a, 37c

93 080 I. VOVK*, B. SIMONOVSKA (*National Institute of Chemistry, Hajdrihova 19, SI-1000 Ljubljana, Slovenia, irena.vovk@ki.si, breda.simonovska@ki.si): Cleaning validation by TLC determination of norfloxacin as residue on pharmaceutical equipment surfaces. *CBS* **89**, 2-3 (2002). HPTLC of norfloxacin on silica gel (prewashed with methanol - chloroform 1:1) with methanol - chloroform - ammonia 51:34:15 in horizontal developing chamber with sandwich configuration. Quantitative determination by fluorescence measurement at 313/>400 nm.

Quality control, quantitative analysis, densitometry,
cleaning validation, norfloxacin

28a, 37

93 081 CH. WEINS* (*Staatliches Institut für Gesundheit und Umwelt des Saarlandes, D-66117 Saarbrücken, Germany): Detection of antibiotic substances in municipal waste water treatment facilities. *CBS* **85**, 10-11 (2000). HPTLC-AMD of water samples on silica gel with 33-step gradient from acetonitrile - ammonia to dichloromethane and dichloromethane - acetic acid to n-hexane. Detection via Chrom Biodip (Merck) by dipping in bacteria solution (*Bacillus subtilis*) followed

Parameters of Planar Chromatography

The articles in this series are dedicated to the important steps of planar chromatography and their parameters which influence the chromatographic result. Hints for optimization are given to help the reader to use planar chromatography most efficiently.

Collecting these pages is recommended.

Derivatization

The possibility of convenient post chromatographic specific or non-specific chemical derivatization which often needs to be completed by a heating step is a particularly strong point of TLC.

Reasons for choosing derivatization as a step in TLC include:

- Changing non-absorbing substances into detectable derivatives
- Improving the detectability (lowering detection limits)
- Detecting all sample components
- Selectively detecting certain substances
- Inducing fluorescence

A variety of derivatizing agents is available (table).

From a technical point of view only one principal decision must be made: how to transfer the reagent to the plate? Derivatization can be achieved through the gas phase, by spraying or by dipping.

Derivatization through gas phase

Derivatization through the gas phase offers rapid and uniform transfer of the reagent. It is unfortunate, that only few reagents are suitable. They include iodine, bromine and chlorine, as well as volatile acids, bases and some other gases like H_2S , and NO . In practice gas phase derivatization can be easily accomplished in twin trough chambers where the reagent is placed or generated (e.g. chlorine from HCl and permanganate) in the rear trough, while the plate facing the inside of the chamber is positioned in the front trough.

Spraying

Spraying is most widely used for reagent transfer onto the TLC plate. It is simple and quick. No expensive equipment is needed and only small volumes of reagent are needed. Spraying is very flexible and indispensable when reagents have to be applied in sequence. Also during method development, when searching for the most suitable reagent, spraying is the primary choice. On the other side spraying generates substantial amounts of obnoxious and hazardous fumes, which must be carefully removed using a spray cabinet. Another problem of spraying is associated with achieving a homogenous and defined derivatization across the plate. Particularly for quantitative evaluation spraying requires great skills in order to obtain reproducible results.

The sprayer must generate a very fine mist and avoid any sputtering. If compressed air is used proper performance is often also a function of the correct gas pressure and the distance of the spray nozzle from the surface of the plate. At the beginning the generated mist is directed somewhere beside the plate until it is getting fine and homogenous. While spraying onto the plate the head of the sprayer is slowly moved in a meander pattern across the plate, typically first up and down, then left to right. Thereby the turning points are located outside the plate. The amount of reagent distributed is difficult to control, but with enough practice it is possible to achieve consistent results.

Dipping

By immersing a TLC plate into the derivatizing reagent a very homogenous reagent transfer can be achieved. Dipping and withdrawing has to be performed smoothly in order to avoid tidemarks. Using an immersion device the reproducibility of the derivatization step can be significantly improved compared to spraying.

The concentration of the reagent is easily maintained and by adjusting the vertical speed and immersion time the derivatization conditions can be standardized. No fumes are generated during this derivatization technique and the exposure to hazardous chemicals is limited. After immersion some excess liquid usually covers the plate. To avoid unnecessary broadening of separated zones the plate should be dried as quickly as possible.

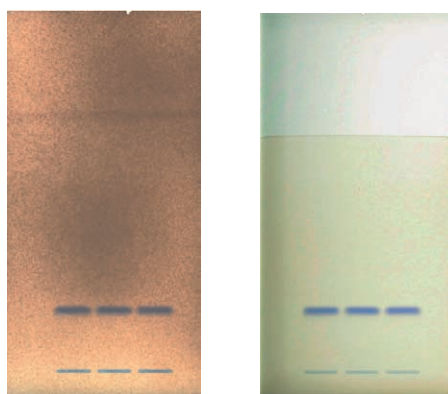


Fig. 1 Derivatization of capsaicin with dichloroquinone chloroimide reagent/ammonia by spraying (1 g/L, left side) and by dipping (0.25 g/L, right side)

In most cases the same kind of reagents can be used for spraying and dipping (Fig. 1), however, the dipping reagents are typically less concentrated (up to ten times, mostly four times) and should be dissolved in less polar solvents.

Heating

Most chemical reactions used in derivatization require heating for completion. The two principal heating devices are ovens and plate heaters. In the daily routine and for quantitative analyses it is most important that the plate is heated evenly and reproducibly. Ovens have two major shortcomings. Due to the (sometimes) aggressive fumes, which are produced during derivatization, corrosion and also cross contamination become an important issue. The other problem is that the derivatization process can usually not be

observed. Plate heaters became a commonly used alternative. These instruments maintain a uniform temperature across their surface. When a plate is placed on the hot surface at temperatures higher than 120 °C glass plates will significantly bend on their edges due to low heat conductivity. This results in more efficient heating at the center of the plate. Within a few minutes these irregularities are usually leveled. For reproducible results the temperature of the derivatization should therefore be adjusted so that the plate is heated at least five minutes.

Conclusion

In practice it seems important to consider the following:

- Derivatization is most often performed to visualize a chromatogram. Depending on the analytical goal and on the type of subsequent evaluation, reproducibility of the result may or may not be important.
- Generally, the outcome of a chemical derivatization – particularly the color and/or fluorescence of derivatized zones – is affected by heating time/temperature and reagent concentration, which can be controlled easily. Other factors, such as drying (temperature and duration) of the developed plate prior to derivatization are sometimes overlooked but can also affect the result (Fig. 2). Therefore the derivatization step has to follow a strict protocol to obtain reproducible results.

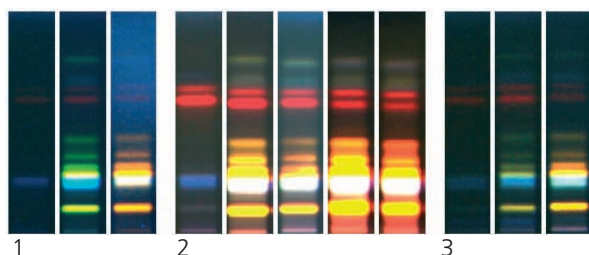


Fig. 2 Derivatization of flavonoids (*St. John's Wort*, *Hypericum perforatum*) with Natural Product reagent (NP) and polyethylene glycol (PEG)

Set 1: drying of plate for 3 min in cold air, left to right: UV 366 nm, NP, NP/PEG

Set 2: drying of plate for 5 min at 105 °C, left to right: UV 366 nm, NP, NP 30 min later, NP/PEG, NP/PEG 30 min later

Set 3: drying of plate for 30 min at 105 °C, left to right: UV 366 nm, NP, NP/PEG

- If visualization is not required, derivatization may not be advantageous. For example, progesterone has a chromophore and absorbs UV light at 254 nm, so it can be analyzed prior to derivatization. If necessary, the substance can be visualized by derivatization with sulfuric acid. The comparison of the standard deviation for the densitometric evaluation of derivatized and non-derivatized progesterone is given below. The best results are obtained without derivatization, derivatization by spraying yields least reproducible results across the plate.

Evaluation of progesterone

Derivatization	No derivatization	Derivatization by immersion	Derivatization by spraying
Densitometry	Abs. 254 nm	Abs. 536 nm	Abs. 536 nm
Progesterone, CV of 16 samples, 6 mm bands	1.1%	2.4%	2.9%

Most common derivatization reagents

Reagent name	Preparation, use	Examination	Detection of
Ammonia vapor	The plate is placed in a chamber saturated with ammonia (25%) vapor. The plate may be heated (opiates, valepotriates).	White light, UV 366 nm	Opiates, mycotoxins, flavonoids, sennosides
Aniline-diphenylamine-phosphoric acid	4 g diphenylamine and 4 mL aniline are dissolved in 160 mL acetone. 30 mL o-phosphoric acid are carefully added. The plate is immersed in the reagent for 1 s, then heated at 120 °C for max. 10 min.	White light	Sugars, glycosides
Anisaldehyde-sulfuric acid	10 mL sulfuric acid are carefully added to an ice-cooled mixture of 170 mL methanol and 20 mL acetic acid. To this solution 1 mL anisaldehyde is added. The plate is immersed in the reagent for 1 s, then heated at 100 °C for 2–5 min.	White light, UV 366 nm	Terpenoids, saponins, sterols, most lipophilic compounds
2,6-Dibromoquinone-(or dichloroquinone)-4-chlorimide (Gibbs' reagent)	50 mg 2,6-dibromoquinone-(or dichloroquinone)-4-chlorimide are dissolved in 200 mL ethyl acetate. The plate is immersed in the reagent, dried in a stream of cold air, then placed in a chamber saturated with ammonia until colors appear.	White light	Arbutin, vitamin B ₆ , phenols, coumarins, thiols, thiones, capsaicin, antioxidants, amines
Dragendorff's reagent	Solution A: 0.85 g basic bismuth nitrate are dissolved in 10 mL acetic acid and 40 mL water Solution B: 8 g potassium iodide are dissolved in 30 mL water. Just before spraying, 1 mL of each solution is diluted with 4 mL acetic acid and 20 mL water.	White light	Alkaloids, heterocyclic nitrogen compounds, polyethylene glycol
Fast blue salt B	0.5 g fast blue salt B are dissolved in 100 mL water. The plate is sprayed or immersed, then dried.	White light, UV 366 nm	Phenolic compounds, tannins, cannabinoids
Iodine (spraying solution) Iodine vapor	0.5 g iodine are dissolved in 100 mL ethanol. The plate is sprayed until background appears light yellow. The plate is exposed to iodine vapor in the chromatographic chamber until zones appear	White light, UV 366 nm (after evaporation of non-bonded iodine)	Conjugated double-bonds, alkaloids, purine derivatives, lipids, carotenoids
Natural products/ polyethylenglycol	Solution A: 1 g of diphenylborinic acid aminoethylester is dissolved in 200 mL ethyl acetate. Solution B: 10 g of polyethylene glycol 400 (macrogol) are dissolved in 200 mL dichloromethane. The plate is heated at 100 °C for 3 min, then dipped while still hot in solution A, dried in a stream of cold air, then dipped in solution B.	UV 366 nm	Flavonoids, carbohydrates, anthocyanines, plant acids
Ninhydrin	0.6 g ninhydrin are dissolved in 190 mL isopropanol, 10 mL of acetic acid are added. Note: Colors produced by the ninhydrin reagent can change with concentration, solvent and modifiers such as collidine, copper sulfate, or cobalt nitrate.	White light	Amino acids, biogenic amines, ephedrine
Phosphomolybdic acid	10 g phosphomolybdic acid are dissolved in 50 mL ethanol. The plate is sprayed and dried. Sometimes heating of the plate is necessary. The color of zones can be optimized by exposing the plate to ammonia vapor.	White light	Fatty oils, phospholipids, reducing substances, steroids, essential oils compounds, morphine
Sulfuric acid	20 mL sulfuric acid are carefully added to 180 mL ice-cold methanol. The plate is dipped in reagent for 1 s, then heated at 100 °C for 5 min.	White light, UV 366 nm	General reagent
Vanillin-inorganic acid	Diverse concentrations are used, mixed with acetic, hydrochloric, phosphoric, sulfuric acid. The plate is sprayed, then heated at 100 °C for 5 minutes.	White light, UV 366 nm	Terpenoids, sterols, salicin, ergot alkaloids, most of lipophilic compounds

Further details about derivatization can be found in:

Jork H., Funk W., Fischer W., Wimmer H.: Thin-Layer Chromatography, Volume 1a and 1b, Physical and chemical detection methods, VCH Verlagsgesellschaft mbH, Weinheim, 1990 and 1993

Wagner H., Blatt S.: Plant Drug Analysis, A Thin-Layer Chromatography Atlas, 2nd Edition, Springer, Berlin, 1995

Stahl E.: Dünnschicht-Chromatographie, Ein Laboratoriumshandbuch, 2nd Edition, Springer, Berlin, 1967



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by incubation at 30 °C over night and spraying with MTT-tetrazolium salt reagent followed by incubation at 30 °C for 5-30 min. Visual evaluation.

