CBS 99 • September 2007
CAMAG BIBLIOGRAPHY SERVICE PLANAR CHROMATOGRAPHY

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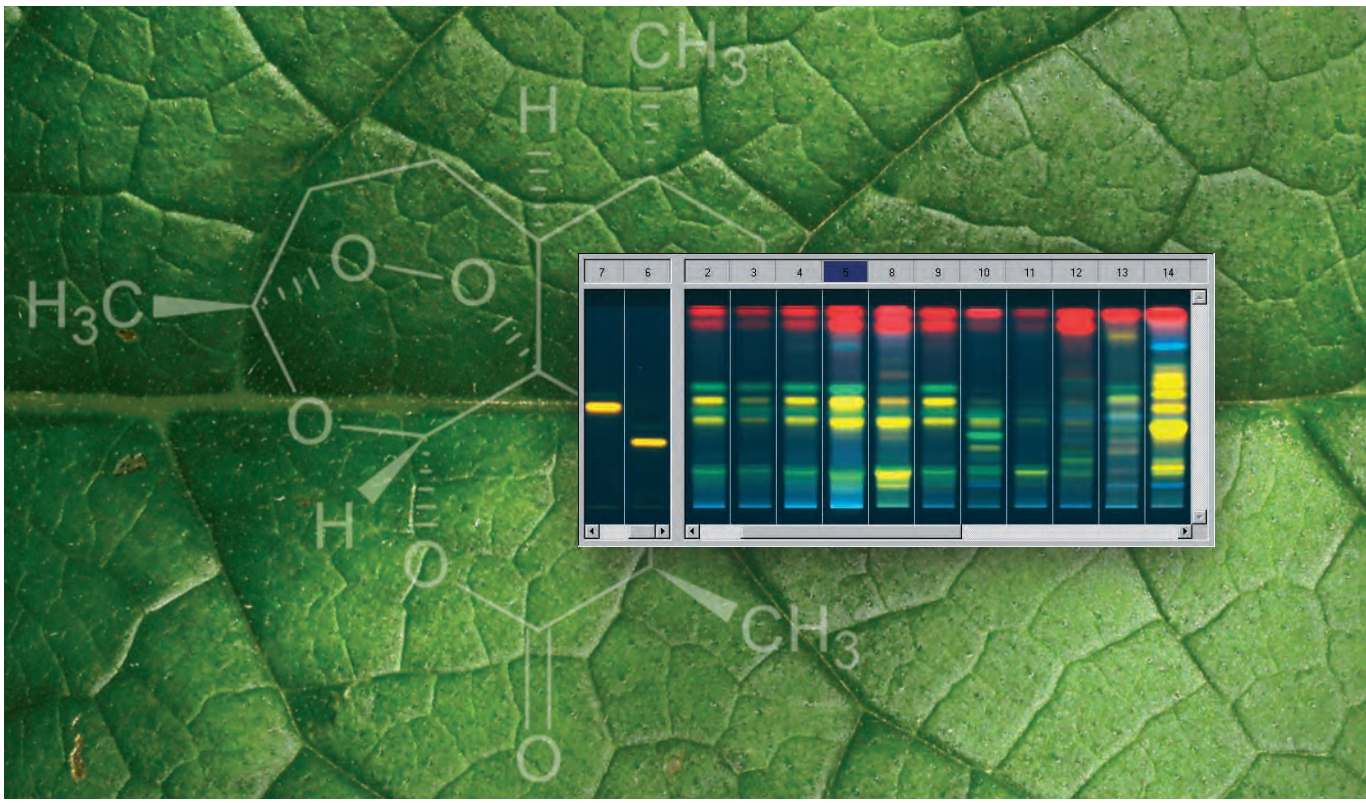

Analysis of herbals using Planar Chromatography – one step ahead due to its image feature

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Planar Chromatography
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Column: Know CAMAG

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American Herbal Pharmacopoeia: HPTLC Analysis of Botanical ingredients

*Roy Upton, PhD** ▶
President of American Herbal Pharmacopoeia

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In recent years, hundreds of common herbal ingredients from Aloe vera to ginseng have been used in the manufacture of a host of products ranging from toilet paper to chewing gum to traditional and modern medicines. The increased use of herbal ingredients requires tools for determining their identity, purity, and quality. These tools have to be technically sufficient, rapid, and cost-effective with a focus on compliance with good manufacturing practices (GMP) requirements.

For medicines, such quality control standards have been included in the European and national pharmacopoeias for decades. However, creating standards for botanical ingredients in North America and much of the developing world is new. The lack of quality standards has resulted in numerous cases worldwide of mild to serious adverse effects ranging from hepatotoxicity to death.

Many if not most of these adverse events would have been avoidable had identity standards and effective analytical tools been available and used by industry. The American Herbal Pharmacopoeia (AHP) was founded for the express purpose of developing quality control and identity standards for botanical products. AHP acknowledges the importance of medicinal plants in health care and recognizes that quality standards must be applied to herbal medicinal products in order to receive their benefits.

For AHP, planar chromatography is an indispensable tool. Using HPTLC multiple samples of plants can be screened in a fraction of the time and at a fraction of the cost that would be required using other analytical tools. As an analytical method HPTLC also provides multiple ways in which a single sample can be viewed and provides a level of interpretive information that is greater than most other commonly used analytical techniques. For basic quality assurance of botanical ingredients and many finished products, HPTLC is the chemical characterization tool of choice for AHP. For industry, it is clearly one of the most versatile, practically useful, and cost-effective analytical tool for the quality assurance of botanicals. The use of HPTLC is growing in North America and its potential for growth is great and greatly warranted.

*Executive Director, American Herbalists Guild, Soquel, CA, USA, herbal@got.net

Planar chromatography – indispensable when checking the quality of botanical preparations

◀ *Prof. Dr. Beat Meier*[*] *President of the Phytochemistry Group of the Swiss Pharmacopoeia* 2

It is probably no coincidence that mentors of thin-layer chromatography like Egon Stahl or Helmut Jork were scientists with a special interest in pharmacognosy. Before the introduction of TLC, the identification of medicinal plants was restricted to their morphologies and a variety of wet chemical color reactions. TLC provided a relatively simple method for obtaining a deeper insight into the plant's "chemistry". There was a variance in the secondary metabolites from one species to another, which facilitated the identification of plants using this method. This was particularly significant in cases where the structural elements of plant were no longer present as a result of extraction and applied to all types of extracts and end products, regardless of whether the preparations were used for medicinal purposes or as food. Unfortunately, for a period of time thin-layer chromatography failed to keep pace technologically with column chromatography and as a result gained a poor reputation for unsatisfactory reproducibility. However, this was not the fault of the method so much as that of users, who paid scant attention to ensuring conditions that would facilitate thin-layer chromatography's reproducibility. TLC was widely perceived as a cheap, simple method, which clearly obstructed the necessary investment in innovative technology. Fortunately, attitudes in recent years have changed.

Take the European pharmacopoeia as an example: a few years ago, the general monograph on thin-layer chromatography was revised to bring it into line with modern requirements. It is now possible – as in HPLC – to work with high-performance materials, which much improves separations, saves solvent, and leads to much faster results. It is obligatory for herbal monographs in the European Pharmacopoeia to establish HPTLC chromatograms that are as specific as possible to facilitate identification. However, HPTLC can be used not only for identification purposes; it is also practical as a means of demonstrating the presence of undesirable substances, such as aristolochic acids, and is an important tool in stability tests.

To date, no other analytic procedure has been able to provide a visual fingerprint showing the complexity of a multicomponent mixture represented by an extract better than the one achieved with HPTLC. Simply being able to apply numerous samples adjacent to each other facilitates in-process control between various source materials at various stages of extract preparation as well as between various batches and products. It also permits us to observe changes over time. As a result, HPTLC has established itself as an indispensable part of medicinal plant analysis.

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Prof. Dr. Beat Meier is also President of the GA (Society of Medicinal Plant Research) Permanent Commitee for the Manufacture and Quality Control of Medicinal Plant Products.

Quantitative HPTLC analysis of artemisinin in dried leafs of *Artemisia annua*

▲ *The CAMAG Laboratory: Dr. Anita Ankli, Valeria Widmer, Dr. Eike Reich, Daniel Handloser, Mario Steiner (from left to right)* 3

The CAMAG-Laboratory* is a team of scientists and application specialists with broad experience in many fields of application and in-depth knowledge of Thin-Layer Chromatography. They work toward the world-wide acceptance of HPTLC as a standardized analytical technique. Providing a broad range of services at various levels it is their mission to help people solve their analytical problems with HPTLC.

Introduction

Currently malaria is pointed out by the WHO as one of the greatest threats to human health. Various medicines have been tried for the prevention and treatment of malaria since the mid-20th century, but increasing resistance to formerly effective cures has once again made malaria therapy a major problem. Currently the most effective medical approach is the so-called artemisinin-based combination therapy (ACT) using artemisinin isolated from Artemisia annua and synthetically derived substances. Because a total synthesis of artemisinin is not yet feasible the focus is put on a large scale growing of plants with high artemisinin content and determining appropriate harvest time and extraction procedures.

Numerous papers have been published on analytical procedures to support these tasks, but most reported methods are very complex, expensive or just not practical. For example the separation of artemisinin from other sample components and the detection by sulfuric acid reagent is insufficient. Hence a specific, yet rapid and simple HPTLC method for the quantitative determination of artemisinin in leafs of Artemisia annua was developed [1]. The absence of any chromophore required derivatization with anisaldehyde reagent. Over a wide concentration range of 0.05 - 3.25 % in the dried leaf the proposed method can be applied for screening the artemisinin content of 9 samples per hour and, in the linear working range, for quantitative assay.

Sample preparation

Dried leafs of *Artemisia annua* were finely milled and 200 mg were mixed with 10 mL of toluene and extracted by sonication for 10 min at room temperature (23 °C). Following centrifugation the supernatant was used as test solution for screening. For the assay samples were diluted appropriately.

Standard solution

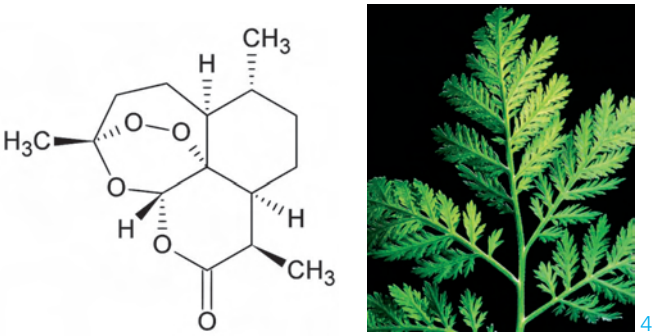
For screening 10 mg artemisinin were dissolved in 100 mL toluene. For assay the solution was diluted with toluene 1:10.

Layer

HPTLC plate silica gel 60 F_{254} Merck, 20 x 10 cm

Sample application

As 8 mm bands using the Automatic TLC Sampler 4, application volume 2–10 µL for samples and standards, track distance 10 mm, first application position 20 mm, distance from lower edge of plate 8 mm



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▲ *Structure of artemisinin and leaf of Artemisia annua*

Chromatography

In a twin trough chamber with cyclohexane – ethyl acetate – acetic acid 20:10:1 pre-saturated (filter paper) for 20 min with 10 mL of mobile phase per trough, migration distance 70 mm from the lower plate edge

Derivatization

Immersion in anisaldehyde reagent (2 mL anisaldehyde dissolved in a mixture of 100 mL ethanol and 80 mL water plus 20 mL acetic acid and 4 mL sulfuric acid) for 1 s using the Chromatogram Immersion Device III. After 1 min the plate is heated on the TLC Plate Heater at 100 °C for 12 min.

Documentation

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

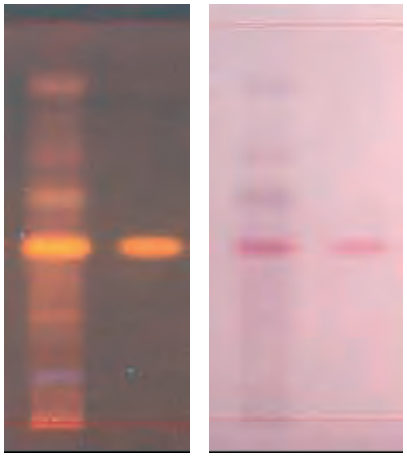
Evaluation

TLC Scanner 3 in fluorescence mode at 520/>540 nm using a tungsten lamp, slit size 4 x 0.2 mm; for screening optional video densitometry by VideoScan

Note: The difference between excitation/emission wavelengths should be at least 30 nm. In this case it is a compromise due to the given edge filter.

Results and discussion

Artemisinin is derivatized to an orange fluorophore using anisaldehyde reagent which can be detected at 520/>540 nm very specifically.



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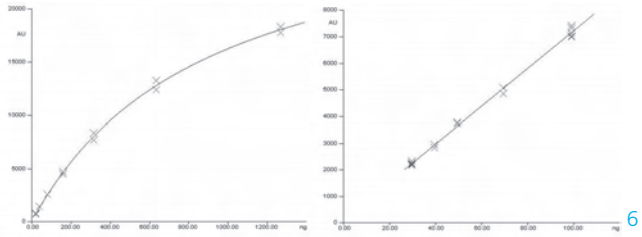
▲ *Chromatogram section of Artemisia annua extract and artemisinin standard*

Compared to a conventional absorption measurement at 535 nm the signal obtained from the same zone in the fluorescence mode is about 3 times higher. Using the Michaelis-Menten 1 regression it is possible to determine artemisinin in a wide concentration range from 0.05 % to theoretical 3.25 % in dried leafs. The linear working range from 30 to 120 ng/zone was established for precise assay.



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▲ *Calibration curve for artemisinin screening (left) and assay (right)*

Validation data

Parameter of validation	Results for artemisinin
Screening range	20–1300 ng/zone (0.05–3.25 %) y = 30722.958 x / (853.168 + x) *RSD* = 3.67 %
Linearity	30–120 ng/zone (0.075–0.3 %) y = 122.6 + 71.04 x *RSD* = 2.92 %, r = 0.9983
Precision	*RSD* = 0.77 % (*n* = 5)
Repeatability	*RSD* = 1.9 % (*n* = 3)[1]
Intermediate precision	*RSD* = 1.2 % (*n* = 3)[2]
Precision of the extraction	*RSD* = 5.2 % (*n* = 5)[3]

[1] One aliquot in triplicate on three plates on the same day

[2] One sample on three plates on different days

[3] 5 aliquots of the same sample on one plate

Further information is available from the authors on request.