Pharmaceutical research, environmental, AMD, qualitative identification, antibiotics, waste water, *Bacillus subtilis*, Chrom Biodip, bio-autography 28a, 37

- 93 082 M. WERTHER* (*Institut für Tierarzneimittel GmbH, Berliner Allee 317-321, D-13088 Berlin, Germany, margit.werther @camag-berlin.de): Validated procedure for determination of Monensin-Na in animal feed. CBS **89**, 10-11 (2002). HPTLC on silica gel with 1) ethyl acetate - chloroform 1:1 over 30 mm followed by drying at room temperature for 15 min and 2) ethyl acetate - chloroform 9:1 over 50 mm in horizontal development chamber. Detection by dipping in anisaldehyde - sulfuric acid reagent followed by heating at 60 °C for 5 min. Quantitative determination by absorbance measurement at 440 nm. Precision (n=6 at 100 mg/kg feed) is found to be 2.7 %.
- Food analysis, quality control, densitometry, quantitative analysis, monensin-Na, feed additives 28a

- 93 077 SH. HEIDARI* (*Institute of Standards and Industrial Research of Iran, P.O. Box 31585-163, Industrial City, Karaj, Iran): Detection of aflatoxins in pistachios. CBS **84**, 9 (2000). HPTLC of aflatoxins on silica gel with chloroform - acetone 9:1. Quantitative determination by fluorescence measurement at 366/>400 nm.
- Food analysis, quality control, AMD, quantitative analysis 28b

- 93 078 J. SKARKOVA*, V. OSTRY, J. RUPRICH (*National Institute of Public Health, Prague, Palacko 1-3, 61242 Brno, Czech Republic, skarkova@chpr.szu.cz): Application of planar chromatography for determination of mycotoxins in the Czech Republic. CBS **87**, 1-3 (2001). HPTLC of aflatoxins, ochratoxin A, patulin, deoxynivalenol, fumonisins, sterigmatocystin, cyclopiazonic acid, and altenuene on silica gel with various mobile phases. Detection by spraying with aluminum chloride or 4-methoxybenzaldehyde. Quantitative determination by fluorescence or absorbance measurement at various wavelengths.
- Food analysis, agricultural, quality control, densitometry 28b

29. Insecticides, pesticides and other agrochemicals

- 93 083 P. P. BERNY*, J. SYNARDET, D. VEY, (*Toxicology Laboratory of the Ecole Nationale Veterinaire de Lyon ENVL, 1, Avenue Bourgelat, BP 83, F- 69280 Marcy L' Etoile, France): Planar chromatography in veterinary toxicology. Analysis of choline esterase inhibitors. CBS **83**, 4-5 (1999). HPTLC-AMD of choline esterase inhibitors on silica gel with a 15-step gradient from methanol via dichloromethane to hexane. Detection by spraying with butyryl choline esterase solution followed by incubation at 37 °C for 30 min, and spraying with fast blue salt mixed with alpha-naphthyl acetate followed by incubation at 37 °C for 5 min. Quantitative determination by absorbance measurement at 254 nm.
- Toxicology, AMD, quantitative analysis, densitometry, choline esterase inhibitors, aldicarb, methomyl 29, 32d
- 93 084 M. BEAUFOUR*, J.-C. CHERTON, A. CARLIN-SINCLAIR (*Laboratoire Sircob, Bat. Lavoisier, 45 Avenue des Etats-Unis, F-78035 Versailles Cedex, France. beaufour@chimie.uvsq.fr): Monitoring of proinsecticides (oxazolines) in biological samples. CBS **90**, 12-13 (2003). TLC of oxazoline Ia on aluminum sheets RP-18 with methanol over 10 mm, followed by water - acetonitrile 1:1 over 55 mm. Ion pair chromatography of oxazoline Ia with phosphate buffer -

acetonitrile 1:1 over 60 mm after dipping in 2 mM cetyltrimethylammonium bromide. TLC of oxazoline Ib and Ic with phosphate buffer - acetonitrile - dioxane 4:3:3 over 65 mm. Determination by absorbance measurement at 200 nm (oxazoline Ia and Ib) and 262 nm (oxazoline Ic) respectively.

Agricultural, densitometry, quantitative analysis, qualitative identification, comparison of methods, oxazolines, proinsecticides 29f, 8b, 23e

93 006 W. KREISS et al., see section 3e

30. Synthetic and natural dyes

93 085 J.A. THAKARDA*, D. SHAH, M. TRIVEDI, B. BHAVSAR (*Dintex Dyechem Research Centre, Ahmedabad, Gujarat, India): Qualitative and quantitative analysis of impurities in MAMASA using HPTLC. CBS **86**, 4-5 (2001). HPTLC of 3-methoxy-4-amino azo meta sulphonic acid (MAMASA) on silica gel with n-butanol - diethylamine - ammonia - methanol 9:5:5:2 with chamber saturation for 5 min followed by drying at 40 °C. Quantitative determination by absorbance measurement at 254 nm.

Densitometry, quantitative analysis, azobenzenes, textile dyes, MAMASA, banned amines 30a, 11a

93 086 M. WERTHER (*Institut für Tierarzneimittel GmbH, Berliner Allee 317-321, D-13088 Berlin, Germany, margit.werther@camag-berlin.de): Identification of water-soluble food colors. CBS **88**, 7 (2002). HPTLC of E 110 Yellow orange S, E 104 Quinoline yellow, E 124 Cochineal red, E 132 Indigotine I, E 151 Brilliant black BN on RP-18 W with methanol - 5 % aqueous sodium sulfate solution 3:4. Identification based on R_f-values, documentation under white light.

Food analysis, qualitative identification, food colors 30a

93 008 D. JÄNCHEN* (Ed.), see section 4d

93 055 W. XIA (Xia Wei) et al., see section 15a

32. Pharmaceutical and biomedical applications

93 087 M. BAKAVOLI*, M. KAYKHAI, (*Dept. of Chem., Fac. of Sci., Ferdowsi Univ., Mashad, 91779, Iran. mbakavoli@yahoo.com): Quantitative determination of diazepam, nitrazepam and flunitrazepam in tablets using thin-layer chromatography-densitometry technique. J. Pharmaceut. Biomed. Analysis **31**, 1185-1189 (2003). TLC on silica gel with chloroform - acetone 9:1 in an unsaturated chamber. Quantitation by densitometry at 254 nm. Comparison of TLC method with reversed phase HPLC analysis.

Diazepam, nitrazepam, flunitrazepam 32a

93 109 D. JÄNCHEN* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland, dieter.jaenchen@camag.com): Process control by HPTLC - a case study. CBS **84**, 12-13 (2000). HPTLC of synthesis product SR 94163 and impurities SR 94 145 and SR 94454. Quantitative determination at 210 nm. Recovery rate for impurities is 98-114 %. For routine analysis in process control the

HPLC method was substituted by the faster HPTLC method.

Pharmaceutical research, densitometry, quantitative analysis, process control 32a

93 001 D. JÄNCHEN* (Ed.), see section 2a

93 110 H. JEHL*, I. MESAROS, S. SIGGERT, B. WOCHNER, D. KLEIBER, N. SINNER (*Quality Control Non-Sterile Products, Altana Pharma AG, Byk-Gulden-Str. 2, D-78467 Konstanz, Germany, Harald.jehle@altanapharma.com): Content uniformity test of cinchocaine hydrochloride. *CBS* **92**, 1-3 (2004). HPTLC of cinchocaine hydrochloride from suppositories on silica gel with 1-butanol - toluene - ethanol - water - 100 % acetic acid 10:8:7:4:1 over 30 mm followed by heating at 110 °C for 10 min. Quantitative determination by fluorescence measurement at 313/>400 nm. Precision is 2 % and recovery 101 %.

Quality control, cinchocaine hydrochloride 32a, 23e

93 122 N. MAHADEVAN*, M.M. HIPOLITH VIJI, T. SUBBURAJU, B. SURESH (*Dept. of Pharmacognosy, J.S.S. College of Pharmacy, Ootacamund – 643 001, India): Estimation of berberine in herbal extract and poly herbal formulations by HPTLC. *Indian Drugs* **41**(1), 46-47, (2004). HPTLC on silica gel plates with n-propanol - formic acid - water 90:1:9. Rf of berberine was 0.39. Quantification by densitometry at 348 nm via linear regression in the range of 40-220 ng. Limit of quantification and limit of detection were found to be 30 ng and 10 ng, respectively. The herbal extract was found to contain 0.402 % w/w of berberine. The percentage of berberine in formulations I, II, III and IV were 0.0886 %, 0.0123 %, 0.0283 % and 0.0091 % w/w, respectively. The standardized method was found to be reproducible, accurate, precise and selective.

Pharmaceutical research, quality control, qualitative identification, densitometry, postchromatographic derivatization 32a

93 127 K. R. MAHADIK *, H. AGGARWAL, N. KAUL (*Department of quality Assurance Technique, Bharati Vidyapeeth Deemed University, Poona College of Pharmacy, Erandwane, Pune – 411038, India): Simultaneous HPTLC estimation of amlodipine besylate and losartan potassium in tablet dosage form. *Indian drugs* **41** (1), 32-35, (2004). HPTLC on silica gel 60 F254 aluminium foil with toluene - methanol - chloroform 4:5:11 with chamber saturation for 30 min at room temperature. Rf of amlodipine besylate and losartan potassium was found to be 0.14 and 0.32, respectively. Quantification by densitometry at 245 nm via linear regression for amlodipine besylate between 0.05 to 0.10 µg and for losartan potassium between 0.5 to 1 µg. Validation regarding to accuracy and precision was performed. The limit of detection and limit of quantification for amlodipine besylate was found to be 0.01 and 0.03 ng/µL - for losartan potassium it was 0.004 and 0.013 ng/µL. The average recovery was determined to be 101.15 % for amlodipine besylate and 100.74% for losartan potassium.

Pharmaceutical research, quality control, densitometry, qualitative identification comparison of methods 32a

93 126 A. PACI* (*Dept. of Clinical Pharm., Inst. Gustave Roussy, 39 Rue Camille Desmoulins, F-94805 Villejuif Cedex, France. apaci@igr.fr): HPTLC applied to the post-production quality assurance of platinum infusion solutions. *CBS* **86**, 7 (2001). HPTLC on silica gel in horizontal development chamber with acetone – water 9:1 with chamber saturation for 15 min. Detection by dipping 1 s in 0.02 % 4-nitrosodimethylaniline in ethanol with 0.01 N HCl (adjusted at pH 2) followed by heating at 150 °C. Quantitative determination by absorbance measurement at 495 nm.

Pharmaceutical research, clinical routine analysis, quality control, densitometry 32a

93 129 E. REICH* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland): HPTLC

applied to the post-production quality assurance of etoposide infusion solutions. CBS **86**, 6 (2001). HPTLC of etoposide solution (in 5% dextrose or 0.9% NaCl) on silica gel in horizontal development chamber in sandwich configuration with diethyl ether – dichloromethane – methanol 24:20:1. Quantitative determination by absorbance measurement at 290 nm.

Pharmaceutical research, clinical routine analysis, quality control, densitometry 32a

- 93 133 SAPNA SHRIKUMAR*, S. SANDEEP, T. K. RAVI, M. UMAMAHESWARI (*Department of Pharmaceutical Analysis, College of Pharmacy, Shri Ramkrishna Institute of Paramedical Sciences, Coimbatore – 641 004, India): A HPTLC determination and fingerprinting of bacoside A in *Bacopa monnieri* and its formulation. Indian Journal of Pharmaceutical Sciences **66** (1), 132-135, (2004). HPTLC of bacoside A in *Bacopa monnieri* and in its commercial monoherbal capsule formulation on silica gel 60 F254 aluminium sheet, 20 x 10 cm, with chloroform – methanol – water 30:15:1. Quantification by densitometry at 540 nm via linear regression in the range of 30–180 µg/mL. Rf of Bacoside A was found to be 0.51. Validation regarding to accuracy, precision, specificity and recovery (97.4–100.1%). The proposed simple and sensitive method provides faster and cost effective qualitative control for routine analysis of bacoside A in formulations containing *Bacopa monnieri* saponins.

Pharmaceutical research, quality control, qualitative identification, densitometry comparison of methods

32a

- 93 141 L. VICARD*, L. CHÉRUZEL (*Hoechst Marion Roussel, Neuville-sur-Saône, France): Process monitoring by example of an esterification reaction. CBS **83**, 14-15 (1999). HPTLC of a mixture of the reaction product and its educts on cyano phase with cyclohexane - ethyl acetate - acetic acid 400:100:1 over 40 mm. Quantitative determination by absorbance measurement at 233 nm.

Densitometry, quantitative analysis, process monitoring, fine chemicals, synthesis parameters

32a, 35

- 93 144 M. WERTHER* (*Institut für Tierarzneimittel GmbH, Berliner Allee 317-321, D-13088 Berlin, Germany, margit.werther@camag-berlin.de): Determination of sulfadimidine-Na in cow and pig. CBS **87**, 4-5 (2001). HPTLC on silica gel with ethyl acetate - chloroform - methanol 6:4:1. Detection by dipping in fluorescamine solution. Quantitative determination by fluorescence measurement at 365/>400 nm.

Pharmaceutical research, quantitative analysis, densitometry, sulfadimidine-Na 32a

- 93 091 S. CAI (Cai Songtao), H. OUYANG (Ouyang Huifang), J. LUO (Luo Jie) (Guangzhou Baiyunshan TCM Factory, Guanzhou 510510, P. R. China): (Study of the quality standard of Ganmaoling tablets.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (12), 964-967 (2004). TLC on silica gel with 1) chloroform - methanol - acetone - formic acid - water 54:14:8:6:1, 2) toluene (saturated with water) - ethyl acetate - formic acid 5:4:1, 3) cyclo hexane - ethyl acetate 5:1. Detection 1) by iodine vapor, 2) under UV 365 nm. Identification by fingerprint techniques. Quantitative determination of acetaminophen and chlorogenic acid by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification, acetaminophen, chlorphenamine maleate, caffeine, chlorogenic acid

32c, 4d

- 93 093 L. CHEN (Chen Lihua), SH. OUYANG (Ouyang Sheng), Y. MAO (Mao You Chang) (Jianxi Coll. TCM, Nanchang, Jiangxi 330006, P. R. China): (Determination of andrographolide

in Qianxi capsules by thin-layer chromatography.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (12), 1017-1019 (2004). TLC on silica gel developed with chloroform - ethyl acetate - methanol 20:15:2 at a temperature lower than 28 °C. Detection under UV 254 nm, or by spraying with 2 % 3,5-dinitrobenzoic acid in ethanol - 2 M KOH 1:1. Identification by standard comparison. Quantitative determination by densitometry at 263 nm. Validation of the method by investigation of its linearity range, precision, repeatability, reproducibility, and recovery. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, traditional medicine, quality control,
densitometry, quantitative analysis

32c

- 93 095 X. CHENG (Cheng Xiumin)*, J. REN (Ren Jian), Y. GAO (Gao Yanhui) (*Pharm. Coll., Shandong Univ., Jinan, Shandong 250012, P. R. China): (An improved method for quick identification of occident ginsengs by thin-layer chromatography.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (8), 665-666 (2004). TLC on silica gel with chloroform - acetone - formic acid 10:5:2. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 80 °C, 2) under UV 365 nm, 3) daylight, and 4) by densitometry at 543 nm. Identification by fingerprint techniques. Running the procedure for 16 ginsengs cultivated in different regions.

Pharmaceutical research, traditional medicine, quality control,
qualitative identification, ginsengs

32c

- 93 097 X. CUI (CUI XIUJUN)*, L. CHENG (CHENG LIFANG) (*Jinan Municip. Hygienical Station for Women & Children, Jinan, Shandong 250001, P. R. China): (Determination of ligustilide in Chanshukang capsules by thin-layer chromatography.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 73-74 (2004). TLC on silica gel with petroleum ether - ethyl acetate 19:1. Detection under UV 365 nm. Identification by standard comparison. Quantification of ligustilide by densitometry at 290 nm. Validation of the method by investigation of its precision (RSD 2.4 % n=5 within plate and 3.0 % n=5 plate-to-plate), reproducibility (2.5 % n=6 within three hours), and recovery (96.7 %, RSD 2.2 % n=5).

Pharmaceutical research, quality control, quantitative analysis,
densitometry, ligustilide

32c

- 93 099 U. DEMME*, R. WERNER, CH. ARNDT (*Hospital of the Friedrich-Schiller University of Jena, Institute for Forensics, Fürstengraben 23, D-07743 Jena, Germany): Improved separation of benzodiazepines by AMD. *CBS* **91**, 5-7 (2003). HPTLC-AMD of benzodiazepines from serum on lichrospher silica gel with a 9-step gradient based on methanol and diisopropyl ether over 80 mm. Densitometric evaluation by absorbance measurement at 230 and 320 nm followed by spectra recording from 200 to 330 nm.

Pharmaceutical research, toxicology, clinical routine analysis, AMD

32c

- 93 101 L. DONG (Dong Lin)*, Y. LI (Li Yi), X. DENG (Deng Xiaohong) (*Sichuan Prov. Inst. Drug Cont., Chengdu, Sichuan 610036, P. R. China): (Study of the quality standard for Yinchai granules.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 29-32 (2004). TLC on silica gel with 1) chloroform - ethyl acetate - methanol - water 15:40:22: 10, 2) chloroform - methanol - water 13:7:2. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C, 2) under UV light. Identification by fingerprint technique. Quantification of chlorogenic acid by HPLC.

Pharmaceutical research, herbal, quantitative analysis, densitometry,
chlorogenic acid

32c, 4d

- 93 102 ZH. DU (Du Zhiqian), G. LI (Li Genlin) (Luoyang Osteopathy Hosp., Luoyang Henan 471002, P. R. China): (Determination of tetrahydropalmatine in Gupi plaster.) (Chinese). *J. Chinese Trad. Patent Med.*, (Zhongchengyao) **10**, 862-864 (2004). TLC on silica gel with cyclo hexane - chloroform - water 65:35:2. Detection by iodine vapor and under UV 254 nm. Identification by standard comparison. Quantitative determination by densitometry at 254 nm. Validation of the procedure by investigation of its linearity range, interference with impurities, precision, reproducibility and recovery. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.
Pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis 32c
- 93 104 Y. GU (Gu Ying)*, Y. GUO (Guo Yubao), H. WANG (Wang Huahong), Y. ZHANG (Zhang Ye) (*Shanxi Acad. TCM, Xi'an, Sahnxi 710003, P. R. China): (Study of the quality control of Shuangxin granules.) (Chinese). *J. Chinese Trad. Patent Med.* (Zhongchengyao) **25** (9), 711-715 (2004). TLC on silica gel with 1) ethyl acetate - butanone - formic acid - water 5:3:1:1, 2) chloroform - methanol - formic acid 200:25:50:1, 3) petroleum ether - diethyl ether 2:1, 4) chloroform - methanol - acetone - glacial acetic acid 30:1:2:3, 5) ethyl acetate - formic acid - glacial acetic acid - water 15:1:1:2. Detection 1) by spraying with 1 % FeCl₃ in ethanol, 2) spraying with 5 % vanillin - H₂SO₄ solution and heating, 3) under UV 365 nm, 4) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C. Identification by fingerprint techniques. Quantification of baicalin by HPLC.
Pharmaceutical research, traditional medicine, quality control, qualitative identification, quantitative analysis, baicalin 32c
- 93 107 G. HAN (Han Guiru)*, R LIU (Liu Renliu) (*Hebei Prov. Inst. Drug Cont., Shijiazhuang, Hebei 050011, P. R. China): (Study of the quality standard of Xiaoerhuichun pills.) (Chinese). *J. Chinese Trad. Patent Med.* (Zhongchengyao) **25** (9), 715 -718 (2004). TLC on silica gel with 1) ethyl acetate - butanone - formic acid - water 5:3:1:1, 2) benzene - ethyl acetate - methanol - isopropanol - conc. ammonia 12:6:3:3:1, 3) chloroform - acetone 19:1, 4) n-butanol - glacial acetic acid - water 7:1:2. Detection 1) by spraying with 5 % FeCl₃ in ethanol, 2) spraying with 5 % vanillin - H₂SO₄ solution and heating at 100 °C for 10 min, 3) under UV 365 nm. Identification by fingerprint techniques. Quantification of berberine hydrochloride by densitometry at 340 nm.
Pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification 32c
- 93 108 N. HUANG (Huang Nojia), W. YANG (Yang Wenhong), SH. XIAO (Xiao Shurong) (Shantou Municip. Inst. Drug Cont., Shantou, Guangdong 515041, P. R. China): (Study of the quality standard of Jiujixingjunsan powder.) (Chinese). *J. Chinese Trad. Patent Med.* (Zhongchengyao) **11**, 874-877 (2004). TLC on silica gel with 1) ethyl acetate - butanone - formic acid - water 5:3:1:1, 2) 2-fold development with ethyl acetate - methanol - water 100:17:13 and toluene - ethyl acetate - formic acid - water 20:10:1:1, 3) chloroform - ethyl acetate - methanol - formic acid 200:25:50:1. Detection 1) by spraying with 1 % FeCl₃ in ethanol, 2) by spraying with 3 % AlCl₃ in ethanol and under UV 365 nm, 3) by spraying with 10 % H₂SO₄ in ethanol and heating. Identification by fingerprint techniques. Quantitative determination of mentholum, borneolum syntheticum by GC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.
Pharmaceutical research, traditional medicine, quality control, qualitative identification, mentholum, borneolum syntheticum 32c, 4d
- 93 114 DE LI (Li Dexun)* (*Zahotong Inst. Drug Cont., Zhaotong, Yunnan 657000, P. R. China): (Quick identification of Zhuabggu Guanjie pills by thin-layer chromatography.) (Chinese). *J. Chinese*

Trad. Patent Med. (Zhongchengyao) **25** (8), 682-682 (2004). TLC of icanine and naringoside on silica gel with ethyl acetate - butanone - methanol - water 10:6:3:1. Detection by spraying with 5 % AlCl₃ in ethanol and heating at 105 °C for 5 min, and under UV 365 nm. Identification by standard comparison.

Pharmaceutical research, traditional medicine, quality control,
qualitative identification, icanine, naringoside

32c

- 93 115 SH. LI (Li Shouzhuo)*, H. PAN (Pan Haifeng) (*Inst. TCM, Chengde Coll. Med., Chengde, Hebei 067000, P. R. China): (Determination of psoralen and isopsoralen in Longbishu capsules by thin-layer chromatography.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (8), 627-629 (2004). TLC on silica gel 2-fold with petroleum ether - ethyl acetate 9:1. Detection 1) under UV 254 nm. Identification by comparison with the standards. Quantitation by densitometry at 300 nm. Validation of the procedure by investigation of its precision, specificity, repeatability, reproducibility, recovery, etc.

Pharmaceutical research, traditional medicine, quality control,
densitometry quantitative analysis

32c

- 93 116 X. LI (L Xiaoyan)*, SH. ZHAO (Zhao Shaohua), X. LU (Lu Xiuli), ZH. LI (Li Zhengjie) (*Hebei Yiling Acad. Med. & Pharm., Shijiazhuang, Hebei 050035, P. R. China): (An improved procedure for the determination of matrine in Chinese traditional herbal drugs and preparations.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **26** (2), 172-173 (2004). TLC on silica gel with 1) benzene - acetone - ethyl acetate - conc. ammonia 10:15:20:2. Detection by spraying with 5 % potassium iodobismuthate solution. Identification by comparison with the standard. Quantification by densitometry at 524 nm. The procedure is validated by investigation of its repeatability, reproducibility, and recovery. Discussion of the merits of the procedure compared to that in China Pharmacopeia (2000 ed).

Pharmaceutical research, quality control, qualitative identification,
densitometry, matrine

32c

- 93 118 G. LIN (Lin Guitao)*, CH. MA (Ma Chengyan), L. ZHAO (Zhao Junling) (*Shandong Univ. TCM, Jinan, Shandong 250014, P. R. China): (Study of the quality standard for Shexiangtongbi Babu plaster.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **26** (2), 100-102 (2004). TLC on silica gel with 1) petroleum ether - ethyl acetate 17:3, 2) n-hexane - ethyl acetate 8:1, 3) petroleum ether - ethyl acetate 10:3, 4) chloroform - methanol 19:1, 5) n-hexane - ethyl acetate 9:1. Detection 1) by spraying with 5 % vanillin - H₂SO₄ solution and heating at 105 °C, 2) under UV 365 nm, 3) under daylight. Identification by fingerprint techniques. Quantification of muscone by GC.

Pharmaceutical research, quality control, qualitative identification, muscone 32c

- 93 119 X. LIU (Liu Xin)*, Y. LIN (Lin Yu), Y. CHEN (Chen Yun), J. YANG (Yang Junxuan), W. PENG (Peng Wanying) (*Chongqing Univ. Med., Chongqing, Sichuan 400016, P. R. China): (Determination of dl-tetrahydropalmatine in Chinese traditional patent medicines by thin-layer chromatography.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **26** (1), 19-23 (2004). TLC on silica gel in a basic developing system containing diethylamine. Detection by iodine vapour. Identification by standard comparison. Quantitative determination by densitometry at 340 nm. Validation of the method by investigation of the procedures, including sample preparation, mobile phase, visualization, scanning wavelength, linearity range (0.28-1.65 µg, r=0.9991), precision (RSD <1.4 % within plate and <2.4 % plate-to-plate), resolution, repeatability, reproducibility, and recovery (87.2 %-99.7 % for different medicine). Comparison of the results with those obtained by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

- Pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis 32c, 4d
- 93 120 H LU (Lu Hua)*, J. XING (Xing Jianguo), T. WU (Wu Tao) (*Section Med., Taixing Hosp., Taixing, Jiangsu 225400, P. R. China): (Study of the quality standard of Shentaipikang pills.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (10), 798-801 (2004). TLC on silica gel with 1) ethyl acetate - butanone - formic acid - water 10:7:1:1; 2) benzene - acetone 10:1; 3) petroleum ether - ethyl formate - glacial acetic acid 18:1:1. Detection 1) under UV 365 nm, 2) by spraying with 5 % solution of vanillin - acetic acid - perchloric acid and heating at 105 °C, 3) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C. Identification by fingerprint techniques. Quantitation of icariine by HPLC.
- Pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification, icariine 32c, 4d
- 93 124 SH. MO (Mo Shaohong)*, X. LU (Lu Xiaojun), L. TAN (Tan Liujuan) (*Nanning Boke Pharm. Co., Ltd., Nanning 53002 1, P. R. China): (Study of the quality standard for Shuanghuo tincture.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (8), 619-622 (2004). TLC on silica gel with 1) petroleum ether - ethyl acetate 17:3; 2) chloroform - methanol - water 32:8:1; 3) chloroform - methanol 100:1; 4) chloroform - methanol - water 13:7:2. Detection 1) under UV 254 nm; 2) by spraying with 5 % H₂SO₄ in ethanol and heating at 105 °C and under UV light; 3) by spraying with 10% H₂SO₄ in ethanol and heating at 110 °C. Identification by fingerprint technique. Quantitation of ginsenoside Rg by densitometry at 510 nm.
- Pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis 32c
- 93 135 J. TANG (Tan Jianning)*, G. LI (Li Guanqing), J. CHEN (Chen Jiaju) (*Guangxi Coll. TCM, Nanning, 530001, P. R. China): (Study of the quality control of Fushukang infusions.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (7), 543-546 (2004). TLC on silica gel with 1) ethyl acetate - methanol - formic acid - water 10:1:1:1; 2) petroleum ether - chloroform - ethyl acetate - conc. ammonia 80:15:5:1; 3) chloroform - ethyl acetate - methanol - formic acid 35:10:5:1; 4) chloroform - ethyl acetate - methanol - formic acid 70:8:20:3; 5) chloroform - methanol - water 13:6:2. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C; 2) under UV 254 nm; 3) by spraying with 5 % vanillin - H₂SO₄ solution and heating at 105 °C. Identification by fingerprint technique. Quantification of astragaloside by densitometry at 530 nm.
- Pharmaceutical research, traditional medicine, quality control, densitometry, quantitative analysis 32c
- 93 136 D. TANG (Tang Daoquan)*, Q. LU (Lu Qian), J. WANG (Wang Jie) (*Dep. Pharm., Xuzhou Med. Coll., Xuzhou, Jiangsu 221002, P. R. China): (Study of the preparation of β-cyclodextrin inclusion compound of volatile oil from Fructus Amomi Rotundus.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (10), 785-787 (2004). Study of the optimum inclusion process conditions for volatile oil by using orthogonal design and determining the utilization ratio of volatile oil from Fructus Amomi Rotundus by TLC on silica gel with benzene - ethyl acetate 19:1 and detection by spraying with vanillin - H₂SO₄ solution. Establishment of the optimum conditions as volatile oil: β-cyclodextrin 1:8 at 50 °C for two hours, giving the utilization ratio of volatile oil of 70.7%.
- Pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification, β-cyclodextrin inclusion compound 32c
- 93 137 Q. TANG (Tang Qiling)*, Y. LIU (Liu Yongjun), D. CUI (Cui Dongmei) (*Yantai Yuhuangding Hosp, Yantai, Shandong 264000, P. R. China): (Determination of chlorogenic acid in Ganggan

granules by thin-layer chromatography.) (Chinese). Chinese J. Hosp. Pharm. (Zhongguo Yiyuan Yaoxue Zazhi) **24** (2), 86-87 (2004). TLC on silica gel with toluene - 70% ethanol - methanol 35:35:10:6. Detection under UV light. Identification by standard comparison. Quantification by densitometry at 310 nm.