[1] Application Note A 86.1 (http://www.camag.com/laboratory/methods/index.html) and J. Liq. Chromatogr. Rel. Techn. 15, 2209, 2007

*www.camag-laboratory.com

Sildenafil determination in pharmaceutical products and aphrodisiac herbal preparations

▲ *Dr. Ehab A. Abourashed*

Dr. Abourashed* is director of quality assurance at ElSohly Laboratories, Inc. (ELI) and adjunct research associate professor at the National Center for Natural Products Research (NCNPR), University of Mississippi, USA. His research focuses on quality assessment/fingerprinting of herbal products as well as drug discovery from natural sources. He also employed HPTLC as a platform for screening and evaluation of antioxidant activity of various extracts and pure compounds using the DPPH free radical scavenging assay [1]. In his view the well-resolved HPTLC fingerprint combined with densitometry is a unique characterization feature.

The quality of the fingerprints is most appreciated by natural product chemists who are used to visual examination of complex extracts. HPTLC has the advantage of simplicity, accuracy/precision, high speed and cost effectiveness and it has all the properties required to perform reliable pharmaceutical analyses.

Introduction

Since its introduction in the 1990th, sildenafil has become one of the most important drugs to treat erectile dysfunction. Due to its relevance, several analytical methods have been reported for sildenafil quantification in pharmaceutical formulations and other matrices. More than 60 % of these methods were based on HPLC while TLC [2] was used only for qualitative purposes. Considering the advantages of HPTLC, a new high-throughput method was developed [3] to quantify sildenafil in pharmaceutical products by HPTLC/UV.

Sample preparation

Pharmaceutical products were extracted with 10 mL of distilled water in an ultrasonic bath for 5 min. 85 mL of methanol were added and the solution was sonicated for another 10 min. The solution was adjusted to 100 mL with methanol and diluted 1:5. For herbal products, the content of three capsules was extracted with methanol by ultrasonication for 15 min. 1 mL clear supernatant was diluted up to 25 mL with methanol.

Standard solutions

Sildenafil citrate was dissolved in methanol (1.052 mg/mL) equivalent to 0.752 mg/mL sildenafil (stock solution) and diluted 1:5 to obtain a 150 µg/mL sildenafil solution.

Layer

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

Sample application

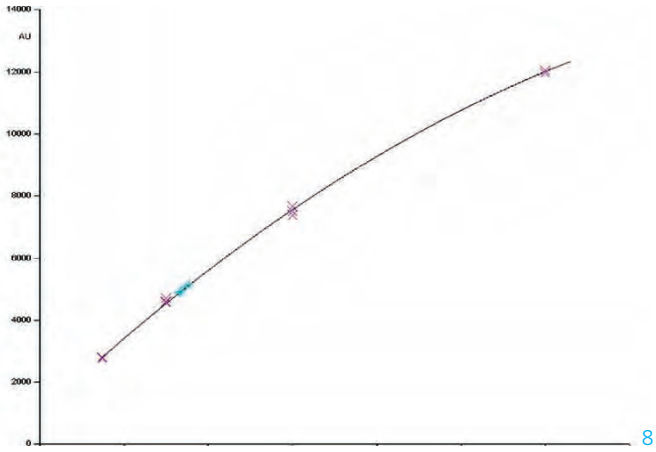
Bandwise with Linomat, application volumes of 4 µL for samples and 1 to 8 µL for the standard solution (150 to 1200 ng/band), band length 5 mm, track distance 9 mm, distance from lower edge 10 mm, distance from both sides 12 mm

Chromatography

In a twin trough chamber pre-saturated for 30 min with the mobile phase chloroform – methanol – diethylamine 9:1:0.1. The migration distance was 80 mm from the lower edge. Then the plates were dried in a stream of warm air for 1 min.

Densitometry

TLC Scanner 3 with winCATS software, absorbance measurement at 305 nm, polynomial calibration via peak area



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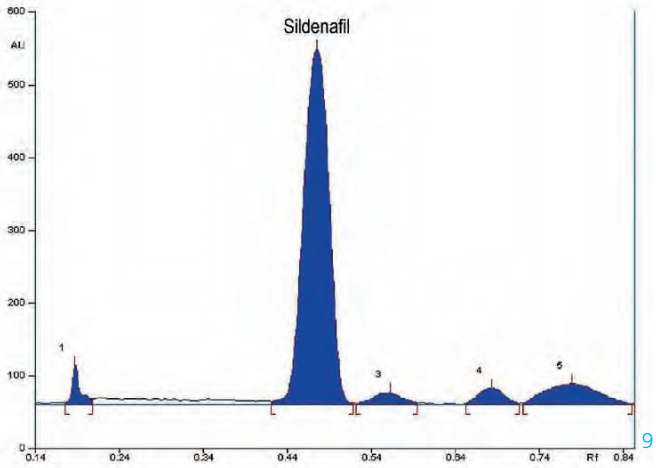
▲ *Sildenafil calibration curve from 150 to 1200 ng/band*



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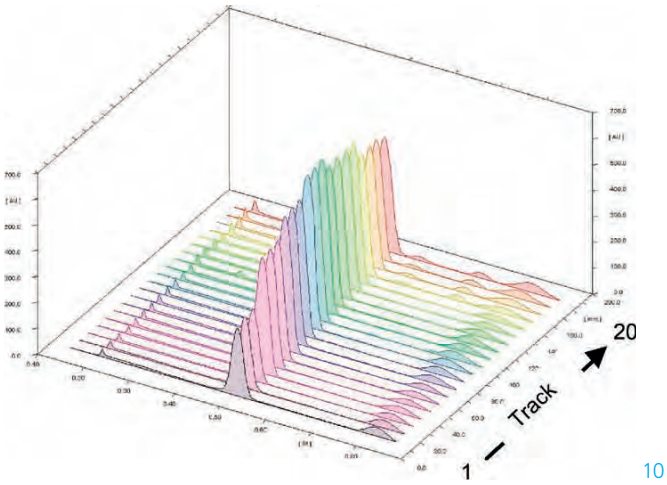
▲ *Chromatogram at 305 nm of a herbal product showing a well-defined sildenafil peak (272 ng/band)*



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▲ *3-D graphic of calibration standards (tracks 1–12; 4 levels in triplicate), pharmaceutical products (tracks 13–16) and herbal products (tracks 17–20)*

Note: Such a track pattern eases the assignment. However, it is recommended to apply standards and samples in alternation.

Results and discussion

The optimal wavelength at 305 nm was selected through the spectrum scan of sildenafil at hR_F 48 ± 1. The 4-level calibration (n = 3) from 150 to 1200 ng/band showed a polynomial regression ($y = -0.003x^2 + 12.726x + 978.663$) with $R > 0.9997$. Repeatability was determined applying the same sample (n = 6) on the same plate, showing relative standard deviations (*RSD*) from 0.7 to 3.1 % for four different plates. Inter-day precision was calculated applying the same sample (n = 6) on four different plates during 10 days, showing a *RSD* <1 %. Method accuracy was evaluated determining the recovery of spiked samples, resulting in a mean recovery of 98.2 % ± 3.3 % (*RSD* 3.4 %) for three different concentration levels. The sildenafil content in four pharmaceutical products ranged from 49.7 to 50.5 mg/tablet, corresponding to 99.4–100.9 % of the labeled value. One herbal product, declared to contain only natural ingredients, showed a sildenafil content of 85 mg/tablet without any indication on the label. The results proved the suitability of planar chromatography for quality control of sildenafil pharmaceuticals as well as to detect its presence as illegal ingredient in herbal products.

Further information is available from the author on request.

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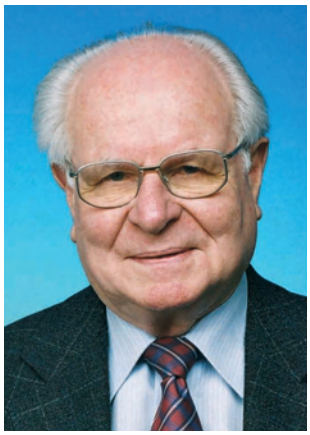
[1] E. A. Abourashed, Z. Naturfortsch. 60b, 1212–1218, 2005

[2] E. Mikami et al., Forensic Sci. Int. 130, 140–146, 2002

[3] E. A. Abourashed et al., JPC 18, 372-376, 2005

Dr. Dieter Jänchen on the occasion of his 80th birthday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When Dr. Jänchen registered a company under the name of "CAMAG Chemie-Erzeugnisse und Adsorptionstechnik (Muttenz) AG" on December 17, 1958, no one dreamt that it would develop into a company of world renown in the field of planar chromatography.

It all started out with a company producing aluminium oxide for chromatographic applications that was soon attracting the interest of bulk industrial users. The fine-grain share represented the entry into thin-layer chromatography, which was beginning to gain worldwide acceptance at this time. It was in this situation that our founder discerned the development of a microanalytical separation process. We owe it to him in particular that the rather simple TLC handling of those pioneering years gradually developed into a range of instruments fully deserving of the description "high-tech" today. At the same time, he found valuable support from creative individuals who shared his interests: for instance, Professors Kaiser, Ebel and Jork, as well as Dr. Burger. In-house he built up a team that opened up new avenues in research and development, production and distribution.

Initially, Dr. Jänchen himself was the most adept marketer of them all. He quickly realized the importance of international markets beyond Germany and Switzerland for the company's growth and development. When TLC's instrumental development entered its decisive phase, his commitment had already ensured his success worldwide. By 1962, he had acquired agencies in 18 countries by himself; a year later, that figure had doubled. One of his strokes of genius was the CAMAG Bibliography Service (CBS). Published twice yearly, it is far more than a company newspaper in the usual sense of the term. The collection of abstracts on important TLC/HPTLC topics (CCBS) has developed into a database on the subject of international renown.

His 100% commitment to instrumental TLC/HPTLC at the highest possible level has made him the sort of boss who is a source of constant inspiration to his staff. Those fortunate enough to know him more closely will be familiar with his humour and very human warmth. He continues to give us the benefit of his enormous experience and his regular presence on site. His efficient, straightforward approach to thinking and decision-making are as much appreciated as ever. Even today, it is the most natural thing in the world for his employees to call him "Boss".

In his leisure time, too, Dr. Jänchen's aspirations were – literally – as high as those in his daily business. For 26 years he was a dedicated flying instructor, qualified to teach both aerobatics and cloud flying, and passing on his valuable expertise to trainees. In gliding, he holds Gold C and three-diamond certification together with a pilot's licence for instrument-controlled flight. His most spectacular powered-flight experience was a transatlantic flight with a small single-engine aircraft.

On the occasion of his 80th birthday in June 2007, we wish our "Boss" all the very best, an endless supply of energy for all his many interests and many more years of happiness with his wife Brigitte!

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PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

Gemäss WHO beläuft sich momentan der weltweite Markt für Phytotherapeutika auf jährlich über 44 Milliarden Euro mit steigender Tendenz. In China beträgt der Anteil an traditionellen pflanzlichen Zubereitungen bis zu 50% des medizinischen Gesamtverbrauchs. In Afrika nutzt sogar bis zu 80% der Bevölkerung die traditionelle Medizin als Basis ihrer Gesundheitsvorsorge.

Bei Verwendung eines Phytotherapeutikums, bei dem die Pflanzenart verwechselt wurde, können gefährliche gesundheitliche Nebenwirkungen auftreten. Gleiches gilt für die unkontrollierte und unangemessene Anwendung der traditionellen Medizin. Zudem kann die steigende Nachfrage für Phytotherapeutika und der grosse wirtschaftliche Gewinn dabei durch die übermässige Ernte der Rohmaterialien eine Bedrohung für den Artenreichtum darstellen oder den Anteil an verfälschten pflanzlichen Arzneimitteln in die Höhe treiben.

Daher ist die Analytik von Pflanzeninhaltsstoffen eine essentielle Basis, um solche negativen Effekte zu kontrollieren. In diesem CBS handeln mehrere Beiträge (S. 2–7) über Phytotherapeutika, und sie zeigen deutlich den gewinnbringenden Einsatz der Planar-Chromatographie. Die Planar-Chromatographie ist hier nicht nur dank der digitalen Bilddokumentation überlegen, sondern auch bei der Bioaktivitäts-basierten Detektion (S.11–13), einem neuartigen Konzept in der Analytik.

Herzlichst Ihre

Gerda Morlock

Gerda Morlock
cbs@camag.com

Dear friends

According to the WHO the global market for herbal medicines currently stands at over US $ 60 billion annually and is growing steadily. In China, traditional herbal preparations account for up to 50% of the total medicinal consumption. In Africa, even up to 80% of the population uses traditional medicine for primary health care.

However, taking a herbal preparation made from the wrong species of plant as well as the unregulated or inappropriate use of traditional medicines can have negative or dangerous effects on health. Additionally the growing herbal market and its great commercial benefit can pose a threat to biodiversity through overharvesting of the raw material or it can soar the adulteration of natural medicinal preparations and products.

Hence, the analysis of botanicals is an essential basis to control such adverse effects. In this CBS issue several articles (p. 2–7) are dealing with botanicals and illustrate impressively the benefits of planar chromatography. Planar chromatography is one step ahead not only due to its image feature, but also due to bioactivity-based detection (p. 11–13), an innovative concept of analysis.

Sincerely,

Gerda Morlock

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CAMAG

SEPTEMBER 2007 99

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

1. Reviews and books

99 002 T. KOWALSKA, J. SHERMA, Eds.: Preparative Layer Chromatography. Chromatographic Science Series, No. 95, CRC Press - Taylor and Francis Group, Boca Raton, 2006, 424 pp. Designed as a practical, comprehensive source of information on the field of classical preparative layer chromatography (PLC), the monograph is a valuable and important supplement to the existing vast chromatographic literature, demonstrating the potential of PLC for separation and isolation of pure compounds, even from very complex mixtures. The book is organized on two parts, the first of which covers the theory and up-to-date procedures of PLC (chapters 1 through 8), while the second (chapters 9 through 16) includes applications to a selection of the most important classes and sample types. Section I: Introduction; Adsorption Planar Chromatography in the Nonlinear Range: Selected Drawbacks and Selected Guidelines; Sorbents and Precoated Layers in PLC; Selection and Optimization of the Moblile Phase for PLC; Sample Application and Chromatogram Development; Application of Horizontal Chambers; Location of Separated Zones by Use of Visualization Reagents, UV Absorbance on Layers Containing a Fluorescent Indicator, and Densitometry; Additional Detection Methods and Removal of Zones from the Layer. Section II: Medical Applications of PLC; PLC of Hydrophilic Vitamins; Preparative Layer Chromatography of Natural Mixtures; The Use of PLC for the Separation of Natural Pigments; Application of PLC to Inorganics and Organometallics; PLC in a Cleanup and Ground Fractionation of Geochemical Samples; The Use of PLC for Isolation and Identification of Unknown Compounds from the Frankincense Resin (Olibanum): Strategies for Finding Marker Substances.

preparative TLC, review 1a

99 003 J. SHERMA*, B. FRIED (*Department of Chemistry, Lafayette College, Easton, PA, USA; shermaj@lafayette.edu).: Thin layer chromatographic analysis of biological samples. A review. J. Liq. Chromatogr. Relat. Technol. 28, 2297-2314 (2005). Review of the use of TLC and HPTLC for the analysis of biological samples of particular interest to biologists, biochemists, hematologists, immunologists, medical diagnosticians, and molecular biologists. Determinations of amino acids, drugs, cabohydrates, lipids, toxins, vitamins, indoles, antibiotics, peptides, pigments, phenols, bile acids, and coumarins in sample matrices such as blood, urine, feces, saliva, cerebrospinal fluids, body tissues, and other biologics are considered. The review discusses the advantage of using modern TLC for biological applications and summarizes important information on stationary and mobile phases and methods used for application of standards and samples, plate development, and zone detection, identification, and quantification.

review 1b

99 001 P. E. WALL: Thin-layer Chromatography. A modern practical approach. The Royal Society of Chemistry, Cambridge 2005, ISBN 0-85404-535-X. This book covers basic theory, concepts and practice of modern thin-layer chromatography. The author summarizes current knowledge obtained by many researchers working on qualitative and quantitative planar chromatography. The manual consists of eight chapters describing each step of typical planar chromatographic process with separate sections devoted to key analytical problems like stationary phase choice and pretreatment, mobile phase optimization, sample preparation and application, plate development and finally spot visualization, detection and quantification. The compact form of this work makes it a really simple, helpful and comprehensive introduction to modern thin-layer chromatography.

comparison of methods, review 1a

2. Fundamentals, theory and general

99 004 V. G. BEREZKIN (A. V. Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, Leninsky pr. 29, 119991 Moscow, GSP-1, Russia; berezkin@ips.ac.ru): Relative retention in TLC r(ij) using column liquid chromatography terms. J. Liq. Chromatogr. & Relat. Technol. 29, 2271-2275 (2006). It is desirable to use the same terms for retention characteristics in CLC

and TLC because the nature of chromatographic processes in column liquid chromatography and thin-layer chromatography is practically the same. The publication suggests a new equation for determination relative retention r(ij) using linear values from the TLC chromatogram.

relative retention 2b

99 005 Monika WAKSMUNDZKA-HAJNOS*, A. HAWRYL, A. PETRUCZYNIK (*Department of Inorganic Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081, Lubin, Poland): Retention of ortho- and para-positional isomers of some model solutes on polar bonded stationary phases in different eluent systems by HPTLC. J. Liq. Chromatogr. Relat. Technol. 28, 907-922 (2005). HPTLC of eight pairs of ortho and para substituted phenols and anilines on amino, diol, and cyano phase, with chamber saturation for 15 min, with nonaqueous eluents consisting of n-heptane and polar modifier (tetrahydrofuran, ethyl acetate, or 2-propanol), or on cyano phase in RP systems with aqueous solutions of methanol or acetonitrile. Evaluation under UV 254 nm.

HPTLC, qualitative identification 2d

3. General techniques

99 006 P. TRIVEDI*, D K. PUNDARIKAKSHUDU (*Department of Pharmaceutical Chemistry, K. B. Institute of Pharmaceutical Education and Research, Sector-23, GH-6, Gandhinagar, 382023, Gujarat, India): Novel TLC Densitometric Method for Quantification Of Solasodine in Various Solanum Species, Market Samples and Formulations. Chromatographia 65 (3-4), 239-243 (2007). Description of a novel TLC densitometric method for the determination of solasodine in various Solanum species (Solanaceae). Solasodine does not show UV absorption therefore TLC of an ion pair complex of solasodine with an acid dye was performed. TLC plates developed by using a solvent with an organic acid ensured in situ color development of the complex. Densitometry at 461 nm. Linearity was 79.2 - 495 ng/zone, with a correlation coefficient of 0.995. The method shows good reproducibility, specificity and accuracy (98.54 ± 2.8%), and eliminates post derivatization steps and the problem of background interference. Validation of the method and application of the method to determine solasodine content in various herb samples, herb extract and their formulations, without matrix interference observed.

pharmaceutical research, herbal, quantitative analysis, HPTLC, densitometry, comparison of methods, qualitative identification 3e

99 059 N. T. BURDZHIEV et al., see section 23e

3d

4. Special techniques

99 007 A. ALPMANN, Gertrud MORLOCK* (*Inst. of Food Chem., Univ. of Hohenheim, Garbenstrasse 28, 70599 Stuttgart; gmorlock@uni-hohenheim.de): Improved online coupling of planar chromatography with electrospray mass spectrometry: extraction of zones from glass plates. Anal. Bioanal. Chem. 386, 1543-1551 (2006). Optimization of a plunger-based extraction device for HPTLC/MS coupling, which was originally designed for extraction on TLC aluminum foils. Some modifications enabled extraction of analytes from HPTLC/TLC glass plates. A buffering of the plunger reduced the occurrence of leakage. The involvement of a torque screwdriver for the fixation resulted in a reproducible contact pressure and avoided breaking the glass plates. Repeatability of the extraction from glass plates, linearity of the signal obtained, and detection capability were shown to be comparable to the original device, which was only usable with aluminum foils. The extraction device was employed for plates from different lots and for plates with different stationary phases thereby proving its general applicability in planar chromatography.

HPTLC, TLC-MS online coupling 4e

99 008 Claudia CIMPOIU (Faculty of Chemistry and Chemical Engineering, “Babes-Bolyai” Universi-

ty, 11 Arany Janos, 400028 Cluj-Napoca, Romania; ccimpoiu@chem.ubbcluj.ro): Qualitative and quantitative analysis by hyphenated (HP)TLC-FTIR technique. J. Liq. Chromatogr. Relat. Technol. 28, 1203-1213 (2005). The (HP)TLC-FTIR coupled method has been widely used for qualitative and quantitative analysis. The potential of this method is demonstrated by its application in various fields of analysis, such as drug analysis, forensic analysis, food analysis, environmental analysis, biological analysis etc. In recent years, much effort has been devoted to the coupling of TLC and HPTLC with spectrometric methods because of the robustness and simplicity of use of (HP)TLC and the need for detection techniques that provide identification and determination of sample constituents. IR as one of the spectroscopic methods that have been coupled with (HP)TLC has a high potential for the elucidation of molecular structures. The review contains introduction, principles, instrumentation and data presentation, qualitative analysis, quantitative analysis, and conclusions.

review 4e

99 009 F. DESTAILLATS, P. A. GOLAY, F. JOFFRE, Maureen DE WISPELAERE, Bernadette HUG, Francesca GIUFFRIDA, Laetitia FAUCONNOT, Fabiola DIONISI (*Nestlé Research Centre, Vers-chez-les-Blanc, P.O.Box 44, CH-1000 Lausanne 26, Switzerland): Comparison of available analytical methods to measure trans-octadecenoic acid isomeric profile and content by gas-liquid chromatography in milk fat. J. Chromatogr. A 1145 (1-2), 222-228 (2007). Pre-fractionation of cis and trans-fatty acids by silver-ion TLC (Ag-TLC) and other methods (silver-ion SPE (Ag-SPE) or HPLC) allows accurate determination of the isomeric profile but is not essential to achieve quantification of total trans-18:1 isomers nor to determine the level of vaccenic (trans-11 18:1) acid in dairy fat. Comparison of different GLC methods suitable to measure the total of trans-18:1 isomers, vaccenic acid and trans-18:1 acid isomeric distribution in milk fat. Pre-separation of cis- and trans-18:1 isomers by Ag-TLC followed by GLC analysis under optimal conditions was selected as the reference method.

quality control, food analysis, comparison of methods 4d

99 010 V. PANCHAGNULA*, A. MIKULSKIS, L. SONG, Y. WANG, M. WANG, Tanya KNUBOVETS, Elaine SCRIVENER, Eva GOLENKO, Ira S. KRULL, M. SCHULZ, H. E. HAUCK, W. F. PATTON (*Biochemistry Department, PerkinElmer Life and Analytical Sciences, Waltham, MA 02451, USA): Phosphopeptide analysis by directly coupling two-dimensional planar electrochromatography/thin-layer chromatography with matrix-assisted laser desorption/ionization time-of-flight mass spectrometry J. Chromatogr. A 1155 (1), 112 - 123 (2007). Presentation of a novel strategy for the fractionation of complex peptide mixtures using two-dimensional planar electrochromatography/thin-layer chromatography. Migration of phosphopeptides is slower in the first dimension, based upon their anionic phosphate residues, and certain predominantly acidic phosphopeptides even migrate in the opposite direction, relative to the bulk of the peptides. Further distinguishability of phosphopeptides are based upon hydrophilicity in the second dimension, which permits a restricted region of the plate to be directly examined for the presence of phosphopeptides by mass spectrometry. Phosphopeptide analysis from the plates by matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF)-MS and tandem MS enabled peptide sequencing and identification.

qualitative identification HPTLC, TLC MS online coupling 4e

5. Hydrocarbons and halogen derivatives

99 011 A. PIENIAK, M. SAJEWICZ, Teresa KOWALSKA*, K. KACZMARSKI, K. TYRPIEN (*Institute of Chemistry, Silesian University, 9 Skolna Street, 40-006 Katowice, Poland; kowalska@us.edu.pl): The impact of mobile phase pressure and velocity on the development of chromatograms in TLC and OPLC - a comparison. J. Liq. Chromatogr. Relat. Technol. 28, 2479-2488 (2005). TLC of tetralin, anthracene, and phenanthrene on silica gel with n-hexane with chamber saturation for 20 min. Densitometry in reflectance mode at 254 nm. In the experiments the separation

performance of TLC proved to be substantially better than that of OPLC.

comparison of methods 5b

7. Phenols

99 012 Claudia CIMPOIU (Faculty of Chemistry and Chemical Engineering, "Babes-Bolyai" University, 11 Arany Janos, 400028, Cluj-Napoca, Romania; ccimpoiu@chem.ubbcluj.ro): Analysis of some natural antioxidants by Thin-Layer Chromatography and High Performance Thin-Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 29, 1125-1142 (2006). (HP)TLC of polyphenols (hydroxybenzoic acids, hydroxycinnamic acids, flavonols, flavones, flavanones); e. g. HPTLC of flavonoids on silica gel with tetrahydrofuran - toluene - formic acid - water 16:8:2:1 in a saturated twin-trough chamber; detection by dipping the warm plate into natural products reagent followed by dipping into PEG 400 solution. Evaluation under UV 254 and 366 nm; densitometry at 254 nm.

pharmaceutical research, qualitative identification, quantitative analysis densitometry 7

99 013 Alina PYKA*, K. BOBER, W. KLIMCZOK, M. STEFANIAK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, PL-41-200, Sosnowiec, Poland; alinapyka@wp.pl): Densitometric investigations of chemical durability of pyrocatechol. J. Liq. Chromatogr. & Relat. Technol. 29, 2997-3007 (2006). TLC of pyrocatechol on silica gel (prewashed with methanol - chloroform 1:1), with methanol - chloroform 1:9. Densitometric measurement at 200 nm. Investigation of stability on silica gel, as well as in solutions, in relation to different storage conditions.

pharmaceutical research, quality control, quantitative analysis, densitometry 7

8. Substances containing heterocyclic oxygen

99 014 M. A. HAWRYL, Monika WAKSMUNDZKA-HAJNOS*, T. INGLOT (*Department of Inorganic Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081, Lubin, Poland): Retention behavior of same phenolic compounds in two-dimensional Thin Layer Chromatography systems using a diol bonded polar stationary phase. J. Liq. Chromatogr. Relat. Technol. 28, 2245-2259 (2005). HPTLC of flavonoids and phenolic acid (astragalin, quercitrin, kaempferol 3-glyco-7-rhamnoside, naringenin 7-glucoside, ferulic acid, chlorogenic acid, elagic acid, caffeic acid, p-, m-, o-coumaric acid, gallic acid, apigenin, naringenin, acacetin, flavone, morine, hesperetin, quercetin, narcisin, kaempferol 3,7-dirhamnosid, naringenin, and rutin) on diol phase (prewashed with methanol) with dichloromethane - 2-propanol 1:9, methanol - diisopropylether 1:4, and methanol - ethyl acetate 1:9. Also 2 D-TLC with dichloromethane - 2-propanol 1:4 and methanol - water 1:4 among numerous other mobile phases. Detection under UV light at 365 nm.

herbal, qualitative identification 8a

99 015 E. REICH*, Anne SCHIBLI, Valeria WIDMER, Ruth JORNS, Evelyne WOLFRAM, Alison DEBATT (*CAMAG Laboratory, Sonnenmattstr. 11, Muttenz CH-4132, Switzerland; eike.reich@camag.com): HPTLC methods for identification of green tea and green tea extract. J. Liq. Chromatogr. & Relat. Technol. 29, 2141-2151 (2006). HPTLC of flavonoids (with rutin, chlorogenic acid, hyperoside, and gallic acid as reference substances) on silica gel in a twin-trough chamber with ethyl formate - toluene - formic acid - water 60:3:8:6. Detection by dipping the hot plate (heated at 100°C for 2 min) into natural products reagent, followed by drying, dipping into polyethylene glycol 400 (10 g in 200 mL dichloromethane), and drying. Evaluation under UV 366 nm. With this method the geographical origin of the material can be determined. Toluene - acetone - formic acid 9:9:2 allows the discrimination of green from black and other speciality teas, based on the polyphenol pattern. Detection by dipping the hot plate (heated at 100°C for 2

min) into a solution of Fast Blue salt B, followed by detection under white light. For investigation of the alkaloid profil ethyl acetate - methanol - water 20:2.7:2, followed by detection under UV 254 nm is used. The amino acid profile is analyzed by using 1-butanol - acetone - acetic acid - water 7:7:2:4. Detection by dipping into ninhydrin reagent, followed by heating at 110°C for 3 min and evaluation under white light. The method for polyphenols was validated regarding specificity, stability, reproducibility, and robustness.

quality control, qualitative identification, HPTLC 8a, 32e

11. Organic acids and lipids

99 016 Katarzyna BOBER*, M. GARUS (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, PL-41-200 Sosnowiec, Poland; katarzynabober@wp.pl): RP-HPTLC application in the investigation of solubility in water of long-chain fatty acids. J. Liq. Chromatogr. & Relat. Technol. 29, 2787-2794 (2006). HPTLC of acids (from octanoic to octadecanoic) on RP-18 with methanol - water 9:1 and ethanol - water 19:1. Detection with iodine vapor. The characterisation by high values of determination coefficients suggest the possibility of using them to calculate and predict the values of solubilities in water of acids investigated.