Clinical chemistry research, quality control, densitometry,
quantitative analysis, chlorogenic acid

32c

- 93 138 H. TIAN (Tian Hong)*, Y. ZHANG (Zhang Yujie), H. HUANG (Huang Haixin) (*Henan Nanyang Inst. Drug Cont., Nayang, Henan, 473061, P. R. China): (Study of the quality standard for Yangyin Jiangtang tablets.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (7), 547-549 (2004). TLC on silica gel with 1) chloroform - methanol - water 13:7:2 ; 2) chloroform - methanol - ethyl acetate - formic acid 20:8:4:1; 3) petroleum ether - ethyl acetate - formic acid 15:5:1; 4) chloroform - methanol - water 14:5:1. Detection 1) by spraying with 10% H₂SO₄ in ethanol and heating at 105 °C; 2) by spraying with 5 % vanillin-H₂SO₄ solution and heating at 105 °C; 3) under UV 365 nm. Identification by fingerprint technique. Quantitation of puerin by HPLC.

Pharmaceutical research, quality control, traditional medicine,
quantitative analysis, qualitative identification, puerin

32c, 4d

- 93 140 H. TU (Tu Honghai)*, R. ZHAO (Zhao Ruhai), Y. WEI (Wei Youliang), C. WANG (Wang Caiyun) (*The Hosp. 68210 PLA, Baoji, Sahnxi 721001, P. R. China): (Study of the quality standard for Shengxue capsules.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (9), 708-711 (2004). TLC on silica gel with 1) n-hexane - ethyl acetate 9:1, 2) ethyl acetate - chloroform - methanol - formic acid - water 10:5:5:1:1, 3) chloroform - methanol - water 90:5:1. Detection 1) under UV 365 nm and by spraying with 20 % perchloric acid solution and heating at 105 °C, 2) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C for 10 min, 3) by ammonia vapor and under UV 254 nm. Identification by fingerprint techniques. Quantitation of astragaloside by densitometry at 530 nm. Discussion of use of the procedures for the quality control of the medicine.

Pharmaceutical research, traditional medicine, quality control,
Densitometry, quantitative analysis

32c

- 93 143 Y. WANG (Wang Yuan)*, Y. WANG (Wang Yingfeng), G. REN (Ren Guifen) (*Dep. Chem., Capital Norm. Univ., Beijing 100037, P. R. China): (Study of in situ identification of thin-layer chromatography - Fourier Raman spectroscopy of yohimbine in Rauvolfia verticillata Bail.) (Chinese). Chinese J. Pharm. Anal. (Yaowu Fenxi Zazhi) **24** (1), 30-34 (2004). TLC on silica gel with chloroform - acetone - diethylamine 5:4:1. Detection under UV 254 nm. Identification by standard comparison. Use of silver gel as the surface enhanced subtract placed on the separated spots for measurement of FT-SERS. Investigation of the minimum detection amount for the compound, and the difference between the SERS and solid spectra.

Pharmaceutical research, quality control, qualitative identification, yohimbine,
TLC-Fourier Raman spectroscopy, FT-surface-enhanced Raman
spectroscopy (SERS)

32c, 4e

- 93 147 Y. XIA (Xia Yufeng), Q. YANG (Yang Qin), L. XIE (Xie Liqong), Q. WANG (Wang Qiang) (China Pharm. Univ., Nanjing 210038, P. R. China): (Study of the quality standard of Yupingfeng oral liquid). (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (12), 968-970 (2004). TLC on silica gel with 1) chloroform - methanol - water 13:7: 2, 2) toluene - ethyl formate - formic acid 5:4:1, 3) n-hexane - benzene - ethyl acetate 14:3:3. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C for 5 min, 2) under UV 365 nm, 3) by iodine vapor. Identification by fingerprint techniques. Quantitative determination of astragaloside by HPLC. TLC

is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, astragaloside 32c, 4d

- 93 148 H. XIE (Xie Hui), CH. MAO (Mao Chunqin), L. DI (Di Liuqing) (Nanjing Univ. TCM, Nanjing 210029, P. R. China): (Study of the quality standard of Huganbadu ointment.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **11**, 882-884 (2004). TLC on silica gel with 1) chloroform - methanol - conc. ammonia 80:4:1, 2) benzene - ethyl acetate 20 :1, 3) chloroform - methanol - water 13:6:2, 4) ethyl acetate - formic acid - water 10:1:1. Detection 1) by spraying with 5 % solution of potassium iodobismuthate, 2) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C, 3) by spraying with 10 % H₂SO₄ in ethanol, heating at 105 °C and under UV 365 nm, or under 254 nm. Identification by fingerprint techniques. Quantitative determination of matrine by densitometry at 520 nm. Validation of the quantitative method by investigation of its linearity range, reproducibility, repeatability, recovery. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

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

- 93 149 Y. XIE (Xie Yumei)* (*Anhui Prov. Inst. Drug Cont., Hefei Anhui 230061, P. R. China): (Determination of cholic acid in Niuhuang Shedan Chuanbei capsules.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (9), 771-773 (2004). TLC on silica gel with isooctane - dibutyl ether - glacial acetic acid 8:5:5. Detection by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C. Identification by standard comparison. Quantification of cholic acid by densitometry.

Pharmaceutical research, traditional medicine, quality control, densitometry,
quantitative analysis 32c, 11a

- 93 150 ZH. XIE (Xie Zhimin)*, M. WANG (Wang Minchun), Y. Wang (Wang Yunxia), X. WANG (Wang Xinli) (*Xi'an Inst. Drug Cont., Xi'an, 710054, P. R. China): (Quick identification of snake bile in Shedan Chuanbeiye syrup.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (7), 583-584 (2004). TLC on silica gel with benzene - ethyl acetate - methanol - glacial acetic acid - water 16:10:8:4:3. Detection under daylight or UV light. Identification by fingerprint technique. TLC screening of many other animal's bile, such as duck bile, chicken bile, cow bile, sheep bile, pig bile and fish bile, etc. Discussion of the possibility of replacing snake bile with some other animal's bile containing similar components.

Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, snake bile 32c, 4d

- 93 151 F. XONG (Xong Fuliang)*, ZH. LI (Li Zhixiong), ZH. CAO (Cao Zhihua), Y. WANG (Wang Yongping) (*Hubei Eng. Tech. Center TCM, Wuhan, Hubei 430052, P. R. China): (Study of the quality standard for Puqinxiaoyan tablets.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (2), 103-105 (2004). TLC on silica gel with 1) ethyl acetate - methanol - formic acid 4:8:1, 2) petroleum ether - chloroform - conc. ammonia 2:8:1. Detection 1) under UV 365 nm. 2) by spraying with 5 % potassium iodobismuthate solution. Identification by fingerprint techniques. Quantification of baicalin by HPLC.

Pharmaceutical research, quality control, clinical routine analysis,
qualitative identification, baicalin 32c

- 93 152 L. XU (Xu Liting), H. XIE (Xie Hua), ZH. JIA (Jia Zhenping), B. LIU (Liu Baiou) (General Hosp., Lanzhou Command, PLA, Lanzhou 730050, P. R. China): (Study of the quality standard

of Tianqi capsules.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **10**, 807-809 (2004). TLC on silica gel plates with 1) chloroform - methanol - water 13:7:2, 2) chloroform - acetone - formic acid 25:10:4. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C, 2) by ammonia vapor and under UV 365 nm. Identification by fingerprint techniques. Quantitative determination of salidroside by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine.

Pharmaceutical research, quality control, traditional medicine,
quantitative analysis, qualitative identification, salidroside 32c, 4d

- 93 153 R. XU (Xu Renliu), G. HAN (Han Guiru), ZH. LIU (Liu Zhe), Y. SONG (Song Yanling) (Hebei Provin. Inst. Drug Cont., Shijiazhuang, Hebei 050011, P. R. China): (Study of the quality standard of Erxiaqingxin tablets.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **25** (12), 970-974 (2004). TLC on silica gel with 1) ethyl acetate - methanol - water 6:1:3, 2) n-butanol - glacial acetic acid - water 4:1:5, 3) chloroform - methanol - water 28:10:1. Detection 1) by spraying with 1 % AlCl₃ and under UV 365 nm, 2) by spraying with 0.5 % ninhydrin in ethanol and heating at 105 °C, 3) under UV 365 nm. Identification by fingerprint techniques. Quantitative determination of puerarin by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, puerarin 32c, 4d

- 93 154 A. YANG (Yang Andong)*, Y. MA (Ma Yan), SH. WU (Wu Shihui) (*Sichuan Prov. Inst. TCM, Chengdu, Sichuan 610041, P. R. China): (Study of the quality standard of Zangfukang suppository.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 26-29 (2004). TLC on silica gel with 1) ethyl acetate - methanol - conc. ammonia 17:2:1, 2) cyclohexane - ethyl acetate - formic acid 30:15:1, or 50:10:1. Detection 1) by spraying with 5 % potassium iodobismuthate solution, 2) by spraying with 5 % vanillin in H₂SO₄ solution and heating at 105 °C, 3) under UV 254 nm. Identification by fingerprint techniques. Quantitative determination of puerarin by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, puerarin 32c, 4d

- 93 155 D. YANG (Yang Dongai)*, B. DAI (Dai Bin), C. QIU (Qiu Cuichang) (*Guangxi Inst. Minor. Pharm. & Med., 530001, P. R. China): (Study of the identification of Huanhua Shudai Paocha.) (Chinese) *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 74-76 (2004). TLC on silica gel with 1) petroleum ether - ethyl acetate - formic acid 30:10:1, 2) petroleum ether - ethyl acetate 25:1, 3) chloroform - methanol - water 13:7:2. Detection 1) under 365 nm, 2) by spraying with 1 % vanillin - H₂SO₄ solution, 3) by spraying with 10 % H₂SO₄ in ethanol. Identification by fingerprint techniques, combined with microscopy of the microcosmic configuration and shape of the medicine, and with visualization by color reaction of anthracene and quinine with 1 % NaOH solution. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research, quality control, traditional medicine,
qualitative identification, anthracene, quinine 32c

- 93 156 M. YANG (Yang Ming)*, ZH. LI (Li Zhi), D. WU (Wu Dazhang), Y. YANG (Yang Rongping), F. WANG (Wang Fei) (*Chengdu Univ. TCM, Chengdu, Sichuan 610075, P. R. China): (Study of the quality standard of Yanqingsong pills.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 23-26 (2004). TLC on silica gel with 1) chloroform - methanol - water 28:10:1, 2) n-hexane - ethyl acetate 9:1, 3) chloroform - methanol 5:1, 4) petroleum ether - diethyl ether 1:

1. Detection 1) under UV 365 nm, 2) by spraying with 5 % vanillin in H₂SO₄ solution. Identification by fingerprint techniques. Quantitative determination of puerarin by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.
- Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, puerarin 32c, 4d
- 93 157 X. YANG (Yang Xixiao)*, B. ZHU (Zhu Binghui), J. FANG (Fang Jihui), D GUO (Guo Dan), CH. WANG (Wang Chunxia), X. CAO (Cao Xixing), L. HOU (Hou Lianbing) (*Pharm. Sec., Nanfang Hosp., The 1st Milit. Med Univ., Guangzhou, Guangdong 510515, P. R. China): (Study of the quality standard of Changtong oral liquid.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (9), 719-720 (2004). TLC on silica gel with 1) chloroform - methanol - water 13:7:2, 2) chloroform - acetone - formic acid 10:5:2, 3) petroleum ether - ethyl formate - formic acid 15:5:1. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating and under UV 365 nm, 2) by ammonia vapor and under UV 365 nm. Identification by fingerprint techniques. Quantitation of danshensu by HPLC. Discussion of use of the procedures for the quality control of the medicine.
- Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, danshensu 32c
- 93 158 X. YANG (Yang Xixong), CH. YANG (Yang Chengxong), L. FU (Fu Lianqun), J. LU (Lu Jinqing) (Jingmen Hosp. No.2, Jingmen, Hubei 448000, P. R. China): (Study of the quality standard of Chongcaoyangshen capsules.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **10**, 858-860 (2004). TLC on silica gel with 1) chloroform - methanol - water 13:7:2, 2) isopropanol - ethyl acetate - water 17:9:9, n-butanol - formic acid - water 15:3:2. Detection 1) by spraying with 10 % H₂SO₄ in ethanol and heating at 110 °C, 2) by spraying with 0.3 % ninhydrin in n-butanol, 3) by spraying with 10 % vanillin solution. Identification by fingerprint techniques. Quantitative determination of ginsenoside by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.
- Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification, ginsenoside rb 32c, 4d
- 93 058 Y. YANG (Yang Yun) et al., see section 17a
- 93 161 X. ZHAI (Zhai Xufeng)*, F. LIU (Liu Fajin), Y. GUO (Guo Yongzhuang), C. LIAO (Liao Caizhen) (*Guangdong Yifang Pharm. Co., Ltd., Nanhai, Guangdong 528244, P. R. China): (Determination of astragaloside IV in extract of Radix Astragali by thin-layer chromatography.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (8), 651-653 (2004). TLC on silica gel with n-butanol - ethyl acetate - ammonia 10% 4:1:5. Detection by spraying with 10 % H₂SO₄ in ethanol and heating at 105 °C for 5-7 min. Identification by standard comparison. Quantification by densitometry at 530 nm. Validation of the procedure by investigation of its precision, specificity, repeatability, reproducibility, recovery, etc. Determination of the title compound in the extracts from five batches of the drug.
- Pharmaceutical research, traditional medicine, quality control,
quantitative analysis, qualitative identification 32c
- 93 163 CH. ZHAO (Zhao Chunxiang)*, G. ZHANG (Zhang Guijie), L. ZHANG (Zhang Li), L. XU (Xu Liangyan) (*Jilin Prov. Inst. Drug Cont., Cahngchun, Jilin 130062, P. R. China): (Study of the identification of Shenronglutai pills by thin-layer chromatography.) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) **25** (8), 680-682 (2004). TLC on silica gel with 1) ethyl acetate - formic acid - water - methanol 35:10:15:2; 2) benzene - ethyl acetate 19:1; 3) chloroform -

ethyl acetate - methanol - water; 4) petroleum ether; 5) petroleum ether - ethyl acetate 17:3; 6) chloroform - methanol - water 13:7:2; 7) ethyl acetate - formic acid - water 10:2:3. Detection 1) under daylight, 2) by spraying with 5 % H₂SO₄ in ethanol and heating at 105 °C, 3) by spraying with 5 % p-dimethylaminobenzaldehyde in 10 % H₂SO₄ in ethanol and heating at 105 °C, 4) by spraying with 2 % 2,4-dinitrophenylhydrazine in ethanol, 5) by spraying with 1 % vanillin - H₂SO₄ solution, 6) by spraying with 5 % FeCl₃ in ethanol, 7) by spraying with 2 % AlCl₃ in ethanol and under UV 365 nm, 8) under UV light. Identification by fingerprint techniques.