HPTLC, qualitative identification 11a

99 017 D. L. MARTIN, J. SHERMA, B. FRIED* (*Department of Biology, Lafayette College, Easton, PA, 18042, USA; friedb@lafayette.edu): High Performance Thin Layer chromatographic analysis of neutral lipids and phospholipids in the medicinal leech Hirudo medicinalis and in leech conditioned water. J. Liq. Chromatogr. Relat. Technol. 28, 2597-2606 (2005). HPTLC of free sterol, and free fatty acids (cholesterol, triacylglycerol and methyl esters) on silica gel (prewashed with dichloromethane - methanol 1:1) with petroleum ether - diethyl ether - glacial acetic acid 80:20:1 in a twin-trough chamber saturated for 15 min. Determination of steryl esters with n-hexane - petroleum ether - diethyl ether - glacial acetic acid 50:25:5:1. Detection by spraying with 5 % ethanolic phosphomolybdic acid solution and heating for 10 min at 115 °C. Determination of polar lipids (cholesterol, phosphatidyl ethanolamine, phosphatidylcholine, lysophosphatidylcholine) with chloroform - methanol - water 65:25:4. Detection by spraying with a 10 % cupric sulfate solution and heating at 140 °C for 10 min. Quantitation by densitometry at 610 nm (for neutral lipids) and 370 nm (for polar lipids).

HPTLC, quantitative analysis 11c

99 018 W. M. INDRASENA*, C. J. BARROW, J. A. KRALOVEC (*Ocean Nutrition Canada Ltd., 101, Research Drive, Dartmouth, Nova Scotia, Canada, B2Y 4T6; windrasena@ocean-nutrition.com).: Effect of hydrogen/air flow rates and scan rate on the flame ionization detection response of phospholipids, and their qualitative and quantitative analysis by Iatroscan (Mark-6s) TLC-FID. J. Liq. Chromatogr. & Relat. Technol. 29, 2111-2127 (2006). TLC of phospholipids on silica gel with chloroform - methanol - water - formic acid 65:35:2:0.04. Detection by spraying with 2',7'- dichlorofluorescein and under UV light. Phosphatidylserine and lysophosphatidylserine were detected by spraying with ethanolic ninhydrin reagent. TLC as screening method prior to the application to TLC-FID by the Iatroscan Chromarod system.

qualitative identification 11c

99 019 M. M. WHITE, B. FRIED*, J. SHERMA (*Department of Biology, Lafayette College, Easton, PA 18042, USA; friedb@lafayette.edu): Determination of the effects of estivation and starvation on neutral lipids and phospholipids in Biomphalaria glabrata (NMRI strain) and Helisoma trivolvis (Colorado strain) snails by quantitative High Performance Thin-Layer Chromatography-densitometry. J. Liq. Chromatogr. & Relat. Technol. 29, 2167-2180 (2006). HPTLC of neutral lipids on prewashed silica gel with petroleum ether - diethyl ether - glacial acetic acid 80:20:1; and of

methyl and steryl esters with hexane - petroleum ether (35-60°C) - diethyl ether - glacial acetic acid 50:25:5:1 in a twin trough chamber saturated for 20 min. Detection by spraying with a 50 % solution of phosphomolybdic acid in ethanol, followed by heating at 115°C for 10 min. Polar lipids were separated with chloroform - methanol - water 65:25:4. Detection by spraying with 10 % cupric sulfate solution, followed by heating at 140 °C for 10 min. Quantitative determination by absorbance measurement at 610 nm for neutral lipids and at 370 nm for polar lipids.

densitometry, quantitative analysis, biological research 11c

99 020 M. MALONEY*, S. BISHOP, G. TORRENCE, M. DELEON (*Department of Biology, Spelman College, Atlanta, GA 30314, USA; mmaloney@spelman.edu): Comparison of total lipid composition in Gb3-positive and Gb3-deficient Burkitt's lymphoma cells. J. Liq. Chromatogr. Relat. Technol. 28, 2571-2580 (2005). TLC of lipids, triacylglycerol, diacylglycerol, and sphingosine on silica gel with hexane - diethyl ether - formic acid 40:10:1 for neutral lipids, and with chloroform - methanol - water 65:25:4 for glycolipids. Phospholipids were separated by two dimensional development with chloroform - methanol - 28 % ammonium hydroxide 65:25:4 in the first direction, followed by drying and development with chloroform - acetone - methanol - acetic acid - water 30:40:10:10:1 in the second direction. Detection of neutral lipids by spraying with charring or phosphomolybdic acid reagent; detection of glycolipids by spraying with orcinol reagent (orcinol in 70 % sulfuric acid), and of phospholipids by spraying with molybdenum blue reagent for phosphate groups or ninhydrin reagent for phospholipids containing free amino groups.

clinical chemistry research, qualitative identification 11c

99 021 S. MOMCHILOVA, D. ANTONOVA, I. MAREKOV, L. KULEVA, Boryana NIKOLOVA-DAMYANOVA* , G. JHAM (*Bulgarian Academy of Sciences, Institute of Organic Chemistry with Centre of Phytochemistry, 1113 Sofia , Bulgaria; bmd@orgchem.bas.bg): Fatty acids, triacylglycerols, and sterols in neem oil (Azadirachta Indica A. Juss) as determined by a combination of chromatographic and spectral techniques. J. Liq. Chromatogr. & Relat. Technol. 30,11-25 (2007). TLC on silica gel with petroleum ether - acetone 25:2; detection by spraying with 50 % ethanolic sulfuric acid and heating at 200°C. Isolation, purification and quantification of lipid classes by preparative TLC; detection by spraying the edges with 2',7'-dichlorofluorescein for visualization under UV. Preparative separation of acylglycerols, free fatty acids, and polar lipids on silica gel with petroleum ether - acetone - acetic acid 70:30:1. Quantitative Ag-TLC on silica gel impregnated by dipping into a 0.5 % methanolic silver nitrate solution - also preparative Ag-TLC. Quantitative TLC on RP by densitometry at 450 nm. Ag-TLC provided the quantitative data for the TAG classes differing in unsaturation, and RP-TLC for the TAG species differing in chain length within a given class.

agricultural, preparative TLC, qualitative identification, quantitative analysis 11c

99 022 S. R. BANDSTRA, K. E. MURRAY, B. FRIED, J. SHERMA* (*Department of Chemistry, Lafayette College, Easton, PA 18042, USA; shermaj@lafayette.edu): High Performance Thin Layer Chromatographic analysis of neutral lipids in the feces of BALB/c mice infected with Echinostoma caproni. J. Liq. Chromatogr. & Relat. Technol. 30, 1437-1445 (2007). HPTLC of steryl esters, methyl esters, triacylglycerols, FFA, and free sterols on silica gel (prewashed by development with dichloromethane -methanol 1:1) with petroleum ether - diethyl ether - glacial acetic acid 80:20:1 in a saturated twin-trough chamber. Detection by spraying with 5 % ethanolic phosphomolybdic acid followed by heating at 150 °C for 110 min. Quantitative determination by absorbance measurement at 610 nm.

HPTLC, quantitative analysis, biological research 11c

99 023 F. RONG (Rong Fei), X. FENG (Feng Xiaogang), C. YUAN (Yuan Chunwei) D. FU (Fu Degang)*, P. LI (Li Ping) (*State Key Laboratory of Bioelectronics, Dept. of Biological Science

and Medical Engineering, Southeast University, Sipailou No. 2, Nanjing 210096, P. R. China; fudegang@seu.edu.cn): Chiral separation of mandelic acid and its derivatives by Thin-Layer Chromatography using molecularly imprinted stationary phases. J. Liq. Chromatogr. & Relat. Technol. 29, 2593-2602 (2006). TLC of mandelic acid and derivatives on molecularly imprinted polymers of L-mandelic acid, L-2-chloromandelic acid and L-4-chloromandelic acid as chiral stationary phases using mixtures of acetonitrile and acetic acid in different concentrations (1, 5, 10 %) with chamber saturation. Detection under UV 254 nm.

pharmaceutical research, qualitative identification 11a

13. Steroids

99 024 M. DOLOWY (Faculty of Pharmacy, Department of Analytical Chemistry, PL-41-200 Sosnowiec, 14 Jagiellonska Street, Poland; mdolowy@wp.pl): Separation of selected bile acids by TLC. IX. Separation on silica gel 60 and on silica gel 60 F254 aluminium plates impregnated with Cu(II), Ni(II), Fe(II), and Mn(II) cations. J. Liq. Chromatogr. & Relat. Technol. 30, 405-418 (2007). TLC of cholic acid, glycocholic acid, glycolithocholic acid, deoxycholic acid, chenodeoxycholic acid, glycodeoxycholic acid, and lithocholic acid on silica gel impregnated with 1 %, 2.5 %, and 5 % aqueous solutions of copper(II) sulfate, manganese sulfate, nickel sulfate, and iron(II) sulfate, using mixtures of n-hexane - ethyl acetate - acetic acid 22:20:5; 25:20:2; and 22:22:5. The use of the mobile phase 25:20:2 on silica gel impregnated with a 5 % solution of copper(II) sulfate allowed separation of all neighbouring pairs of investigated bile acids, compared to non impregnated plates. Detection of bile acids by spraying with 10 % aqueous sulfuric acid reagent, followed by heating at 120 °C for 10 min.

qualitative identification, biological research 13d

99 025 J. JARUSIEWICZ, J. SHERMA*, B. FRIED (*Department of Chemistry, Lafayette College, Easton, PA, 18042, USA; shermaj@lafayette.edu).: Separation of sterols by reversed phase and argentation Thin Layer Chromatography. Their identification in snail bodies. J. Liq. Chromatogr. Relat. Technol. 28, 2607-2617 (2005). HPTLC and TLC of sterols (cholesterol, cholestanol, beta-sitosterol, stigmasterol, ergosterol, campesterol, desmosterol, and brassicasterol) on RP-18, RP-18 W, RP-2, RP-8, amino, cyano, diol, and phenyl bonded phase, hydrocarbon impregnated layers, and silica gel impregnated with 10 % silver nitrate, with 25 mobile phases. Optimal separation of sterols was achieved on RP-18 with acetonitrile - chloroform 8:7, or petroleum ether - acetonitrile - methanol 1:2:2. Detection by spraying with ethanolic phosphomolybdic acid and heating at 115 °C for 10 min.

13c

99 026 H. KALÁSZ, E. LIKTOR-BUSA, G. JANICSÁK, Mária BÁTHORI* (*Department of Pharmacognosy, University of Szeged, Eotovs utca 6, H-6720 Szeged, Hungary; bathori@pharm.u-szeged.hu): Role of preparative rotation planar chromatography in the isolation of ecdysteroids. J. Liq. Chromatogr. & Relat. Technol. 29, 2095-2109 (2006). Analytical and preparative TLC of ecdysteroids (e. g. dacryhainansterone, 20-hydroxyecdysone) on silica gel in unsaturated glass chambers with dichloromethane - methanol - benzene 25:5:3 and ethyl acetate - 96% ethanol - water 16:2:1. Detection under UV 254 nm or by spraying with vanillin-sulfuric acid reagent, followed by detection under white light and under UV 366 nm. Densitometric absorbance measurement at 254 nm.

herbal, qualitative identification, preparative TLC 13e

99 027 Alina PYKA*, M. BABUSKA (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska St., PL-41-200 Sosnowiec, Poland; alinapyka@wp.pl).: Lipophilicity of selected steroid compounds. I. Investigations on RP-18 W stationary phase

by RP-HPTLC. J. Liq. Chromatogr. Relat. Technol. 29, 1891-1903 (2006). HPTLC of androsterone, epi-androsterone, dehydro-epi-androsterone, testosterone, stigmasterol, beta-sitosterol, estradiol, hydrocortisone, and cholesterol on RP-18 W with methanol - water, and acetonitrile - water in different composition, with chamber saturation. Detection by spraying with sulfuric acid - methanol 1:9 and heating at 120 °C for 15 min. Densitometric determination of RF values. The aim of the work was to compare the lipophilicity of selected steroids determined by RP-HPTLC on RP-18 W plates using different mobile phases with lipophilicity values estimated by computational methods.

pharmaceutical research, qualitative identification HPTLC densitometry 13a

99 028 Alina PYKA*, M. BABUSKA, K. BOBER, D. GURAK, W. KLIMCZOK, M. MISZCZYK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska St., PL-41-200 Sosnowiec, Poland; alinapyka@wp.pl): Influence of temperature of silica gel activation on separation of selected biologically active steroid compounds. J. Liq. Chromatogr. Relat. Technol. 29, 2035-2044 (2006). TLC of androsterone, epi-androsterone, dehydro-epi-androsterone, testosterone, stigmasterol, beta-sitosterol, estradiol, hydrocortisone, and cholesterol on silica gel with chloroform - acetone 17:3 and activation at 100 °C, 120 °C, 150 °C, and 200 °C during 15, 30, 60, and 120 min. Activation time temperature influenced Rf values and order of separated compunds.

qualitative identification 13a

99 029 Alina PYKA*, M. DOLOWY (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, Jagiellonska 4, 41-200, Sosnowiec, Poland; alinapyka@wp.pl): Lipophilicity of selected bile acids as determined by TLC. II. Investigations on RP-18 W stationary phases. J. Liq. Chromatogr. Relat. Technol. 28, 297-311 (2005). TLC of cholic acid, glycocholic acid, glycodeoxycholic acid, chenodeoxycholic acid, deoxycholic acid, lithocholic acid, and glycolithocholic acid on RP-18 W with methanol - water, (acetonitrile - methanol 1:1) - water, acetone - water, dioxane - water in different volume compositions. Detection by spraying with 10 % aqueous solution of sulfuric acid or by dipping in 10 % ethanolic solution of phosphomolybdic acid followed by heating at 120 °C for 20 min.

qualitative identification 13d

99 033 Alina PYKA*, M. DOLOWY, D. GURAK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska St., PL-41-200 Sosnowiec, Poland; alinapyka@wp.pl): Separation of selected bile acids by TLC. V. Influence of temperature on the separation. J. Liq. Chromatogr. Relat. Technol. 28, 631-640 (2005). TLC of cholic acid, glycocholic acid, glycolithocholic acid, deoxycholic acid, chenodeoxycholic acid, glycodeoxycholic acid, and lithocholic acid on RP-2 and silica gel - Kieselguhr at 18 °C and 40 °C with n-hexane - ethyl acetate - acetic acid in different volume compositions. Detection by spraying with 10 % sulfuric acid in water, followed by heating at 120 °C for 20 min. The obtained results indicate that the separation of some bile acids can be improved by proper choice of temperature.

qualitative identification 13d

14. Steroid glycosides, saponins and other terpenoid glycosides

99 036 P. D. TRIVEDI, K. PUNDARIKASHUDU*, S. RATHNAM, K. S. SHAH (* L. J. Institute of Pharmacy, Near Nagdev Kalyan Mandir, Sanand Cross Roads, Ahmedabad 382210 Gujarat, India; kil_pundarik@yahoo.co.in).: A validated quantitative thin-layer chromatographic method for estimation of diosgenin in various plant samples, extract, and market formulation. J. Assoc. Off. Anal. Chem. 90, 358-363 (2007). TLC of diosgenin on silica gel with n-hexane - ethyl acetate 4:1 with chamber saturation for 15 min. Detection by dipping into a modified anisaldehyde-sulfuric acid reagent (0.1 % anisaldehyde in 2 % sulfuric acid) in order to reduce charring and back-

ground interference, followed by drying for 10 min under hot air and heating at 105 °C for 10 min. Quantitative determination by absorbance measurement at 428 nm.

herbal, quality control, quantitative analysis, densitometry 14

99 037 Q. DU (Du Qizhen)*, S. GAO (Gao Shijun) (*Institute of Food and Biological Engineering, Zhejiang Gongshang University, Hangzhou 310035, China; qizhendu@163.com): Preparative separation of saponins from the Luffa Cylindeica (L.) Roem. by slow rotary countercurrent chromatography. J. Liq. Chromatogr. & Relat. Technol. 29, 2451-2456 (2006). TLC of saponins (lucyoside Q and lucyoside H) on silica gel with chloroform - methanol - water 7:3:1. Detection by spraying with 5 % sulfuric acid in ethanol followed by heating at 110 °C.

herbal, qualitative identification 14

99 038 Q. DU (Du Qizhen)*, J. YUAN (Yuan Jie) (*Institut of Food and Biological Engineering, Zhejiang Gongshang University, 149 Jiaogong Road, Hangzhou 310035, P.R. China; qizhendu@mail.hzic.edu.cn): Preparation of diterpene saponins from the fruit of Momordica Charantia L. by high speed countercurrent chromatography (HSCCC). J. Liq. Chromatogr. Relat. Technol. 28, 1717-1724 (2005). TLC of triterpene saponins (goyaglycoside-e, momordicoside L and momordicoside K, and goyaglycoside-a) on silica gel with methyl tert-butyl ether, n-butanol, methanol, water 1:2:1:5 and 1:3:1:5; and chloroform - methanol - water 15:4:1. Detection by spraying with 5 % ethanolic sulfuric acid followed by heating at 110 °C.

food analysis, qualitative identification 14

99 039 N. K. SATTI, K. A. SURI*, P. DUTT, O. P. SURI, M. AMINA, G. N. QAZI, A. RAUF (*Regional Research Laboratory (CSIR), Canal Road, Jammu Tawi 180001, India; kasuril@rediffmail.com): Evaluation of Asparagus racemosus on the basis of immunomodulating sarsasapogenin glycosides by HPTLC. J. Liq. Chromatogr. Relat. Technol. 29, 219-227 (2006). HPTLC of selected sarsasapogenins (shataverin-IV and immunoside) on silica gel with ethyl acetate - methanol - water 75:13:5:10. Detection by spraying with ceric ammonium sulfate followed by heating at 100 °C for 5 min. Quantitation by scanning at 450 nm.

herbal, HPTLC, quantitative analysis 14

99 040 M. M. EL-SAYED, E. S. ABDEL-HAMEED*, H. A. EL-NAHAS, E. A. EL-WAKIL (*Laboratory of Medicinal Chemistry, Theodor Bilharz Research Institute, Warrak El-Hadar, Giza, Egypt, B. O. Box 30 Imbaba; sayed_sa@hotmail.com): Isolation and identification of some steroidal glycosides of Furcraea selloa. Pharmazie 61, 478-482 (2006). Analytical and preparative TLC of two steroidal glycosides 6-O-beta-D-glucopyranosyl-(1-4)-beta-D-glucopyronoside chlorogenin and 3-O-beta-D-glucopyranosyl-(1-4)-beta-D-glucopyranoside crestagenin on silica gel with chloroform - methanol - water 30:10:1. Detection by spraying with 40 % sulfuric acid followed by heating at 120 °C.

herbal, preparative TLC qualitative identification 14

15. Terpenes and other volatile plant ingredients

99 041 O. B. ABDEL HALIM, G. T. MAATOOQ, A. M. MARZOUK * (*Department of Pharmacognosy, Faculty of Pharmacy, Mansoura University, Mansoura 35516, Egypt; amarzouk2003@yahoo.co.uk): Metabolism of parthenin by Beauveria bassiana ATCC 7159. Pharmazie 62, 226-230 (2007). TLC of two metabolites of the sesquiterpene lactone parthenin on RP-18 and silica gel with dichloromethane - methanol 19:1. Detection by spraying with vanillin-sulfuric acid followed by heating. Also TLC method for detection of hydroperoxy radical: the developed chromatogram was air dried, sprayed with a freshly prepared spray reagent (50 mL of 1 % ethanolic solution of ferrous ammonium sulfate were mixed with 5 mL of 1 M sulfuric acid, and added to 5 mL etha-

nolic solution of ammonium thiocyanate). A dark red coloured zone was considered as a positive result. Ascaridol and hydrogen peroxide were used as positive controls.

pharmaceutical research, qualitative identification 15a

99 042 M. J. MAO (Mao Man-Jun)*, B. JIANG (Jiang Biao), Z. J. JIA (Jia Zhong-Jian) (*Department of Chemistry, Tongji University, 1239 Siping Road, Shanghai 200092, P. R. China; maomanj@yahoo.com.cn): Six new sesquiterpenes from Cacalia ainsliaeflora. Pharmazie 60, 313-316 (2005). Preparative TLC of 3beta-angeloyloxy-8-oxo-eremophil-6(7)-en-12-oic acid and 3beta-angeloyloxy-10ss-hydroxy-8-oxo-eremophil-6(7)-en on silica gel with petroleum ether - acetone 4:1 and petroleum ether - toluene - acetone 1:3:1. Detection under UV light.

pharmaceutical research, herbal, preparative TLC 15a

99 043A. M. EL-SHAMY, S. S. EL-HAWARY, M. E. M. RATEB* (*Cairo University, Faculty of Pharmacy, Pharmacognosy Department, Kasr El-Aini St, 11562, Cairo, Egypt, mostafa19772002@yahoo.com): Quantitative estimation of parthenolide in Tanacetum parthenium (L.) Schultz-Bip. cultivated in Egypt. J. Assoc. Off. Anal. Chem. 90, 21-27 (2007). TLC of parthenolide on silica gel with chloroform - ethyl acetate 4:1. Detection by spraying with anisaldehyde-sulfuric acid reagent, followed by heating at 100 °C for 5 min. Quantitative absorbance measurement at 565 nm.

herbal, qualitative identification 15a

99 044 A. M. YANG (Yang Al-Mei), X. LIU (Liu Xia), R. H. LU (Lu Run-Hua), Y. P. SHI (Shi Yan-Ping)* (*Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, P. R. China; shiyp@lzb.ac.cn): Triterpenoids from Pyrethrum tatsienense. Pharmazie 61, 70-73 (2006). TLC of olean-12-en-3beta,11a,16beta-triol-3-O-palmitate, ursa-12-en-3beta,11beta,16beta-triol-3-O-palmitate, olean-12-en3beta,16beta-diol-3-O-palmitate, beta-amyrin, alpha-amyrin, methylursolate, taraxasterol, taraxast-20(30)-ene-3beta,16beta-diol-3-O-palmitate, pseudotaraxasterol, lup-3beta-O-palmitate on silica gel. Detection UV light or by spraying with 98 % sulfuric acid - ethanol 1:19 followed by heating at 110 °C.

traditional medicine, herbal, qualitative identification 15a

99 045 Alina PYKA*, D. GURAK, K. BOBER, W. KLIMCZOK, G. JANIKOWSKA, A. STOLARCZYK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska St., PL-41-200 Sosnowiec, Poland; alinapyka@wp.pl).: Influence of the mobile and stationary phases on the separation of selected essential oil components and aroma substances investigated by TLC. J. Liq. Chromatogr. Relat. Technol. 28, 2525-2537 (2005). TLC of 15 selected essential oil components, alcohols (geraniol, linalool, menthol, (+)-borneol), phenols (vanillin, eugenol, guaiacol, thymol), ether ketones, and aldehydes (cineole, trans-anethole, (R)-(-)-carvone, (R)-(-)-fenchone, coumarin, camphor, cinnamic aldehyde) on silica gel and alumina with 26 mobile phases after saturation for 30 min. Aluminium oxide plates and carbon tetrachloride - acetone 49:1 were best suited for the separation of the investigated alcohols; aluminium oxide plates and carbon tetrachloride - acetone 17:3 for the separation of the investigated phenols, and alumina with chlorobenzene - acetone (19:1 for the separation of coumarin, cineole, carvone, cinnamic aldehyde, campher, fenchone, and trans-anethole. Detection by spraying with a 5 % solution of potassium dichromate in 40 % sulfuric acid.

cosmetics, qualitative identification 15b

17. Amines, amides and related nitrogen compounds

99 046 J. BIALECKI, L. YUAN (Yuan Li-Hua), B. GONG (Gong Bing)* (*Department of Chemistry, University at Buffalo, State University of New York, NY, USA, bgong@buffalo.edu): A branched, hydrogen-bonded heterodimer: a novel system for achieving high stability and specifici-

ty. Tetrahedron 63, 5460-5469 (2007). Preparative TLC of branched oligoamides derived from methyl salicilate on silica gel with dichloromethane - acetone 3:1. Detection under UV 254 nm. Dimerization increases the hRf value from 12 to 46.

pharmaceutical research, preparative TLC 17c

99 058 Ute JAUTZ et al., see section 23e

99 047 Dorota KAZMIERCZAK*, W. CIESIELSKI, R. ZAKRZEWSKI (*Department of Instrumental Analysis, University of Lódz, Pomorska 163, Lódz 90-236, Poland; dorotakazmier@uni.lodz.pl): Application of the iodine-azide procedure for detection of biogenic amines in TLC. J. Liq. Chromatogr. & Relat. Technol. 29, 2425-2436 (2006). HPTLC and TLC of phenyl isothiocyanate derivatives of biogenic amines (2-phenylethylamine, tyramine, octopamine, dopamine, adrenaline, histamine, tryptamine, putrescine, spermidine, spermine, and calamine) on silica gel and RP-18 in a horizontal chamber saturated for 30 min with hexane - dioxane 1:1; 1:2; and 2:3; hexane - ethanol 1:1, methanol - dioxane 2:1, and hexane - dioxane - toluene 1:1:1. Prechromatographic derivatization with PITC directly on the plate. Detection of NP-TLC plates (silica gel) by spraying with a mixture of sodium azide and starch solution. For RP-TLC sodium azide solution with starch was incorporated into the mobile phase, and then the plates were exposed to iodine vapor. The stability of the resulting white zones on a violet-grey background lastet for several minutes. The results of the detection limits proved to be advantageous to other commonly used detection techniques (UV and iodine chamber).

qualitative identification, HPTLC, biological research 17a

18. Amino acids and peptides, chemical structure of proteins

99 048 W. F. PATTON*, V. PANCHAGNULA, E. ROCKNEY, I. S. KRULL (*PerkinElmer Life and Analytical Sciences, Life Sciences Division, 549 Albany Street, Building 100-3, Boston, Massachusetts 02118, USA; wayne.patton@perkinelmer.com): Taking a walk on the wild side with planar electrochromatography and thin-layer electrophoresis: of peptides, proteins, and proteomics. J. Liq. Chromatogr. Relat. Technol. 29, 1177-1218 (2006). Examination of planar electrochromatography (PEC) and thin-layer electrophoresis (TLE) for their potential application to peptide and protein analysis, employing one-dimensional and two-dimensional separations, which could potentially be used for proteomics applications. TLC of BSA tryptic peptide digest on cellulose with 1) 2-butanol - 25 % ammonia - pyridine - water 39:10:2:269, and 2) 2-butanol - acetic acid - pyridine - water 15:3:10:12. Detection by spraying with ninhydrin.

review, biological research 18

99 049 E. GERE-PÁSZTI, T. CSERHÁTI*, E. FORGÀCS, Z. DEYL, I. MIKSIK, A. ECKHARDT, Z. ILLÈS (*Research Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, P. O. Box 17, 1525, Budapest, Hungary; tevi@chemres.hu): Interaction of hydroxypropyl-ss-cyclodextrin with peptides, studied by reversed-phase Thin-Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 28, 2619-2632 (2005). TLC of homopeptides of alanine, glycine, lysine, and phenylalanine on alumina (impregnated by overnight development in n-hexane - paraffin oil 19:1) with water and 0.05 M aqueous solution of LiCl, NaCl, RbCl, and CsCl, containing different amounts of HP-beta-CD. Development in sandwich chambers, followed by drying at 100 °C. Detection by spraying with ninhydrin reagent (0.3 g ninhydrin in 100 mL n-butanol containing 3 mL acetic acid). In order to increase the sensitivity of detection, the plates were sprayed with 2 M aqueous acetic acid prior to the ninhydrin reaction.

pharmaceutical research, qualitative identification 18

99 050 D. KAZMIERCZAK, W. CIESIELSKI, R. ZAKRZEWSKI* (*Department of Instrumental Ana-

lysis, University of Lódz, Pomorska 163, 90-236 Lódz, Poland; robzak@chemul.uni.lodz.pl): Detection and separation of amino acids as butylthiocarbamyl derivatives by Thin-Layer Chromatography with the iodine-azide detection system. J. Liq. Chromatogr. Relat. Technol. 28, 2261-2271 (2005). HPTLC of 21 butylthiocarbamyl-amino acids on silica gel in a saturated horizontal chamber. Pre-chromatographic derivatization with 2-propanol - BITC - triethylamine was carried out after application of the amino acids. For the reaction the plate was placed in a glass chamber in a thermostat at 40 °C for 30 min. Then the plate was developed with ethanol - methanol - chloroform 1:1:2. Detection by spraying with a freshly prepared mixture of 3 % sodium azide and 0.5 % starch solution adjusted to pH 5.5 and exposed to iodine vapor for 5 s. Quantities in the range of 2-90 pmol per spot were detected.

qualitative identification 18a

22. Alkaloids

99 051 A. H. MERICLI*, S. SUEZGEC, L. BITIS, F. MERICLI, H. OEZCELIK, J. ZAPP, H. BECKER (*Istanbul University, Faculty of Pharmacy, Department of Pharmacognosy, 34116 Beyazit, Istanbul, Turkey; alimer@istanbul.edu.tr): Diterpenoid alkaloids from the roots of Aconitum cochleare. Pharmazie 61, 483-485 (2006). Preparative and analytical TLC of talatisamine, 14-O-acetyltalatisamine, senbusine C, and condelphine on silica gel with toluene - ethyl acetate - diethylamine 9:2:1, and 7:2:1; and chloroform - methanol - ammonia 5:3:1, detection under UV light.

herbal, qualitative identification, preparative TLC 22

99 052 E. HERNÁNDEZ-DOMÍNGUEZ, F. VÁZQUEZ-FLOTA* (*Unidad de Bioquímica y Biología Molecular de Plantas and Graduate Program in Plant Sciences and Biotechnology, Centro de Investigación Científica de Yucatán, Calle 43 # 130, Chuburna, 97200, Mérida Yucatán, México; felipe@cicy.mx): Monoterpenoid alkaloid quantitation by in situ densitometry-Thin Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 29, 583-590 (2006). TLC of ajmalicine, catharanthine, and vindoline on silica gel with chamber saturation using 12 different mobile phases. Detection under UV 254 nm. For confirmation the spots were individually eluted und subjected to two dimensional TLC; quantitation by in situ densitometry at 280 nm.