Pharmaceutical research, traditional medicine, quality control,
qualitative identification, ginsengs

32c

- 93 164 Y. ZHENG (Zheng Yi)* (*Pingshan Pharm. Factory, Fuzhou, Fujian 350003, P. R. China): (Study of the quality standard of Ganmaoyankeling tablets.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **11**, 885-887 (2004). TLC on silica gel with 1) n-butanol - 3 M NH₄OH - ethanol 5:2:1, 2) toluene - ethyl acetate - formic acid 5:4:1. Detection 1) under UV 254 nm, 2) by spraying with 10 % FeCl₃ in ethanol. Identification by fingerprint techniques. Quantitative determination of quercetin by HPLC. TLC is widely applied in the quality control for Chinese traditional medicine, both medicinal herbal drugs and their preparations.

Pharmaceutical research traditional medicine quality control,
quantitative analysis, qualitative identification, quercetin

32c, 4d

- 93 083 P. P. BERNY et al., see section 29

- 93 145 M. WERTHER*, B. MUELLER, A. STREY (*Institut für Tierarzneimittel GmbH, Berliner Allee 317-321, D-13088 Berlin, Germany, margit.werther@camag-berlin.de): HPTLC - Method for quantitative determination of levamisol in tissue and organs of pigs. *CBS* **83**, 12-13 (1999). HPTLC of levamisol in pig tissue on silica gel with chloroform over a developing distance of 70 mm, then with chloroform - methanol - formic acid 400:50:1 over 50 mm. Quantitative determination by absorbance measurement at 220 nm. Limits of detection are 0.6 - 1.5 µg/kg.

Food analysis, quality control, quantitative analysis, densitometry,
levamisol, residue analysis, antihelmintic

32d

- 93 088 A. BLATTER* (*Department of Pharmaceutical Biology, University of Basel, CH-4056 Basel, Switzerland): HPTLC investigations on St. John's Wort. *CBS* **88**, 9-11 (2002). HPTLC of flavonoids, hypericin and pseudohypericin on silica gel and diol phases (for quantification of hypericin) respectively, with ethyl acetate - dichloromethane - formic acid - acetic acid - water 100:25:10:10:11. Detection by dipping the warm plate in modified natural products reagent (0.5% in ethyl acetate). Selective HPTLC of hyperforin on silica gel with toluene - dichloromethane 4:1. Visual detection by spraying with Godin reagent. Quantitative determination by absorbance measurement at 310 nm without derivatization.

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

32e

- 93 089 A. BLATTER*, E. REICH (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland): Quality control of *Stephania tetrandra*. *CBS* **92**, 13-15 (2004). HPTLC on silica gel with toluene - ethyl acetate - methanol - ammonia 25 % 10:10:50:3 over 70 mm with chamber saturation for 30 min. Detection by dipping in iodine solution (0.05 g in 10 mL ethanol 96 %). Visual evaluation under white light. Quantitative determination by absorbance measurement at 210 nm. Repeatability measuring 6 replicates of the same sample on one plate is between 1.6 % and 3.7 %, repeatability of the means from 3 different plates is 1.2 %.

Quality control, herbal, traditional medicine, quantitative analysis

32e

- 93 090 C. BODINET*, R. MENDEL, U. WEGNER, U. LINDEQUIST, E. TEUSCHER, J. FREUDENSTEIN (*Schaper & Brümmer GmbH & Co. KG, Bahnhofstr. 35, D-38259 Salzgitter, Germany): Effect of oral application of an immunomodulating plant extract on Influenza virus Type A infection in mice. *Planta med.* **68**, 896-900 (2002). TLC of cichoric acid, echinacoside, maackianin, and umbelliferone on silica gel with ethyl acetate - AcOH - acetic acid - water 100:11:11:27 and formic acid - ethyl formate - toluene 3:10:10. Detection after spraying with 1 % 2-aminophenyl diphenyl borate in methanol and 5 % PEG 400 in methanol under UV light at 366 nm.
Pharmaceutical research, herbal, qualitative identification 32e
- 93 092 J. CHANG, L.-J. XUAN, Y.-M. XU, J.-S. ZHANG* (*Shanghai Institute of Materia Medica, Shanghai Institute of Biological Sciences, Chinese Academy of Sciences, 294 Taiyuan Road, Shanghai 200031, P. R. China): Cytotoxic terpenoids and immunosuppressive phenolic glycosides from the root bark of *Dictamnus dasycarpus*. *Planta med.* **68**, 425-429 (2002). TLC of glycosides (dasycarpaside A, B, 2-methoxy-4-hydroxymethylphenol 1-O- α -rhamnopyranosyl-(1''-6')- β -glucopyranoside, 2-methoxy-4-acetylphenol 1-O- α -rhamnopyranosyl-(1''-6')- β -glucopyranoside, 2-methoxy-4-(8-hydroxyethyl)-phenol 1-O- α -rhamnopyranosyl-(1''-6')- β -glucopyranoside) on silica gel with benzene - formic acid - ethyl acetate 3:2:5. Detection by spraying with sulfuric acid - ethanol 1:4 reagent followed by heating. In addition acid hydrolysis on HPTLC silica gel plates with chloroform - methanol - water 10:4:3 and visualization by spraying with phenylamine-o-benzene dicarboxylic reagent followed by heating.
Pharmaceutical research, qualitative identification, *Dictamnus dasycarpus*, phenolic glycosides 32e
- 93 094 J.-J. CHEN, Y.-C. HUANG, Y.-C. CHEN, Y.-T. HUANG, S.-W. WANG, C.-Y. PENG, C.-M. TENG, I.-S. CHEN* (*School of Pharmacy, Kaohsiung Medical University, Kaohsiung, Taiwan, Republic of China): Cytotoxic amides from *Piper sintonense*. *Planta med.* **68**, 980-985 (2002). Preparative TLC of pipersintenamide, piperarboricoline, sintonpyridone, β -sitosterol, β -sitostenone, and stigmasta-4,22-diene-3-one on silica gel with n-hexane - ethyl acetate 10:1, dichloromethane, dichloromethane - ethyl acetate 10:1, 20:1 and 30:1, chloroform, chloroform - methanol 30:1, and chloroform - acetone 15: 1 and 20:1.
Pharmaceutical research, herbal, preparative TLC, *Piper sintonense* 32e, 13c, 17c
- 93 096 J. CRECHE* (*Plant Molecular Biology and Biochemistry Department, EA 2106, Plant Biocompounds and Biotechnology, Faculty of Pharmacy, University of Tours, 31 avenue Monge, F-37200 Tours, France. creche@univ-tours.fr): Rapid analysis of indole alkaloids in tissue cultures. *CBS* **90**, 10-11 (2003). HPTLC of periwinkle cell samples on prewashed silica gel with ethyl acetate - diethylamide 9:1 in horizontal developing chamber over 25 mm. Detection by radiation with shortwave UV 254 nm. Quantitative determination by fluorescence measurement at 254 nm or 365 nm.
Pharmaceutical research, herbal, densitometry, quantitative analysis, ajmalicine, serpentine, vinca-alkaloids, cell culture 32e
- 93 103 N. F. DE MOURA, E. SIMIONATTO, C. PORTO, S. C. S. HOELZEL, E. C. S. DESSOY, N. ZANATTA, A. F. MORE L* (*Departamento de Química, Universidade Federal de Santa Maria, Campus de Camobi, Santa Maria-RS, Brazil): Quinone alkaloids, coumarins and volatile constituents of *Helietta longifoliata*. *Planta med.* **68**, 631-634 (2002). Preparative TLC of helietidine, γ -fagarine, flindersine, kokusagenine, and maculosine on silica gel with chloroform - methanol 49:1. Detection by bioautography.
Pharmaceutical research, herbal, preparative TLC, *Helietta longifoliata* 32e, 22

- 93 098 M. DEL ROSARIO HERNÁNDEZ-MEDEL, R. PEREDA-MIRANDA* (*Departamento de Farmacia, Facultad de Química, Universidad Nacional Autónoma de México, 04510, Mexico City, Mexico): Cytotoxic anthraquinone derivatives from *Picramnia antidesma*. *Planta med.* **68**, 556-558 (2002). TLC of chrysophanol, emodin, uveoside, and picramnioside E on silica gel with chloroform - methanol 9:1.
Pharmaceutical research, qualitative identification, *Picramnia antidesma*, anthraquinone derivatives 32e
- 93 100 U. DOLD* (*Institute of Pharmacy, University of Basel, Switzerland): Screening of *Vitex agnus castus* extracts by HPTLC. *CBS* **86**, 13-15 (2001). HPTLC on silica gel (prewashed with methanol) with THF - toluene - formic acid - water 16:8: 2:1 for flavonoids and toluene - ethyl acetate 9:2 for diterpenes. Detection of flavonoids by dipping in natural products reagent followed by dipping in PEG 6000 solution. Detection of diterpenes by dipping in anisaldehyde reagent. Evaluation with video technology.
Pharmaceutical research, quality control, herbal, densitometry 32e
- 93 105 M. GUO (Guo Mei)*, Y. LI (Li Yingdong), J. SHAO (Shao Jing), X. SHI (Shi Xia), X. YU (Yu Xiaohui) (* Gansu Inst. TCM, Lanzhou, Gansu 730000, P. R. China): (Effect of different drying method on the quality of *Radix Angelicae Sinensis*.) (Chinese). *J. Chinese Trad. Patent Med. (Zhongchengyao)* **26** (1), 36-38 (2004). TLC of the extracts of the drug dried in different conditions (by fuming, sun-baking and airing) on silica gel with benzene - ethyl acetate - methanol 40:10:1. Detection by spraying with 2 % ferricyanide- 0.01 % FeCl₃ in 2 N HCl solution. Identification by fingerprint technique. Quantification of ferulic acid by HPLC. The results showed that fuming was the best among the three drying methods.
Pharmaceutical research, herbal, quantitative analysis, densitometry, ferulic acid 32e
- 93 106 E. HAHN-DEINSTROP*, A. KOCH (*Kleingeschaidter Str. 23, D-90542 Eckental, Germany. elke.hahn_deinstrop@arco r.de): Identification of true Saffron. *CBS* **91**, 9-11 (2003). HPTLC on silica gel with ethyl acetate - 2-propanol - water 13:5:2 over 65 mm with chamber saturation. Detection by spraying with anisaldehyde - sulfuric acid reagent, followed by heating at 110 °C for 5-10 min. Visual evaluation in white light and at 254 nm.
Food analysis, herbal, quality control, qualitative identification, saffron 32e
- 93 111 G. JÜRGENLIEMK, A. NAHRSTEDT*, (*Institute for Pharmacy, Biology and Phytochemistry, Westfälische Wilhelms-University of Münster, Hittorfstr. 56, 48149 Münster, Germany): Phenolic compounds from *Hypericum perforatum*. *Planta med.* **68**, 88-91 (2002). TLC of neochlorogenic acid, protocatechuic acid, 3-O-[Z]-p-coumaroylquinic acid, 3-O-[E]-p-coumaroylquinic acid, cryptochlorogenic acid, mangiferin, isoorientin, cyanidin-3-O-a-L-rhamnoside, rutin, hyperoside, isoquercitrin, miquelianin, astilbin, guaijaverin, quercitrin, quercetin, biapigenin, amantoflavone, pinocembrin-7-methyl ether, pseudohypericin, hyperforin, hypericin on silica gel with ethyl acetate - formic acid - water 30:2:3. Detection by derivatization with Naturstoff reagent.
Pharmaceutical research, quality control, qualitative identification, *hypericum perforatum*, phenolic compounds 32e
- 93 112 A. KOCH*, R. RICHTER, (*Frohme Apotheke, Frohmestrasse 14, D-22457 Hamburg, Germany, koch@frohme-apotheke.de): Analysis of constituents of devil's claw and determination of their bioactivity by HPTLC. *CBS* **89**, 4-7 (2002). HPTLC of devil's claw extract on lichrospher silica gel with ethyl acetate - ethanol (70 %) 7:3 over 70 mm with chamber saturation. Detection by dipping in anisaldehyde - sulfuric acid reagent followed by heating at 105 °C until colors develop. Determination of bioactivity with DPPH-biotest by spraying with 0.05 % solution of