herbal, quantitative analysis, densitometry 22

99 053 C. O. OKUNJI, M. M. IWU, Y. ITO*, P. L. SMITH (*Center of Biochemistry and Biophysics, National Heart, Lung, and Blood Institute, National Institutes of Health, Bldg 50, Rm. 3334, Bethesda, MD 20892-8014, USA; itoy@nhlbi-nih.gov).: Preparative separation of indole alkaloids from the rind of Picralima nitida (Stapf) T. Durand & H. Durand by pH-zone-refining countercurrent chromatography. J. Liq. Chromatogr. Relat. Technol. 28, 775-783 (2005). TLC of Picralima alkaloids (akuammigine, picraline, akuammicine, picranitidine, akuammine, akuammiline, akuammidine, akuammigine) on silica gel with benzene - ethyl acetate - methanol - isopropanol - 25 % ammonia 12:6:3:3:1; toluene - ethyl acetate - diethylamine, saturated with 25 % ammonia 7:2:1, and toluene - ethyl acetate - diethylamine 7:2:1. Detection under UV light or by spraying with Dragendorff reagent.

herbal, pharmaceutical research, qualitative identification 22

99 054 Á. SÁRKOEZI*, G. JANICSÁK, L. KURSINSZKI, Á. KÉRY (*Department of Pharmacognosy, Semmelweis University, Ülloi Str. 26, 1085 Budapest, Hungary): Alkaloid Composition of Chelidonium majus L. Studied by Different Chromatographic Techniques. Chromatographia 63 (Supplement 13), S81 - S86 (2006). TLC of isoquinoline alkaloids (chelidonine, chelerythrine, sanguinarine, coptisine and berberine) in Chelidonium plant organs on silica gel with chloroform - methanol 2:1, and methylene chloride - methanol 97:3. Quantification by densitometry at UV 254 nm. Detection is very sensitive because of fluorescence of alkaloids without purification. Comparison with HPLC, showing that the TLC methodis the most simple, accurate, reproducible

and convenient analytical technique for fast investigations and routine determination of Chelidonium alkaloids.

herbal, comparison of methods, quantitative analysis, qualitative identification 22

99 055 A. VRONDELI, P. KEFALAS, E. KOKKALOU* (*School of Pharmacy, Department of Pharmacognosy, Aristotle University of Thessaloniki, 54124 Greece; kokkalou@pharm.auth.gr).: A new alkaloid from Narcissus serotinus L. Pharmazie 60, 559-560 (2005). TLC of 4-methoxy-5-methyl-1,2,3,5,6,6aR-hexahydro-[1,3]dioxolo[4',5':6,7]isochromeno[3,4-c]indol-8-one, isomer of 3-epimacronine, on silica gel with dichloromethane - methanol - ammonia 95:5:1; and 90:10:1; and dichloromethane - isopropanol - ammonia 70:30:1. Detection with Dragendorff reagent.

herbal, qualitative identification 22

99 056 H. WIEDENFELD*, C. MONTES, B. TAWIL, A. CONTIN, R. WNYSMA (*Pharmazeutisches Institut der Universität, An der Immenburg 4, D-53121 Bonn, Germany; wiedenfeld@uni-bonn.de): Pyrrolozodine alkaloid level in Senecio bicolor (Willd.) Tod., ssp. cineraria (DC.) from middle Europe. Pharmazie 61, 559-561 (2006). Preparative TLC of seven pyrrolizidine alkaloids (senecionine, seneciphylline, integerrimine, jacobine, jacoline, jaconine, jacobine-acetate) on silica gel with dichloromethane - methanol - 25 % ammonia 75:24:1. Detection under UV light.

pharmaceutical research, herbal, preparative TLC 22

23. Other substances containing heterocyclic nitrogen

99 057 S. CHOPRA*, S. K. MOTWANI, Z. IQBAL, F. J. AHMAD, R. K. KHAR (*Faculty of Pharmacy, Department of Pharmaceutics, Jamia Hamdard, Hamdard Nagar, New Delhi 110 062, India; shrutichopra21@yahoo.com).: Quantitative determination and stress degradation studies on a biomarker trigonelline by a validated stability-indicating HPTLC method. J. Liq. Chromatogr. & Relat. Technol. 30, 557-574 (2007). TLC of trigonelline (3-carboxy-1-methylpyridinium hydrochloride) and degradation products on silica gel with n-propanol - methanol - water 4:1:4 in a twin-trough chamber. Quantitative determination by absorbance measurement at 269 nm.

HPTLC, quantitative analysis 23d

99 058 Ute JAUTZ, Gertrud MORLOCK* (*Inst. of Food Chem., Univ. of Hohenheim, Garbenstrasse 28, 70599 Stuttgart; gmorlock@uni-hohenheim.de): Validation of a new planar chromatographic method for quantification of the heterocyclic aromatic amines most frequently found in food. Anal. Bioanal. Chem. 387, 1083-1093 (2007). A new HPTLC method for trace analysis (low µg/kg range) of the five heterocyclic aromatic amines (PhIP, MeIQx, 4,8-DiMeIQx, norharmane, and harmane) in meat samples has been established. After preconditioning of the HPTLC silica gel layer with ammonia vapour the plate was developed with methanol - chloroform 1:9. Quantitative determination by absorbance measurement at UV 262 nm and 316 nm, and fluorescence measurement at UV 366/>400 nm. The UV wavelength 316 nm was later substituted by 313/>340 nm for a more selective and sensitive determination of PhIP in the meat matrix. Mass spectrometric analysis was performed in ESI+ mode for confirmation of positive findings. The method was validated according to ICH guidelines. Repeatability was better than 3.3 % (n=14), intermediate precision better than 2.0 % (n=6). Reproducibility of the migration distance was better than 1.3 % (n=6). LODs of the 5 HAA ranged between 0.4 and 5 ng/band. In the working range RSDs of the calibration functions were between 1.9 and 3.6 %.

food analysis, quality control, HPTLC, heterocyclic aromatic amines, validation 23e, 17a

99 059 N. T. BURDZHIEV, C. E. PALAMAREV, M. D. PALAMAREVA* (*Department of Chemistry, University of Sofia, 1, James Bouchier Avenue, Sofia 1164, Bulgaria; mpalamareva@chem.uni-sofia.bg): Automatic selection of mobile phases. VI. Thin-Layer Chromatography on silica

of libraries of piperidinones. J. Liq. Chromatogr. Relat. Technol. 29, 2045-2057 (2006). TLC of substituted trans-piperidinones comprising 15 amidolactames and 7 aminolactames on silica gel with 13 mobile phase systems with LSChrom software. The procedure takes into account the adsorption properties of the mobile phase (parameter epsilon), stationary phase, and sample structure expressed by the relevant group.

qualitative identification 23e, 3d

24. Organic sulfur compounds

99 060 E. BOURLÈS, R. ALVES DE SOUSA, E. GALARDON, M. SELKTI, A. TOMAS, I. ARTAUD* (*Laboratoire de Chimie et Biochimie Pharmacologique et Toxicologique, CNRS Université Paris, Paris, France, isabelle.artaud@univ-paris5.fr): Synthesis of cyclic mono- and bis-disulfides and their selective conversion to mono- and bis-thiosulfinates. Tetrahedron 63, 2466-2471 (2007). Preparative TLC of two couples of cyclic bis(thiosulfinates) cis/trans stereoisomers derived from bis-disulfides on RP-18 phase by three successive migrations with hexane - ethyl acetate 2:3. Identification after alkaline cleavage of the two S(O)-S bonds followed by metalation with Ni (II).

pharmaceutical research, preparative TLC 24

27. Vitamins and various growth regulators

99 061 S. ADACHI, E. MIYAMOTO, F. WATANABE*, T. ENOMOTO, T. KUDA, M. HAYASHI, Y. NAKANO (*Department of Health Science, Kochi Women's University, Kochi 780-8515, Japan; watanabe@cc.kochi-wu.ac.jp): Purification and characterization of a corrinoid compound from a Japanese salted and fermented salmon kidney "Mefun". J. Liq. Chromatogr. Relat. Technol. 28, 2561-2569 (2005). TLC of cyanocobamides (benzimidazolyl, h-hydroxybenzimidazolyl, and 7-adeninyl cyanocobamides) on silica gel with 1-butanol - 2-propanol - water 10:7:10, and 2-propanol - 28 % ammonium hydroxide - water 7:1:2 in the dark. Detection under visible and UV light.

qualitative identification 27

99 062 B. ARTHUR, B. FRIED*, J. SHERMA (*Department of Biology, Lafayette College, Easton, PA 18042, USA; friedb@lafayette.edu): Effects of estivation on lutein and beta-carotene concentrations in Biomphalaria glabrata (NMRI strain) and Helisoma trivolvis (Colorado strain) snails as determined by quantitative High Performance Reversed Phase Thin Layer Chromatography. J. Liq. Chromatogr. & Relat. Technol. 29, 2159-2165 (2006). HPTLC of beta-carotene and lutein on RP-18 (prewashed with dichloromethane - methanol 1:1) with petroleum ether (35-55°C) - acetonitrile - methanol 1:1:2 in a twin-trough chamber with chamber saturation for 15 min. Evaluation under white light. Experiments were done rapidly in subdued light to prevent pigment degradation. Quantification by densitometry at 448 nm for lutein and 455 nm for beta-carotene.

HPTLC, quantitative analysis, densitometry, biological research 27

99 063 Claudia CIMPOIU*, A. HOSU (*Babes-Bolyai University, Faculty of Chemistry and Chemical Engineering, 11 Arany Janos, 400028, Cluj-Napoca, Romania; ccimpoiu@chem.ubbcluj.ro): Thin Layer Chromatography for the analysis of vitamins and their derivatives. J. Liq. Chromatogr. & Relat. Technol. 30, 701-728 (2007). TLC on silica gel, RP phase, and cellulose as a common analytical method for screening, separation, and preliminary identification of hydrophilic vitamins (vitamin C and B complex: B1, B2, B3, B5, B6, B9, B12, and vitamin H), and lipophilic vitamins (vitamin A, E, D, and K) in tablets, food, and body fluids.

food analysis, quality control, qualitative identification, review 27

99 064 Claudia CIMPOIU*, D. CASONI, A. HOSU, V. MICLAUS, T. HODISAN, G. DAMIAN (*"Babes-Bolyai" University, Faculty of Chemistry and Chemical Engineering, 11 Army Janos, 3400 Cluj-Napoca, Romania; ccimpoiu@chem.ubbcluj.ro): Separation and identification of eight hydrophilic vitamins using a new TLC method and Raman spectroscopy. J. Liq. Chromatogr. Relat. Technol. 28, 2551-2559 (2005). HPTLC of eight hydrophilic vitamins (B1, B2, B3, B5, B6, B9, B12, and C) on silica gel with mixtures of methanol and benzene in a saturated N-chamber. Detection under UV 254 nm. Vitamin B5 was detected by spraying with ninhydrin reagent (2 % in ethanol). Raman spectra were recorded.

food analysis, quality control, qualitative identification 27

99 065 S. GOCAN*, S. COBZAC, N. GRINBERG (*Analytical Chemistry Department, Babes-Bolyai University, RO-400084, Cluj-Napoca, Romania; simiongocan@gmail.com): Prediction of the lipophilicity of some plant growth stimulators by RP-TLC and relationship between slope and intercept of TLC equations. J. Liq. Chromatogr. & Relat. Technol. 30, 1669-1676 (2007). HPTLC of 14 new growth stimulators (such as amido esters of ethanolamine and maleic and succinic acid derivatives) on RP-18 with methanol - water mixtures in saturated chambers. Detection under UV light at 254 nm.

agricultural, qualitative identification, HPTLC 27

99 066 F. WATANABE*, E. MIYAMOTO, Y. TANIOKA, T. ENOMOTO, T. KUDA, Y. NAKANO (*Department of Health Science, Kochi Women's University, Kochi 780-8515, Japan; watanabe@muses.tottori-u.ac.jp): TLC analysis of corrinoid compounds in the halophilic lactic acid bacterium Tetragenococcus halophilus. J. Liq. Chromatogr. & Relat. Technol. 29, 2153-2158 (2006). TLC of a bacterial vitamin B12 extract and vitamin B12 on silica gel with 2-propanol - 28% ammonia - water 7:1:2 in the dark. After drying extraction and determination of the B12 activity by a microbiological B12 assay method.

qualitative identification 27

28. Antibiotics, Mycotoxins

99 067 Irena CHOMA*, I. KOMANIECKA (*Department of Chromatographic Methods, M. Curie-Sklodowska University, M. Sklodowska Sq. 3, 20-031 Lublin, Poland; ichoma@hermes.umcs.lublin.pl): Matrix solid-phase dispersion combined with Thin-Layer Chromatography - direct bioautography for determination of enrofloxacin and ciprofloxacin residues in milk. J. Liq. Chromatogr. Relat. Technol. 28, 2467-2478 (2005). TLC of enrofloxacin and ciprofloxacin on silica gel in sandwich chambers with dichloromethane - methanol - 2-propanol - 25 % ammonia 3:3:5:2. Detection by bioautography using nutrient medium and B. subtilis spore suspension. Establishing of conditions for a semiquantitative TLC-DB (direct bioautography).

food analysis, qualitative identification 28a

99 068 M. M. AL-AJLANI, Shahida HASNAIN* (*Department of Microbiology and Molecular Genetics, University of the Punjab, Quaid-e-Azam Campus, Lahore, Pakistan; genetic@brain.net.pk): Simple and rapid isolation of a novel antibiotic from Bacillus subtilis Mz-7. J. Liq. Chromatogr. Relat. Technol. 29, 639-647 (2006). TLC of a novel antibiotic on silica gel with chloroform - methanol - diethyl ether 1:7:2. For detection the TLC plates were placed in bioassay plates and overlaid with Muller-Hinton agar which had been seeded with B. fusiformis.

qualitative identification, bioassay 28a

99 069 I. M. CHOMA (Department of Chromatographic Methods, University of M. Curie-Sklodowska, M. Sklodowska sq. 3, Lublin 20-031, Poland; ichoma@hermes.umcs.lublin.pl): Thin-Layer

Chromatography - direct bioautography of flumequine residues in milk. J. Liq. Chromatogr. Relat. Technol. 29, 2083-2093 (2006). TLC of flumequine (9-fluoro-6,7-dihydro-5-methyl-1-oxo-1H,5H-benzo[ij]quinolizine-2-carboxylic acid) on silica gel in a sandwich chamber with dichloromethane - methanol - 2-propanol - 25 % aqueous ammonia 3:3:5:2. The plates were developed to the top and then continuously for 1 h. Bioautography with nutrient medium and Bacillus subtilis spore suspension. After incubation the plates were sprayed with MTT-solution.