- (2,2-di-(4-tert-octylphenol)-1-picrylhydrazyl in methanol. Determination of antibiotic properties with Merck Chrom Biodip test by dipping in bacteria solution and incubation at 26 °C for 20 h, followed by spraying with yellow MTT tetrazolium salt.
- Pharmaceutical research, quality control, traditional medicine, herbal 32e
- 93 113 S. LAVOINE*, J.-F. ARNAUDO, D. COUTIERE, (*Studio de Creation de Parfumerie, ZAC Font de l'Orme, BP411, F-06254 Mougins Cedex, France): HPTLC analysis of the major components in Vanilla bean extracts. CBS **81**, 14-15 (1998). HPTLC-AMD of extracts on silica gel with 16-step gradient based on methanol containing 0.1 % acetic acid via dichloromethane to n-hexane. Quantification by absorbance measurement at 255, 260, 290 and 310 nm (multi wavelength scan). Precision is determined to be 3%.
- Food analysis, quantitative analysis, densitometry, AMD, vanilla, vanillic acid, p-hydroxybenzoic acid, p-hydroxybenzaldehyde 32e
- 93 117 S.-H. LI, H.-J. ZHANG*, X.-M. NIU, P. YAO, H.-D. SUN, H. H. S. FONG* (*Program for Collaborative Research in the Pharmaceutical Sciences, M/C 877, College of Pharmacy, University of Illinois at Chicago, 833 S. Wood St., Chicago, IL 60612, USA): Taxuyunnanines S-V, new taxoids from *Taxus yunnanensis*. *Planta med.* **68**, 253-257 (2002). Preparative TLC of taxuyunnanine S on silica gel with i-propanol - chloroform 2:1. Visualization by spraying with 10 % sulfuric acid in ethanol.
- Pharmaceutical research, preparative TLC, *Taxus yunnanensis* 32e
- 93 123 E. MALDONADO JIMÉNEZ*, F. R. TORRES, M. MARTINEZ, A. L. PÉREZ-CASTORENA (*Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510 D. F., México): 18-Acetoxywhitanolides from *Physalis chenopodifolia*. *Planta med.* **70**, 59-64 (2004). Analytical and preparative TLC of withasteroids (physachenolide E and D) on silica gel with n-hexane - acetone 6:5 by 8-fold development and chloroform - acetone 7:3 by 4-fold development. Detection under UV light and by spraying with cerium sulfate - sulfuric acid reagent.
- Pharmaceutical research, herbal, quality control, qualitative identification, preparative TLC, *Physalis chenopodifolia* 32e
- 93 125 T. MORIKAWA, H. MATSUDA, I. YAMAGUCHI, Y. PONGPIRIYADACHA, M. YOSHIKAWA*, (*Kyoto Pharmaceutical University, Misasagi, Yamashina-ku, Kyoto 607-8412, Japan): New amides and gastroprotective constituents from the fruit of *Piper chaba*. *Planta med.* **70**, 152-159 (2004). Analytical HPTLC on silica gel RP-18 and preparative TLC on silica gel of piperine, piperamine, piperlonguminine, and methyl piperate with n-hexane - ethyl acetate 1:1. Detection by spraying with 1 % cerium sulfate - 10 % aqueous sulfuric acid followed by heating on a plate heater.
- Pharmaceutical research, herbal, preparative TLC, qualitative identification, *Piper chaba* 32e
- 93 128 E. REICH* (Ed.) (*CAMAG, Sonnenmattstr. 11, CH-4132 Muttenz, Switzerland): Analysis of *Arctostaphylos Uva ursi* leaves. CBS **85**, 14-15 (2000). HPTLC on silica gel with ethyl acetate - formic acid - water 45:5:3. Detection by spraying with dichloroquinonechlorimide followed by spraying with 10 % aqueous sodium carbonate solution. Visual evaluation.
- Pharmaceutical research, quality control, herbal, qualitative identification, arbutin, hydroquinone 32e

- 93 130 J. RICHTER, K. KABRODT, I. SCHELLENBERG* (*Fachgruppe Bioanalytik, Hochschule Anhalt (FH), Strenzfelder Allee 28, D-06406 Bernburg, Germany, schellenberg@loel.hs-anhalt.de): Characterization of tannins from rhubarb by TLC/HPTLC. CBS **88**, 4-6 (2002). HPTLC of rhubarb extract on silica gel (prewashed with isopropanol) with acetone - water - formic acid 18:1:1 or toluene - acetone - formic acid 3:6:1 over 75 mm after partial chamber saturation. Detection by 1) dipping in a 1 % solution of vanillin in ethanol and heating at 50 °C for 5 min followed by exposing to HCl atmosphere, 2) spraying with iron (III) chloride 3 % in methanol, 3) spraying with 4-dimethylaminocinnamaldehyde 1 % in ethanol - HCl (37 %) 1:1, 4) spraying with natural products reagent 1 % in methanol. Documentation under white light and at 366 nm.
Quality control, herbal, qualitative identification, rhubarb, tannin 32e
- 93 131 S. ROENGSUMRAN*, K. MUSIKUL, A. PETSOM, T. VILAIVAN (*Department of Chemistry, Faculty of Science, Chulalongkorn University, Bangkok 10330, Thailand): Croblongifolin, a new anticancer clerodane from *Croton oblongifolius*. *Planta med.* **68**, 274-277 (2002). TLC of croblongifolin, crovatin, and nidorellol on silica gel with n-hexane - ethyl acetate 9:1 and chloroform - methanol 19:1.
Pharmaceutical research, qualitative identification, *Croton oblongifolius*, croblongifolin, crovatin, nidorellol 32e
- 93 132 A. SCHMID* (*Institute of Pharmacy, University of Basel, Switzerland): HPTLC and videotechnology for stability tests of plant extracts. CBS **86**, 10-12 (2001). HPTLC of valerian root extracts on silica gel (prewashed with methanol) with chloroform - ethyl acetate - propanol 15:10:1. Detection by dipping for 1 s in 15 % sulfuric acid in methanol followed by heating at 120 °C for 10 min and cooling to room temperature. Evaluation with video technology (VideoStore/VideoScan).
Pharmaceutical research, quality control, herbal, densitometry 32e
- 93 134 A. SIEVERS, L. OSHINOWO, W. SCHULTZE*, A. KOCH, R. RICHTER (*Institut für Pharmazie, Abteilung Pharmazeutische Biologie und Mikrobiologie, Bundesstrasse 45, D-20146 Hamburg, Germany): Simple thin layer chromatographic test for antioxidative compounds using the DPPH assay. CBS **88**, 14-15 (2002). HPTLC of mushroom extracts on silica gel with dichloromethane - ethyl acetate - methanol 3:1:1 and HPTLC of hydroquinone, rutin, resorcinol, and ascorbic acid on silica gel with toluene - methanol - acetic acid 45:8:4 over 80 mm with chamber saturation. Determination of bioactivity with DPPH-biotest by spraying with 5 mg (2,2-di-(4-tert-octylphenol)-1-picrylhydrazyl in 10 mL acetone.
Pharmaceutical research, herbal, qualitative identification, densitometry, bioautography, antioxidative compounds, DPPH assay 32e, 4e
- 93 139 I.-L. TSAI*, C.-H. HUNG, C.-Y. DUH, I.-S. SHEN (*School of Pharmacy, College of Pharmacy, Kaohsiung Medical University, Kaohsiung, Taiwan, Republic of China): Cytotoxic butanolides and secobutanolides from the stem wood of *Formosan Lindera communis*. *Planta med.* **68**, 142-145 (2002). Preparative TLC of lincomalide C, D, secolincomalide A, B, isoobtusilactone A, secoisolancifolide on silica gel with n-hexane - ethyl acetate 20:1, 6:1, 4:1, 3:1 and n-hexane - acetone 5:1. Visualization under UV light.
Pharmaceutical research, qualitative identification, preparative TLC, cytotoxic butanolides, secobutanolides, *Formosan Lindera communis* 32e
- 93 142 F. WAHLI* (*Department of Pharmaceutical Biology, University of Basel, CH-4056 Basel, Switzerland): Stability tests of *Vitex agnus castus* (chaste tree) extracts. CBS **91**, 12-13 (2003). HPTLC of flavonoids with tetrahydrofuran - toluene - formic acid - water 16:8:2:1, diterpenes

with toluene - ethyl acetate 9:2, and iridoids with ethyl acetate - methanol - water 77:15:8 (with chamber saturation), in horizontal developing chamber over 52 mm. Detection of flavonoids by dipping warm plate in natural products reagent (0.5 % in ethyl acetate) followed by dipping in PEG 400 solution (5 % in dichloromethane), of diterpenes by dipping in 10 % methanolic sulfuric acid followed by heating at 105 °C, and iridoids by dipping in 4-dimethylaminobenzaldehyde reagent (1 % in 1 N methanolic HCl). Visual evaluation at 366 nm (flavonoids), white light (diterpenes), and white light with sharp cut filter 560 nm (iridoids). Stability of extracts was investigated under drastic stress conditions (acid, base, light, heat, humidity).

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

- 93 146 V. WIDMER* (*Department of Pharmaceutical Biology, University of Basel, CH-4056 Basel, Switzerland): Analysis of flavone glycosides from hawthorn and passion flower. *CBS* **91**, 14-15 (2003). HPTLC of hawthorn extract on silica gel with ethyl acetate - methanol - water - formic acid 50:2:3:6, of passion flower extract on silica gel with tetrahydrofuran - toluene - formic acid - water 16:8:2:1, and of chrysin on RP-18 W phase with tetrahydrofuran - methanol - water - formic acid 4:6:14:1. Detection by dipping the warm plate in natural products reagent (0.5 % in ethyl acetate) followed by dipping in PEG 400 solution (5 % in dichloromethane). Visual evaluation at 366 nm.

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

- 93 159 C. YANG, Y.-P. SHI, Z.-J. JIA* (*Department of Chemistry, Lanzhou University, Lanzhou, Gansu 730000, P. R. China): Sesquiterpene lactone glycosides, endesmanolides, and other constituents from *Carpesium macrocephalum*. *Planta med.* **68**, 626-630 (2002). Preparative TLC of 2-a-O-β-D-glucopyranosyl-5aH-endesma-4(15),11(13)-dien-12,8β-olide on silica gel with dichloromethane - methanol 6:1 by 2-fold development.

Pharmaceutical research, herbal, preparative TLC, sesquiterpene lactone glycosides, endesmanolides, *Carpesium macrocephalum* 32e, 15a

- 93 160 J. YIN, K. KOUDA, Y. TEZUKA, Q. L. TRAN, T. MIYAHARA, Y. CHE, S. KADOTA* (*Department of Natural Products Chemistry, Institute of Natural Medicine, Toyama Medical and Pharmaceutical University, 2630 Sugitani, Toyama 9 30-0194, Japan): New diarylheptanoides from the rhizomes of *Dioscorea spongiosa* and their antiosteoporotic activity. *Planta med.* **70**, 54-58 (2004). Analytical and preparative TLC of diospongin A, B, and syringaresinol on silica gel and the monoacetone and diacetone of diospongin C, piperitol, and sesaminone on RP-18 with methanol - water 5:2. Analytical TLC of bis(4-N,N-dimethylaminobenzoate) on silica gel using chloroform - methanol 20:1. Detection under UV light at 254 nm.

Pharmaceutical research, traditional medicine, preparative TLC, qualitative identification, *Dioscorea spongiosa* 32e

- 93 162 Y. ZHANG, T. AKAO*, N. NAKAMURA, C.-L. DUAN, M. HATTORI, X.-W. YANG, J.-X. LIU (*Faculty of Pharmaceutical Sciences, Toyama Medical and Pharmaceutical University, 2630 Sugitani, Toyama 930-0194, Japan): Extremely low bioavailability of magnesium lithospermate B, an active component from *Salvia miltiorrhiza*, in rat. *Planta med.* **70**, 138-142 (2004). Analytical and preparative TLC of lithospermate B salt mixture on silica gel with chloroform - ethyl acetate - benzene - formic acid - methanol 15:20:10:10:1.

Pharmaceutical research, herbal, qualitative identification, preparative TLC, *Salvia miltiorrhiza* 32e

- 93 121 O. M. HYUNE* (Oh Mi Hyune) (*Department of Herbal Medicine Evaluation, Korean Food and Drug Administration, Korea, ekkims@hanmail.net): Quality control of root extracts from

Platycodon grandiflorum. CBS **87**, 6-7 (2001). HPTLC on silica gel with chloroform - methanol - water 6:4:1. Detection by spraying with 10 % ethanolic sulfuric acid solution, followed by heating at 120 °C. Quantitative determination by absorbance measurement at 450 nm.

Quality control, traditional medicine, herbal, densitometry 32g

35. Other technical products and complex mixtures

93 165 F.M. TRIOLO, J. STANTON, M.J. CALANDRA* (*Firmenich, Inc., P.O. Box 5880, Princeton, NJ 08543, USA): Determination of antioxidant potency with planar chromatography. CBS **87**, 13-15 (2001). HPTLC of antioxidants on silica gel with cyclohexane - ethyl acetate 49:1 containing 2 mg/mL butylated hydroxytoluene over a distance of 25 mm after preconditioning with solvent vapor for 5 min. Quantitative determination by absorbance measurement at 442 nm.