food analysis, quality control, qualitative identification 28a

29. Pesticides and other agrochemicals

99 070 M. MISZCZYK, Alina PYKA* (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, PL 41-200, Sosnowice, Poland; alinapyka@wo.pl): Investigation of selected sulfonylurea herbicides by TLC and HPLC. J. Liq. Chromatogr. & Relat. Technol. 29, 2437-2449 (2006). TLC of thifensulfuron methyl, triasulfuron, chlorsulfuron, rimsulfuron, amidosulfuron, and tribenuron methyl on silica gel with benzene - methanol 9:1 in a chamber saturated for 30 min, and on RP phase with acetonitrile - methanol - 0.1% phosphoric acid 7:7:6. Detection by iodine vapor or by spraying with a solution of potassium dichromate (5 g) in sulfuric acid (40 %; 100 g), followed by heating to 150 °C.

agricultural, qualitative identification 29d

99 071 G. OROS, T. CSERHATI*, Z. ILLÉS (*Research Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, P. O. Box 17, H-1525 Budapest, Hungary; tevi@chemres.hu): Retention behavior of some thiophosphorylglycinamide fungicides in adsorption and reversed-phase Thin-Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 29, 2009-2018 (2006). TLC of 37 thiophosphorylglycinamide derivatives on silica gel, alumina, and silica gel impregnated with paraffin oil, in sandwich chambers at 4-5 °C with 11 ternary mobile phases from n-pentane - benzene - acetone with different composition. Detection by exposure to iodine vapor. Determination of the free energy of adsorption, the surface area of adsorption and the molecular lipophilicity of the TPGA derivatives.

agricultural, quantitative analysis 29e

99 072 P. TANUJA, N. VENUGOPAL, R. B. SASHIDAR* (*Department of Biochemistry, Osmania University, Hyderabad-500 007, Andhra Pradesh, India; sashi_rao@yahoo.com): Development and evaluation of Thin-Layer Chromatography-Digital Image-Based analysis for the quantitation of botanic pesticide azadirachtin in agricultural matrixes and commercial formulations: Comparison with ELISA. J. Assoc. Off. Anal. Chem. 90, 857-863 (2007). TLC of azadirachtin on silica gel with dichloromethane - ethanol 20:1. Detection by spraying with acidified vanillin reagent (3 g vanillin in 10 mL ethanol with 1.5 mL concentrated sulfuric acid) followed by heating at 110 °C for 3 min. Quantitation by densitometry.

agricultural, food analysis, quantitative analysis, comparison of methods, postchromatographic derivatization 29f

99 073 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081, Lublin, Poland; ttuzim@panaceum.am.lublin.pl): Two-stage fractionation of a mixture of 10 pesticides by TLC and HPLC. J. Liq. Chromatogr. Relat. Technol. 28, 463-476 (2005). TLC of 10 pesticides (triadimenol, metazachlor, triadinefon, quinoxyfen, fenoxycarb, propaquizafob, piperonyl butoxide, quizalofop-P, buprofezin, oxyfluorfen) on silica gel with ethyl acetate - diisopropyl ether 1:9. Evaluation under UV 254 nm. The separated eight fractions were separated with NP and RP systems on RP-18 W and on cyano phase. Evaluation by densitometry and video densitometry. In addition preparative TLC on silica gel with a non-aqueous eluent.

agricultural, densitometry, quantitative analysis qualitative identification, preparative TLC 29

99 074 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Skubiszewski Medical University, Lublin, Poland, tomasz.tuzimiski@am.lublin.pl): A new procedure for separation of complex mixtures of pesticides by multidimensional planar chromatography. J. Sep. Sci. 30, 964-970 (2007). Multidimensional planar chromatography of a mixture of five groups of pesticides: (1) diuron, isoproturon, and lenacil; (2) monolinuron, propoksur, carbaryl, and simazine; (3) alachlor and dinoseb; (4) trifluralin, tetradifon, p,p′-DDT, and 4,4′-dibromobenzophenone; (5) hexachlorobenzene. The silica gel plate was developed in the first dimension with ethyl acetate - n-heptane 1:3, and then turned by 90°. Portions of the stationary phase were sequentially removed to ensure that the mobile phase of the following developments reaches only the target spots: (2) chloroform - n-heptane 19:1 (4) acetone - n-heptane 1:59, (3) toluene, and (1) ethyl acetate - dichloromethane 1:9. The plate was dried between 5 and 15 min before each development. Detection under UV light at 254 nm.

agricultural, environmental, HPTLC, qualitative identification 29d

99 075 T. TUZIMSKI*, J. WOJTOWICZ (*Department of Physical Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081 Lublin, Poland; ttuzim@panaceum.am.lublin.pl).: Separation of a mixture of pesticides by 2D-TLC on two-adsorbent-layer Multi-K SC5 Plate. J. Liq. Chromatogr. Relat. Technol. 28, 277-87 (2005). HPTLC of 16 pesticides (propaquizafop, quizalofop-P, triadimefon, tridimenol, fenoxycarb, quinoxyfen, cyromazine, oxyfluorfen, fluoroglycofen, acetochlor, metazachlor, piperonyl butoxide, furalaxyl, pyriproxifen, buprofezin, clofentezine) on silica gel and RP-18; two-dimensional separation on dual plates (3 cm zone of silica gel parallel to RP-18 layers) with 1.) ethyl acetate - diisopropyl ether 2.5:97.5 and 2.) acetonitrile - water 17:3; or methanol - water 4:1. Detection under UV 254 nm.

agricultural, environmental, HPTLC, qualitative identification 29

30. Synthetic and natural dyes

99 076 O. S. IDOWU*, O. A. ADEGOKE, A. IDOWU, A. A. OLANIYI (*University of Ibadan, Faculty of Phar,macy, Department of Pharmaceutical Chemistry, Ibadab, Nigaria; olakunleid@yahoo.com): Computational models for structure-hydrophobicity relationships of 4-carboxyl-2,6-dinitrophenyl azo hydroxynaphthalenes. J. Assoc. Off. Anal. Chem. 90, 291-298 (2007). TLC of 4-carboxyl-2,6-dinitrophenyl azo hydroxynaphthalenes (6-hydroxy-, 8-hydroxy-5-(4-carboxy-2,6-dintrophenyl azo)-naphthalene, 6-hydroxy-5-(4-carboxy-2,6-dintrophenyl azo)-naphthalene-2-(propan-2-oic acid), 6-hydroxy-5-(4-carboxy-2,6-dintrophenyl azo)-naphthalene-2-(butan-2-one)) on silica gel (impregnated by development with a solution of 5% liquid paraffin in n-hexane) with aqueous mixtures of methanol, acetone, and dimethylformamide. Visual evaluation in white light.

pharmaceutical research, quantitative analysis 30a

99 077 M. WATANABE, T. AOYAMA, Y. TAKASU, K. INOUE, M. TERAO, Y. ITO, H. OKA*, T. GOTO, H. MATSUMOTO (*Aichi Prefectural Institute of Public Health, 7-6, Nagare, Tsuji-machi, Kita-ku, Nagoya 462-8576, Japan; hisao_oka@pref.aichi.lg.jp): A reversed-phase Thin-Layer Chromatography/scanning densitometric method for the qualitative analysis of carthamus yellow in foods. J. Liq. Chromatogr. Relat. Technol. 28, 325-334 (2005). TLC of carthamus yellow in 35 commercial foods, on RP-18 with 2-butanone - methanol - 5 % sodium sulfate - 5 % acetic acid 3:2:5:5 without chamber saturation. Measurement of visible absorption spectrum using scanning densitometry.

food analysis, densitometry, qualitative identification 30

32. Pharmaceutical and biomedical applications

99 078 M. A. A. MOHAMMAD, N. H. ZAWILLA, F. M. EL-ANWAR, S. M. EL-MOGHAZY ALY*

(*Cairo University, Faculty of Pharmacy, Pharmaceutical Department, Kasr El-Aini St, Cairo 11562, Egypt; smoghazy@hotmail.com): Column and Thin-Layer chromatographic methods for the simultaneous determination of acediasulfone in the presence of cinchocaine, and cefuroxime in the presence of its hydrolytic degradation products. J. Assoc. Off. Anal. Chem. 90, 405-413 (2007). TLC of acediasulfone, cinchocaine, and cefuroxime on silica gel with butanol - methanol - tetrahydrofurane - concentrated ammonium hydroxide 10:10:10:1 with chamber saturation. Detection under UV at 254 nm. Quantitative determination by absorbance measurement at 262 nm.

quality control, densitometry, quantitative analysis 32a

99 079 P. A. CHAMPANERKAR*, V. V. VAIDYA, Sunita SHAILAJAN, G. R. SINGH, W. J. SHAH (*Analytical Chemistry Laboratory, S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai 400019, vaidya_vikas@yahoo.com): High-Performance Thin-Layer Chromatographic method for quantification of beta-Sitosterol from Cynodon Dactylon (Linn.) Pers. Indian Drugs 44(1), 43-47 (2007). HPTLC of beta-sitosterol in methanolic extracts of powdered Cynodon dactylon (Linn.) Pers., on silica gel with chloroform - toulene 19:1. Detection with Libermann-Burchard reagent. Quantitative determination by densitometry at 366 nm. Beta-sitosterol response was linear over the range of 40 µg/mL to 90 µg/mL. The amount of beta-sitosterol in the whole plant powder of Canodon dactylon (Linn.) Pers was found to be 0.60 mg. The validated HPTLC method can be used for routine quality control of Cynodon dactylon (Linn.) Pers. whole plant powder and quantification of beta-sitosterol.

traditional medicine, HPTLC, quantitative analysis, densitometry 32a

99 080 N. A. GOMES*, V. V. VAIDYA, H. S. KARMALKAR, G. GUNDI (*Department of Chemistry, S.P.Mandali's Ramnarain Ruia College, Matunga,Mumbai 400019, vaidya_vikas@yahoo.com): Simultaneous determination of Mosapride Citrate and Pantoprazole in pharmaceutical preparation using High Performance Thin Layer Chromatography. Indian Drugs 44 (2), 111-116 (2007). HPTLC for simultaneous determination of mosapride citrate and pantoprazole from pharmaceutical formulations by using loratadine as an internal standard. HPTLC on silica gel with toluene - acetone - methanol 16:4:1. Quantitative determination by absorbance measurement at 254 nm. Mosapride citrate response was linear over the range 0.075 - 0.225 µg/mL, and that for pantoprazole was 0.2 - 0.6 µg/mL. Recovery was 99.6 - 101.3 % for both compounds. The developed method was validated regarding accuracy, precision, and stability, and can be used for routine quality control of formulations containing mosapride citrate and pantoprazole.

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

99 081 M. A. RAVIOLO, Margarita C. BRINÓN* (*Departamento de Farmacia, Facultad de Ciencias Químicas, Universidad Nacional de Córdoba, Ciudad Universitaria, 5000, Córdoba, Argentine; macribi@dqo.fcq.unc.edu.ar): Comparative study of hydrophobicity parameters of novel 5'-carbamates of zidovudine. J. Liq. Chromatogr. Relat. Technol. 28, 2195-2209 (2005). HPTLC of 5'-carbamates of zidovudine (3'-azido-3'-deoxythymidine) and thymidine on RP-18 with methanol - buffer pH 7.4 mixtures with methanol contents between 30 and 80 %; or acetone - buffer mixtures with modifier contents between 20 and 80 % in 5 or 10 % increments. Detection after drying at 40°C developed under UV radiation.

HPTLC 32a

99 082 F. AHMED, M. ALI*, O. SINGH (*Phytochemistry Research Laboratory, Faculty of Pharmacy, Jamia Hamdard (Hamdard University), New Delhi-62, India; mali_chem@yahoo.co.in).: New compounds from Commiphora myrrha (Nees) Engl.. Pharmazie 61,728-731 (2006). TLC of myrracadinol A, myrracalamene A, myrracalamene B, myrracadinol B. triacont-1-ene, myrracalamene C, and myrracadinol C on silica gel with petroleum ether, toluene, petroleum ether - chloroform 4:1, toluene - chloroform 4:1, benzene, benzene - ethyl acetate - diethyl amine 6:3:1,

or chloroform- methanol 17:3. Detection under UV light, by exposure to iodine vapors and by spraying with ceric ammonium sulfate.

herbal, qualitative identification 32e

99 083 H. B. TAMPUBOLON, E. SUMARLIK, M. YUWONO, G. INDRAYANTO* (*Assessment Service Unit, Faculty of Pharmacy, Airlangga University, Jl. Dharmawangsa Dalam, Surabaya 60286, Indonesia; gunawanindrayanto@yahoo.com): Densitometric determination of allylestrenol in tablets, and validation of the method. J. Liq. Chromatogr. Relat. Technol. 28, 267-275 (2005) . TLC of allylestrenol on silica gel in a twin-trough chamber with n-hexane - ethyl acetate - dichloromethane 45:10:1. Detection by spraying with anisaldehyde-sulfuric acid reagent followed by heating at 100 °C for 5 min. Quantitative determination by absorbance measurement at 609 nm.

quality control, densitometry, quantitative analysis 32a

99 085 H. B. TAMPUBOLON, E. SUMARLIK, S. D. SAPUTRA, S. CHOLIFAH, W. F. KARTINASARI; G. INDRAYANTO* (*Assessment Service Unit, Faculty of Pharmacy, Airlangga University, Jl. Dharmawangsa dalam, Surabaya 60286, Indonesia; gunawanindrayanto@yahoo.com): Densitometric determination of tadalafil citrate in tablets. J. Liq. Chromatogr. & Relat. Technol. 29, 2753-2765 (2006). TLC of tadalafil [6R,12aR)-2,3,6,7,12a-hexahydro-2-methyl-6-[3,4-(methylenedioxy)phenyl]pyrazino-[1',2':1,6]pyrido[3,4-b]indole-1,4-dione] on silica gel with n-hexane - ethyl acetate - methanol 4:3:1. Quantitative determination by absorbance measurement at 285 nm.

quality control, clinical chemistry research, densitometry, quantitative analysis 32a

99 086 S. BABIC*, D. MUTAVDZIC, D. ASPERGER, A. J. M. HORVAT, M. KASTELAN-MACAN (*Laboratory of Analytical Chemistry, Faculty of Chemical Engineering and Technology, University of Zagreb, Marulicev trg 20, 10000 Zagreb, Croatia).: Determination of Veterinary Pharmaceuticals in Production Wastewater by HPTLC-Videodensitometry. Chromatographia 65 (1-2), 105-110 (2007). HPTLC of seven pharmaceuticals, enrofloxacine, oxytetracycline, trimethoprim, sulfamethazine, sulfadiazine, sulfaguanidine and penicillin G/procaine in production wastewater (obtained by solid-phase extraction on hydrophilic-lipophilic balance cartridges with methanol) on cyano phase with 0.05 M oxalic acid - methanol 81:19 after optimization of chromatographic separation by systematic variation of the mobile phase composition using genetic algorithm approach. Quantification by videodensitometry at 254 and 366 nm. Validation of the method by investigation of linearity ranges (1.5 - 15.0 µg/L for enrofloxacine, 100 - 500 µg/L for oxytetracycline, 150 - 600 µg/L for trimethoprim, 300 - 1100 µg/L for sulfaguanidine and 100 - 400 µg/L for sulfamethazine, sulfadiazine and penicillin G/procaine, $R > 0.991$), its mean recoveries (74.6 - 117.1% and 55.1 - 108.0% for wellspring water and production wastewater, respectively). Application of the method in determination of pharmaceuticals in wastewater samples from pharmaceutical industry.