Food analysis, antioxidants 35b, 15a

93 141 L. VICARD et al., see section 32a

93 009 D. JÄNCHEN* (Ed.), see section 5d

37. Environmental analysis

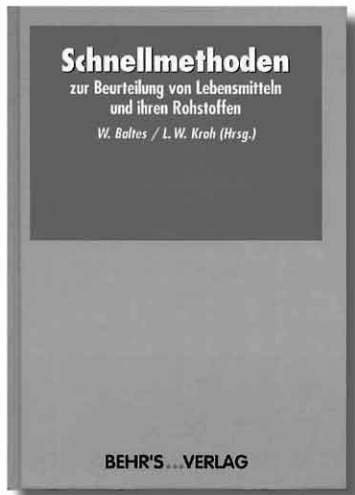
93 006 W. KREISS et al., see section 3e

93 010 D. JÄNCHEN* (Ed.), see section 5b

93 079 L. VICARD et al., see section 28a

93 080 I. VOVK et al., see section 28a

93 081 CH. WEINS et al., see section 28a



W. Baltes/L.W. Kroh (Hrsg.)

Schnellmethoden zur Beurteilung von Lebensmitteln und ihren Rohstoffen

Behr's Verlag, Hamburg (2004)
ISBN 3-89947-120-2 – 429 Seiten

Im Vorwort zur 3. Auflage sagt der Herausgeber (Kroh) »... So ist es ein schwieriges Unterfangen, die(se) schnelle Entwicklung der Analytik in Form eines Buches zu begleiten, ohne sich den Vorwurf machen zu lassen, dass schon mit dem Erscheinen des Buches Teilgebiete »veraltet« sind. ...«

Soweit es das 26 Seiten umfassende Kapitel Dünnschicht-Chromatographie (K. Zieloff, M. Werther) betrifft – nur mit diesem hat sich der Unterzeichner befasst – ist dieser Gefahr hervorragend begegnet worden. Nach einer kurzen Einführung werden die Besonderheiten der DC/HPTLC, die geeignet sind, sie zur Methode der Wahl zu machen, erläuternd dargestellt. Sodann werden die Arbeitsschritte Auswahl des Schichtmaterials – Probenauftragung – Fließmittelauswahl und Chromatogrammentwicklung – postchromatographische Derivatisierung – quantitative Auswertung – Dokumentation, alle strikt aus der Sicht des heutigen Standes der Technik beschrieben und die möglichen Varianten diskutiert. Im Teil Anwendungsbeispiele aus dem Gebiet der Lebensmittelchemie werden behandelt: Unerwünschte Stoffe wie Mykotoxine, Pflanzenschutz- und sonstige Mittel, Stoffe mit pharmakologischer Wirkung und Zusatzstoffe. In der Zusammenfassung werden hervorgehoben: Einsatz der DC/HPTLC als Screening-Methode, ihre Bedeutung als quantitatives Analysenverfahren und Entwicklungstrends.

Das Kapitel gibt nicht nur dem Analytiker, der der instrumentellen Dünnschicht-Chromatographie noch reserviert gegenüber steht, einen professionellen Einblick in die Möglichkeiten der Methode, es bietet auch dem erfahrenen Praktiker eine Übersicht über den aktuellen Stand der Technik.

Wenn die übrigen Kapitel dem gleichen Standard entsprechen, was aufgrund der Auswahl von Themen und Autoren sowie der äusserlich erkennbaren Darstellung zu erwarten ist, kann man dem Herausgeber und den Autoren zu diesem Buch nur gratulieren.

Eine englisch-sprachige Ausgabe ist vorgesehen, doch kann über das voraussichtliche Erscheinungsdatum noch nichts gesagt werden.

Dieter Jänchen

Exchange in TLC 2004



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◀ Acknowledgement of Prof. Dr. Ebel's achievements

This year the international symposium "Planar Chromatography 2004" in honor of Prof. Dr. Ebel was held at Visegrad, Hungary, 23–25 May. The host, Prof. Dr. Nyiredy, acknowledged a record: 60 contributions (lectures and posters) were received and published in the 600-page proceedings of the symposium and about 80 researchers participated in the conference.

The first 2004 meeting of the French TLC Club (CCCM) – we reported in detail about this club in CBS 90 – took place on 3 June at Aventis of Neuville sur Saone. Interesting papers and research results as well as an active exchange of ideas enriched the event. The second meeting this year is scheduled for 21 October. It will be hosted by Parfums Christian Dior, LVMH research group, of Saint Jean de Braye near Orleans.



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▲ Snapshot during coffee break (left to right): Prof. Dr. Sliwiok, founder of the symposium series 1977, Prof. Dr. Voelkel, Dr. Geiss, unknown participant, Prof. Dr. Soczewinski

About 250 participants gathered for the XXVIIIth Symposium on "Chromatographic Methods for Investigating of Organic Compounds" on 7–9 June at Katowice-Szczyrk, Poland. 124 scientific posters were shown and 37 speakers from 8 different countries presented their work. Among them were Prof. Dr. Tanaka and Prof. Dr. Jinno from Japan. The next symposium, the XXIXth, will take place 8–10 June 2005 at the same location.



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▲ Organization committee of Prof. Dr. Glowniak (seated) and Ms. Zych, Donau Lab (3. from left, upper row) in Kazimierz Dolny

The "4. International Symposium on Natural Products" met 14–17 June at Kazimierz Dolny, Poland. 39 papers and 167 posters represented the state of science in phytochemistry. The next symposium is planned for 2006 at Lublin.



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▲ Speakers of the "International Chromatographers Meeting" at Nuremberg-Lauf (left to right): Dr. Morlock, Dr. Mümmeler, Prof. Dr. Kaiser, Prof. Dr. Ebel, Prof. Dr. Nyiredy, Dr. Renger, Mrs. Richter, Dr. Koch, Mrs. Hahn-Deinstrop, Dr. Schäfer

Elke Hahn-Deinstrop celebrated her retirement from active professional life with an "International Chromatographers Meeting" 25–27 June in Nuremberg-Lauf. It was an exciting event of scientific exchange in a family type atmosphere. Among more than 100 invited guests the lifework of Mrs. Hahn-Deinstrop was appreciated which is characterized by her highly committed dedication to planar chromatography.

Determination of progesterone in drug release media



▲ Dr. Ahmad Jamshidi

Dr. Jamshidi*, analytical chemist at the department of Novel Drug Delivery Systems (NDDS) at Iran Polymer and Petrochemical Institute (IPPI), employs chromatographic separation techniques especially instrumental planar chromatography to develop new and improved quantitative analytical methods for determination of drugs, steroids, antibiotics, vitamins, additives and naturally occurring compounds in a variety of sample matrices. He prefers HPTLC because it is flexible, inexpensive, time-saving and does not produce toxic waste.

Introduction

The main objective of controlled release drug delivery systems is to prolong the duration of action of an active agent to maintain effective drug levels over a desired period of time. A variety of polymers can aid controlled drug release which results from the interaction of polymer and the drug. Among the new polymeric delivery systems, silicon-based progesterone releasing systems have drawn attention. Optimization of the release profile demands large numbers of progesterone determinations in the release media covering a wide range of analyte concentration.

In this case, HPTLC determination is very suitable because it is inexpensive and time-saving. Up to 15 samples (in duplicate) can be determined simultaneously in less than 15 min. The solvent consumption is only about 10 mL. By pre-washing in suitable mobile phases followed by drying HPTLC plates can be recycled up to 10 times, without significant consequences for the reliability of analytical results. The proposed method is simple, rapid, accurate and precise. It is suited as a high-throughput method with a significant reduction in working time, supplies and solvents.

Chromatogram layer

HPTLC plates silica gel 60 F₂₅₄ (Merck), 20×10 cm, prewashed by developing first in chloroform – methanol 1:1 and then in the mobile phase followed by drying on the TLC plate heater at 80 °C for 15 min.

Sample application

Bandwise with Automatic TLC Sampler, 36 tracks, band length 2 mm, track distance 4 mm, distance from the side 20 mm, distance from lower edge 10 mm, application volume 50–3000 nL depending on sample concentration.

Chromatography

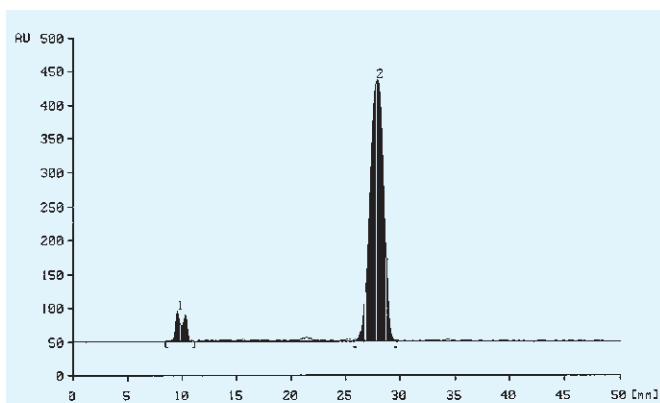
In AMD2 system with toluene – 2-propanol 10:1, without pre-conditioning, drying time 10 min, migration distance 60 mm. AMD2 was employed also for prewashing of the layer, because it is fully automated and GLP compliant.

Densitometry

TLC Scanner 3 with CATS software, absorption measurement at 252 nm followed by spectra recording from 200 to 360 nm

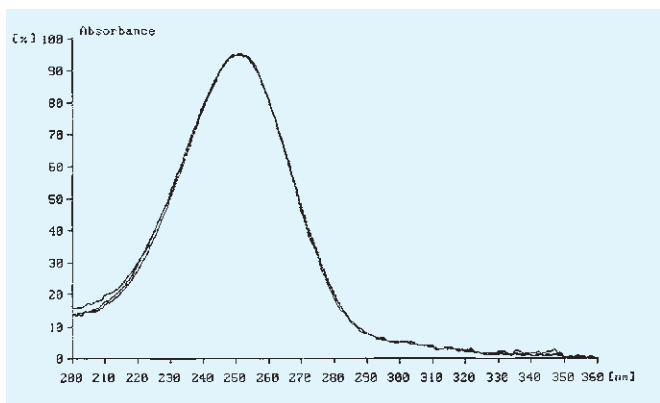
Results and discussion

A representative densitogram of a simulated sample is shown. During evaluation of the chromatogram, the simulated and spiked samples gave the same hR_f values as the standard and were well separated from matrix components. Positive identity of progesterone is indicated by spectra comparison of sample and standard. The optimum wavelength at 252 nm for densitometric evaluation was confirmed by the in-situ UV spectrum. Calibration was performed for a wide range (25.7–515.0 ng/zone) and the linear working range was established in a limited calibration range (25.7–154.5 ng/zone).



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▲ Densitogram of simulated sample of progesterone (2= progesterone peak) at UV 252 nm



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▲ Comparison of UV-spectra of progesterone sample and standard from 200–360 nm



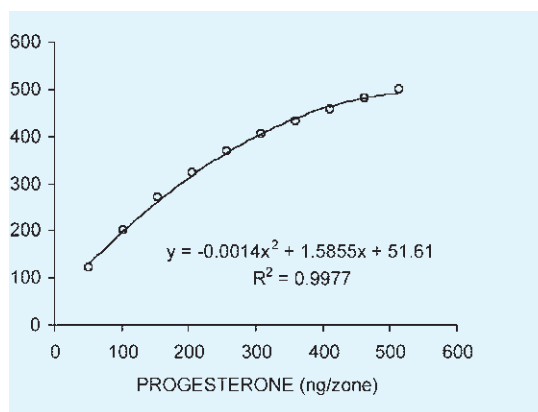
27

CAMAG TLC Scanner 3

Dr. Jamshidi employs classical densitometry with the CAMAG Scanner 3 for highest accuracy of quantitative evaluation. The use of the optimal measuring wavelength is indispensable when spectral selectivity and sensitivity is essential.

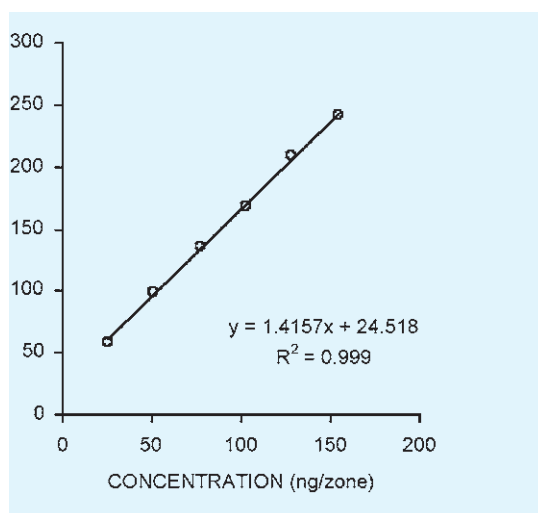
He also uses spectra recording as additional confirmation which is only offered by classical densitometry. The spectral range of 200–360 nm used by him is only a selected part of the complete spectral range from 190 to 800 nm which can be utilized for spectra recording. Absorption spectra (for fluorescent substances also excitation spectra) can be recorded for additional confirmation of identity of substances. Correlation of sample and standard spectra as well as correlation of spectra within a peak (peak purity) can be calculated.

CAMAG TLC Scanner 3 with the software “winCATS Planar-Chromatography Manager” is the most advanced work station for densitometric evaluation of planar chromatograms. For additional requirements various options are available.



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▲ Polynomial calibration curve of progesterone from 25.7–515.0 ng/zone



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▲ Linear working range of progesterone from 25.7–154.5 ng/zone

Repeatability of the method was determined as standard deviation calculated from the amounts of seven simulated progesterone samples determined on the same plate at three concentration levels in the lower, middle and upper range. Recovery data of simulated samples indicated high recovery of progesterone in the linear working range. Reproducibility data based on recycled plates show that HPTLC plates can be recycled without significant consequences on the reliability of analytical results. X-fold re-use of recycled plates has yet to be investigated and must be carefully monitored during utilization.

Repeatability

Measurements	Progesterone concentration (ng/zone)		
	I	II	III
1	27.14	72.80	144.96
2	27.69	73.83	144.11
3	27.70	73.81	144.11
4	28.00	73.92	143.90
5	28.14	73.81	144.00
6	28.15	74.80	143.95
7	27.98	73.73	143.96
Mean (ng/zone)	27.83	73.81	144.14
SD	0.36	0.58	0.37
RSD	1.29	0.78	0.26

Recovery of progesterone

Expected (ng/zone)	27.70	72.81	144.89
Found (ng/zone)*	27.97	72.72	145.14
Recovery rate (%)	100.97	99.88	100.17

*Average of seven different experiments

Reproducibility based on recovered plates

x-fold recov. plates	Mean ± SD (n=3)			Recovery (%)			RSD (%)		
	I	II	III	I	II	III	I	II	III
1	28.14 ± 0.44	72.81 ± 0.55	144.80 ± 0.11	101.59	100.00	99.93	1.56	0.75	0.07
2	28.15 ± 0.36	73.02 ± 0.48	144.11 ± 0.09	101.62	100.28	99.46	1.28	0.66	0.06
3	26.69 ± 0.27	73.73 ± 0.44	143.96 ± 1.32	96.35	101.26	99.36	1.01	0.60	0.92
4	27.03 ± 0.60	70.81 ± 1.02	144.00 ± 0.19	97.58	97.25	99.38	2.22	1.44	0.13
5	28.00 ± 0.55	72.13 ± 0.09	143.90 ± 0.58	101.08	99.06	99.32	1.96	0.12	0.40
6	27.14 ± 0.32	74.80 ± 0.88	144.11 ± 0.60	97.97	102.7	99.46	1.18	1.18	0.41
7	27.69 ± 0.39	73.73 ± 0.35	143.95 ± 0.87	99.96	101.26	99.35	1.41	0.47	0.60
8	27.70 ± 0.50	73.81 ± 0.44	144.96 ± 0.10	100.00	101.37	100.05	1.80	0.60	0.07
9	28.15 ± 0.54	71.90 ± 0.07	144.14 ± 0.39	101.62	98.75	99.48	1.91	0.09	0.27
10	27.02 ± 0.48	73.81 ± 0.36	144.00 ± 1.00	97.54	101.37	99.38	1.77	0.49	0.69

Further information is available on request from the author.

*Dr. Ahmad Jamshidi, Department of Novel Drug Delivery Systems, Iran Polymer and Petrochemical Institute, P.O. Box 14965/115, Tehran, Iran, a.jamshidi@ippi.ac.ir

News from China

In China, the State Food and Drug Administration (SFDA) has recently initiated a „Mobile Drug Testing Program“ in order to promote the safe use of drugs in rural areas and to better enforce the relevant regulations. This program was co-developed by the National Institute for the Control of Pharmaceutical and Biological Products (NICBPB). These mobile units are equipped with two operating systems: a comprehensive drug database and a rapid drug identification system. Both systems are in place, in order to provide a highly efficient platform to identify adulterants or counterfeit drugs found in the market particularly in the smaller cities and villages.

The identification system utilizes mainly three different analytical methods: TLC (Thin Layer Chromatography), NIR (Near-Infrared) and chemical analysis. In January of 2004, during the annual working meeting of the SFDA; Ms. Yi Wu, Health minister and Vice Premier of the People’s Republic of China, has personally inspected these specially equipped vehicles and approved this program for promoting the drug control in the remote areas of China. Ms. Wu has requested the SFDA to further expand this program across the country. In March 2004, the China’s Ministry of Finance has approved a budget for 344 mobile drug-testing vehicles by 2006, to be distributed throughout the country.

CAMAG and CAMAG China Supporting Unit, as the leader of TLC in both instrument manufacturing and method developments, are honored to be appointed to actively participate in the technical and training aspects of this program and to be a major instrument supplier for this new health promotion program.



▲ One of the SFDA’s Mobile Drug Testing trucks. TLC is one of the major analytical tools of the equipment in these trucks, as is shown on the bulletin board.



▲ The inspection of the SFDA mobile drug testing program by the Vice Premier of the People’s Republic of China and PRC Health minister, Ms. Wu (lady in the middle); Director of SFDA, Mr. Zhang (on the left) and Deputy Director of National Institute of the Control of Pharmaceutical & Biological Products P.R.China, Mr. Jin (behind Ms. Wu)

New HPTLC-MS method for determination of heterocyclic aromatic amines



▲ Research group of planar chromatography: Stephanie Häberle, Ute Jautz, Dr. Gerda Morlock* (from left to right)



▲ Prof. Dr. Wolfgang Schwack

For over 15 years the working group of Professor Schwack, Institute of Food Chemistry at the University of Hohenheim, Stuttgart, has been investigating the photochemistry of pesticides in plant cuticles, the formation and determination of bound residues in and on foodstuff derived from plants, the residue analysis of dithiocarbamate fungicides, the photochemistry of UV filters in cosmetic sun protection agents as well as the analysis and bio-availability of carotenoids. Besides immuno-chemical methods all chromatographic techniques are employed, incl. LC-MS, GC-MS and HSCCC, as well as IRMS. In addition, since 2003 one focus is on research in modern planar chromatography. A new HPTLC procedure for the determination of heterocyclic aromatic amines (HAA) is presented. For coupling with mass spectrometry a new online extractor has been employed.

Introduction

Heterocyclic aromatic amines (HAA) are formed during heating of meat and fish products. Based on results of animal tests they are considered mutagenic and carcinogenic. In food more than 20 different HAA with similar structure have been identified at the low $\mu\text{g}/\text{kg}$ -level. They are formed from the precursors creatinine, amino acids and reducing sugars in complex reactions at high temperatures. For quantitative determination commonly RP-HPLC with combined UV-DAD and fluorometric detection is used. Further analytical methods are LC-MS, LC-MS/MS, GC-MS or CE-UV/DAD. These procedures are all demanding and their daily sample throughput is low. A further difficulty of the HPLC method is associated with sample preparation by solid phase extraction. Proper separation of both substance groups into polar and non-polar fractions is problematic, because some HAA are eluted in both fractions. Evaluation of those has to be performed by determination of the peak sum.

A procedure for rapid, cost-efficient yet simple quantitative determination of HAA was desired for a long time. A new planar chromatographic method based on Automated Multiple Development (AMD) was developed. Owing to parallel analysis, planar chromatographic separation is faster than HPLC by a factor of seven and enables a more effective screening for affected samples. Consumption of solvent is reduced about 90%.

Because HPTLC stores all substance zones on the plate the mass spectrometer can be selectively employed to confirm positive results. This allows rapid, cost-effective, and still selective and sensitive planar chromatographic determination with only limited occupation of the MS device.

Sample preparation

30 g sample are homogenized and mixed with Extrelut^{®NT}. HAA are extracted and adsorbed on cation exchanger and on a C18 cartridge subsequently, each eluted and concentrated.

Layer

HPTLC plates silica gel 60 WRF_{254s} (Merck), 20 x 10 cm, prewashed with methanol by chromatography and dried for 30 min at 120 °C

Sample application

Bandwise with Automatic TLC Sampler 4, 16 tracks, band length 8 mm, track distance 10 mm, distance from the side 20 mm, distance from lower edge 8 mm, application volume 2 µL

Chromatography

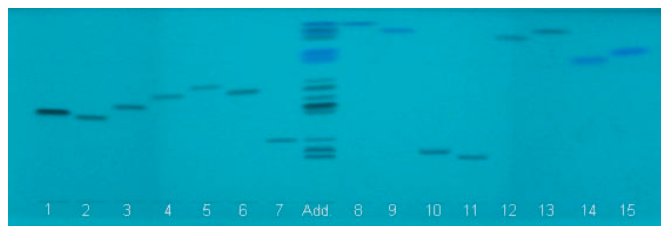
In AMD2 system multiple development over 6 steps with diethyl ether, methanol and chloroform, alkaline conditioning via gas phase, max. migration distance 60 mm

Densitometric evaluation

TLC Scanner 3 with winCATS software, multi-wavelength (MWL) scan of absorption at 252, 262, 316 nm and fluorescence at 366/>400 nm, polynomial evaluation via peak area and height

Results and discussion

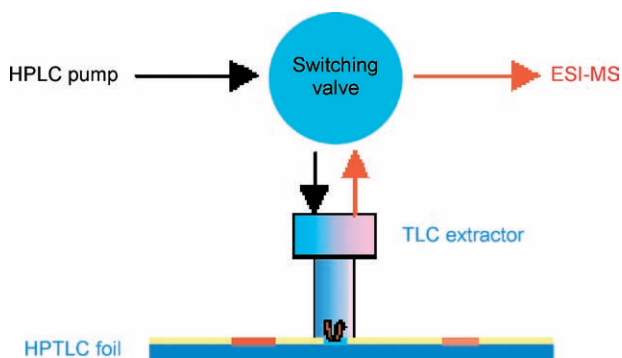
Limit of quantitation of the 15 HAA standard substances was established with respect to the signal to noise ratio in the lower nanogram range (between 1 and 45 ng absolute on the plate) for fluorescence and absorption measurement, respectively. Fluorescence enhancement which could improve LOQ of fluorescent HAA by a factor of 10–100 is still under investigation.



▲ Chromatogram of 15 HAA at UV 254 nm (reflectance), AMD separation on HPTLC plates silica gel 60 WRF_{254s}, track 1–15 standard substances, middle track standard mixture

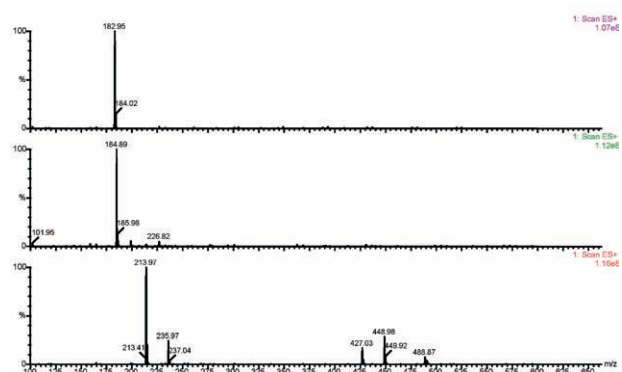
For online coupling of HPTLC with MS a new extractor developed by Dr. Luftmann** was employed. The selected system is appealing because of its simplicity and viability. For an existing LC-MS system only the extractor device is required to confirm within one minute a positive result of interest by an online mass

spectrum. The principle of electro spray ionization which is widely used and proven in LC-MS is thus adopted for planar chromatography.



▲ Schematic of the HPTLC-MS online extractor (courtesy of Dr. Luftmann)

Rapid and contamination-free online extraction (ca. 0,5 min time of elution/zone) with minimal solvent consumption is significantly faster than scraping off, elution and injection via direct MS probe. The principle of extraction can be employed for all layer materials. At the moment it is limited to aluminum or plastic foils as plate support because foils provide a better seal to the extractor head than glass.



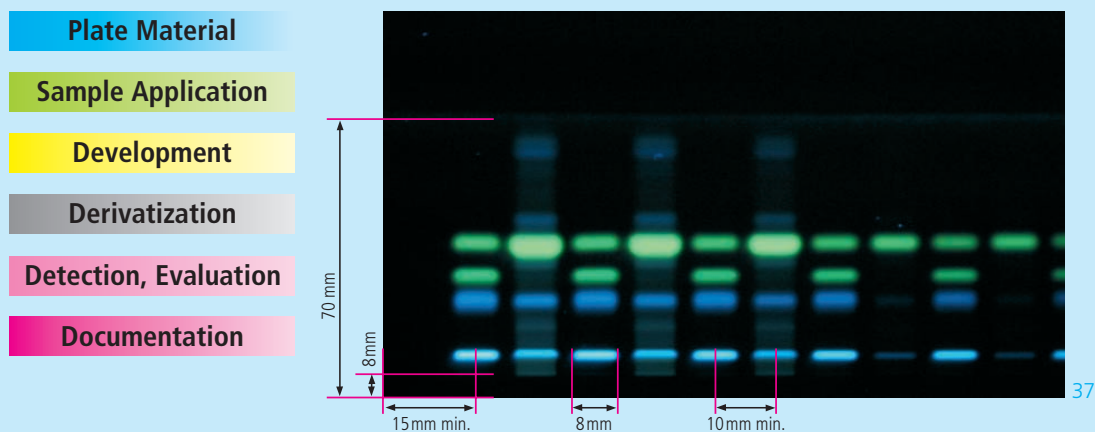
▲ Online HPTLC-MS spectrum of harman (top, m/z 183 [M+H]⁺), Glu-P-2 (middle, m/z 185 [M+H]⁺) and MelQx (bottom, m/z 214 [M+H]⁺, m/z 236 [M+Na]⁺, m/z 427 [2M+H]⁺, m/z 449 [2M+Na]⁺)

Further information is available on request from the authors.

* Dr. Gerda Morlock, Universität Hohenheim, Institut für Lebensmittelchemie (170), Garbenstr. 28, 70599 Stuttgart, gmorlock@uni-hohenheim.de

** Luftmann H (2004) Anal Bioanal Chem 378: 964-968, email Luftman@uni-muenster.de

Standardization – a key element of modern Planar Chromatography



The availability of state of the art instrumentation for all steps of the planar chromatographic process, the use of HPTLC plates, and the control of all instruments used by the powerful winCATS – Planar Chromatography Manager software enable modern planar chromatography to stand strong among today's high-performance separation techniques.

Due to its off-line principle, HPTLC can offer an enormous flexibility and an almost unlimited number of choices in the selection of individual parameters. Unfortunately, up to now there is no agreement about what analysts consider as "standard parameters" or "standard procedure". This fact often limits the repeatability of published HPTLC methods and has caused some concerns about the reliability of qualitative and quantitative data in general. On the other hand, if methods are properly documented, validated, and executed, precise and accurate results can be obtained. Therefore, standardization becomes not only the key element of planar chromatography but also one of its major growth factors. Obviously it can help to increase transparency and transferability of data.

Currently the general chapter of TLC in the European, Chinese and United States Pharmacopoeias is reviewed with the goal of incorporating advancements in scientific knowledge and developments in technology. Thus, the chances for introducing a standardized and internationally harmonized approach to modern planar chromatography are better than ever. CAMAG is prepared to support this important process with emphasis. A Standard Operating Procedure for HPTLC is proposed by CAMAG. This SOP can be found on the website www.camag.com together with information about recently published papers on the topic. Your comments are always welcome (email to info@camag.com).