environmental, densitometry, quantitative analysis 32c

99 087 M. C. P. A. ALBUQUERQUE, T. G. SILVA, M. G. R. PITTA, A. C. A. SILVA, P. G. SILVA, E. MALAGUENO, J. V. SANTANA, A. G. WANDERLEY, M. C. A. LIMA, S. L. GALDINO, J. BARBE, I. R. PITTA* (*Universidade Federal de Pernambuco, Departimento de Antibioticos, BR-50670-901 Recife, Brasil; irpitta@aol.com): Synthesis and schistosomicidal activity of new substituted thioxo-imidazolidine compounds. Pharmazie 60, 13-17 (2005). TLC of 3-benzyl-5-(4-fluoro-benzylidene)-1-methyl-2-thioxo-imidazolidin-4-ones, 5-benzylidene-3-(4-nitro-benzyl)-2-thioxo-imidazolidin-4-ones, and 4-acridin-9-ylmethylene-1-benzyl-5-thioxo-imidazolidin-2-ones (e. g. 5-(4-fluoro-benzylidene)-1-methyl-2-thioxo-imidazolidin-4-one, 3-benzyl-5-(4-fluoro-benzyidene)-1-methyl-2-thioxo-imidazolidin-4-one) on silica gel with chloroform - methanol 96:4; 99:1; and 98:2; n-hexane - ethyl acetate 7:3; 6:4; and 5:5; and benzene - ethyl acetate 7:3. Detection under UV light.

pharmaceutical research, qualitative identification 32a

99 088 P. CHEN* (Chen Ping), Z. DAI (Dai Zhong), Y. GAO (Gao Yongli), R. LIN (Lin Ruichao) (*Quanzhou Municip. Inst. drug Cont., Quanzhou, Fujian 362000, China): (Study of the quality standard for Tangniaoling tablets) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao), 27 (8), 903 - 905 (2005). TLC of Tangniaoling tablets on silica gel with 1) chloroform - methanol 20:1; 2) chloroform - methanol 10:1. Detection 1) by spraying with 5 % vanillin in H_2SO_4 solution and heating at 105°C. Identification by fingerprint techniques. Quantification of puerarin by HPLC.

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

99 089 S. CHOLIFAH, A. NOVIANSARI, W. F. KARTINASARI, G. INDRAYANTO* (*Assessmant Service Unit, Faculty of Pharmacy, Airlangga University, Jl. Dharmawangsa dalam, Surabaya 60286, Indonesia; gunawanindrayanto@yahoo.com): Densitometric determination of fenbendazole in veterinarian suspension. J. Liq. Chromatogr. & Relat. Technol. 30, 489-498 (2007). TLC of fenbendazole (methyl [5-(phenylthio)-1H-benzimidazole-2-yl]-carbamic acid methyl ester) on silica gel using dichloromethane - ethyl acetate - formic acid - methanol 60:5:3:3. Quantitative determination by absorbance measurement at 293 nm. Linearity (peak area) is given between 500 and 1400 ng/spot.

quality control, quantitative analysis, densitometry 32a

99 090 C. D. BIRK, G. PROVENSI, Grace GOSMANN*, F. H. REGINATTO, E. P. SCHENKEL (*Faculdade de Farmácia, Universidade Federal do Rio Grande do Sul, (UFRGS), Av. Ipiranga, 2752 Porto Alegre, RS 90610-000, Brazil; grace.gosmann@ufrgs.br): TLC Fingerprint of flavonoids and saponins from Passiflora species. J. Liq. Chromatogr. Relat. Technol. 28, 2285-2291 (2005). TLC of flavonoids with vitexin and quadrangulosides as standards on silica gel with ethyl acetate - acetone - acetic acid - water 6:2:1:1, and saponins with chloroform - ethanol - acetic acid 12:8:1. Detection by spraying with anisaldehyde-sulfuric acid reagent, then heating to 100 °C. Also detection by spraying with a 0.5 % methanolic solution of diphenylboryloxyethylamine (natural products reagent) followed by spraying with 5 % PEG 400. Evaluation under white light and UV 365 nm.

quality control, qualitative identification 32e

99 091 W. D. XIE (Xie Wei-Dong), X. GAO (Gao Xue), T. SHEN (Shen Tong), Z. J. JIA (Jia Zhong-Jian)* (*Department of Chemistry, Lanzhou University, Lanzhou, 730000, P. R. China; jiazj@lzu.edu.cn): Two new benzofurans and other constituents from Ligularia przewalskii. Pharmazie 61, 556-558 (2006). Preparative TLC of two benzofurans euparin, friedelin, and beta-sitosterol on silica gel with petroleum ether - ethyl acetate 20:1. Detection under UV light.

traditional medicine, herbal, pharmaceutical research, preparative TLC 32e

99 092 H. DE MELLO, Aurea ECHEVARRIA* (*Departamento de Química, Intituto de Ciencias Exatas, Universidade Federal Rural do Rio de Janeiro, Seropédica/RJ 23851-970, Brazil; echevarr@ufrrj.br). : Hydrophobicity study for some pyrazolo-pyridine derivatives by RP-TLC and RP-HPLC. J. Liq. Chromatogr. Relat. Technol. 29, 1317-1330 (2006). TLC of 13 1H-pyrazolo[3,4-b]pyidine derivatives (e. g. 4-(3'- or 4'-X-phenylamino)-5-carbethoxy-1,3-dimethl-1-H-pyrazolo[3,4-b]pyridine derivatives) on hydrocarbon impregnated silica gel with acetone - phosphate buffer (0.01 M; pH 7.4) mixtures with concentrations ranging from 40-70 % in acetone. Detection under UV 254 nm.

pharmaceutical research 32a

99 093 A. DELAZAR, F. BIGLARI, S. ESNAASHARI, H. NAZEMIYEH, A. TALEBPOUR, L.

NAHAR, S. SAKER* (*School of Biomedical Sciences, University of Ulster at Coleraine, Londonderry, Northern Ireland, UK, s.sarker@ulster.ac.uk): GC-MS analysis of the essential oils, and the isolation of phenylpropanoid derivatives from the aerial parts of Pimpinella aurea. Phytochemistry 67, 2176-2181(2006). Preparative TLC of the aerial parts of Pimpinella aurea (ethyl acetate fraction of dichloromethane extracts) on silica gel with ethyl acetate - hexane - acetic acid 120:80:1. Detection under UV 254 nm. Isolation of erythro-1′-(4-methoxyphenyl)-propan-1′,2′-diol (Rf=0.32) and erythro-1′-(4-(sec-butyl)-phenyl)-propan-1′,2′-diol (Rf=0.38) with chemotaxonomic significance.

traditional medicine, herbal, preparative TLC 32e

99 094 Q. DU (Du Qizhen), W. DAIJIE (Daijie Wang), Y. ITO* (*Laboratory of Biophysical Chemistry, National Heart, Lung, and Blood Institute, National Institutes of Health, Building 50, Room 3334, 50 South Drive MSC 8014, Bethesda, MD 20892, USA; itoy@nhlbi.nih.gov): Preparation of solanesol from a tobacco leaf extract using high speed countercurrent chromatography. J. Liq. Chromatogr. & Relat. Technol. 29, 2587-2592 (2006). TLC of solanesol on silica gel with petroleum ether - ethyl acetate 4:1. Detection by spraying with sulfuric acid - anisaldehyde - glacial acetic acid 5:5:90, followed by at 110 °C.

herbal, qualitative identification 32d

99 095 C. ENGELHARDT, F. PETEREIT, J. ANKE, A. HENSEL* (*Institut für Pharmazeutische Biologie und Phytochemie, Westfälische Wilhelms-Universität, Hittorfstr. 56, D-48149 Münster, Germany; ahensel@uni-muenster.de): A new arbutin derivative from the herb of Myrothamnus flabellifolia Welw. Pharmazie 62, 558-559 (2007). TLC of the ethyl acetate soluble fraction of an acetone/water extract (2,3-di-O-galloylarbutin) on silica gel with ethyl acetate - acetic acid - water 18:1:1. Visualization by spraying with natural products reagent, vanillin/hydrochloric acid or anisaldehyde/sulfuric acid reagent.

herbal, qualitative identification 32e

99 096 S. FENG (Feng Suomin), S. NI (Ni Shifeng), W. SUN (Sun Wenji)* (*Biomedical Key Laboratory of Northwest University , No. 229, Taibai North Road, Xi'an, Shaanxi 710069, P. R. China; fengsuomin@126.com): Preparative isolation and purification of the lignan pinoresinol diglucoside and liriodendrin from the bark of Eucommia Ulmoides Oliv. by high speed countercurrent chromatography. J. Liq. Chromatogr. & Relat. Technol. 30, 135-145 (2007). TLC of pinoresinol diglucoside and liriodendrin on silica gel (impregnated with 1 % carboxymethyl cellulose sodium) with the lower phase of chloroform - methanol - water 65:35:16. Detection by spraying with 10 % ethanolic sulfuric acid.

traditional medicine, herbal, qualitative identification 32e

99 097 U. FRIEDRICH, K. SIEMS, P. N. SOLIS, M. P. GUPTA, Kristina JENETT-SIEMS* (*Institut für Pharmazie (Pharmazeutische Biologie), Königin-Luise-Str. 2-4, D-14195 Berlin, Germany; kjsiems@zedat.fu-berlin.de).: New prenylated benzoic acid derivatives of Piper hispidum. Pharmazie 60, 455-457 (2005). Preparative TLC of nervogenic acid, nervogenic acid methyl ether, 2,2-dimethyl-8-(3-methyl-2-butenyl)-2H-chromene-6-carboxylic acid, dillapional, dillapiol aldehyde, N-trans-feruloyltyramine, omega-hydroxyisodillapiole, 4-hydroxy-3-(3-methyl-2-butenyl)benzoate, and three new 4-hydroxy-benzoic acid derivatives, 4-methoxy-3,5-bis-(3-hydroxy-3-methyl-1-butenyl)benzoate, 3-hydroxy-2-(1-hydroxy-1-methylethyl)-2,3-dihydroxybenzofuran-5-carboxylic acid, and 3-hydroxy-2-(1-hydroxy-1-methylethyl)-2,3-dihydrobenzofuran-5-carboxylic acid methyl ester on silica gel with chloroform - ethyl acetate - formic acid - 90:10:1; chloroform - methanol 9:1; 8:2; and ethyl acetate - formic acid - water 82:9:9. Detection under UV light.

herbal, preparative TLC 32e

99 098 S. FROELICH, K. SIEMS, M. A. HERNÁNDEZ, R. A. IBARRA, W. G. BERENDSOHN, Kristina JENETT-SIEMS* (*Institut für Pharmazie, Pharmazeutische Biologie, Freie Universität Berlin, Königin-Luise Str. 2-4, D-14195 Berlin, Germany; kjsiems@zedat.fu-berlin.de): Phenolic glycosides from Exostema mexicanum leaves. Pharmazie 61, 641-644 (2006). Preparative TLC of two novel acylated flavonol glycosides and three glycosides (structurally belonging to the group of 4-phenylcoumarins) on silica gel with formic acid - water - ethyl acetate 9:9:82. Detection by spraying with 1 % methanolic solution of diphenylboric acid 2-aminoethylester (natural products reagent), followed by drying. Evaluation under UV 366 nm.

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

99 099 M. G. BOGDANOV, M. I. KANDINSKA, CH. E. PALAMAREV, M. D. PALAMAREVA* (*Department of Chemistry, University of Sofia, 1, James Bouchier Avenue, Sofia 1164, Bulgaria: mpalamareva@chem.uni.-sofia.bg): Automatic selection of mobile phases. V. Thin-Layer Chromatography on silica gel vs. alumina of 3,4-disubstituted isochroman-1-ones including spiro analogues. J. Liq. Chromatogr. Relat. Technol. 28, 2539-2550 (2005). TLC of 8 substituted isochromanones and 4 spiro analogues on silica gel and alumina with 10 computer selected mobile phases. The procedure takes into account the adsorption properties of the mobile phase (parameter epsilon and tuning parameters m and P'), stationary phase and sample structure expressed by the relevant group. Silica gel was more effective and did not cause decomposition in contrast to the alumina phase.

qualitative identification 32e

99 100 B. G. CHAUDHARI*, N. M. PATEL, P. B. SHAH, K. P. MODI (*Shri B. M. Shah College of Pharmacy, Modasa 383315, Shri B. M. Shah College of Pharmaceutical Education & Research , Modasa 383315, India): Development and validation of a HPTLC method for the simultaneous estimation of atorvastatin calcium and ezetimibe. Indian J. Pharm. Sci. 68 (6), 793-796 (2006). HPTLC of atorvastatin calcium and ezetimibe in combined dosage form on silica gel with chloroform - benzene - methanol - acetic acid 60:30:10:1. Detection under UV 250 nm.The method was validated in terms of linearity, accuracy, precision and specificity.The calibration curve was found to be linear between 0.8 and 4.0 µg/spot for atorvastatin calcium and 0.1 and 1.0 µg/spot for ezetimibe. The limit of detection and the limit of quantification for atorvastation calcium were found to be 170 ng/spot and 570 ng/spot, respectively, and for ezetimibe 20 ng/spot and 70 ng/spot, respectively.The recovery was in the range of 99.9 - 102.7 %.

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

99 101 M. GANDHIMATI*, T. K. RAVI, Nilima SHUKLA (*Department of Pharmaceutical Analysis,Sri Ramkrishna College of Pharmacy, 395, Sarojini Naidu Road, Coimbatore 641044, India): Validated High Performance Thin Layer Chromatography Method for Simultaneous Estimation of Ofloxacin and Ornidazole in Tablet Dosage Form. Ind. J. Pharm. Sci. 68 (6), 838-840 (2006). HPTLC of ofloxacin and ornidazole in tablet dosage form on silica gel with n-butanol - ethanol - ammonia 5:5:4. Quantitative determination by absorbance measurement at 295 nm. The method was found to be linear in the concentrate range of 1-5 ng/spot with recovery of 99.5-102.5 % for both compounds. The method was validated for linearity, accuracy, precision, repeatability, and specificity.

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

99 102 K. GOERLITZER*, S. HUTH, P. G. JONES (*Institut für Pharmazeutische Chemie, Beethovenstrasse 55, D-38106 Braunschweig, Germany; k.goerlitzer@tu-bs.de): Zur Farbreaktion von Chlorhexidin und Proguanil mit Hypobromit (Colour reaction of chlorhexidine and proguanil with hypobromite) (German). Pharmazie 60, 269-272 (2005). TLC of (E)-3-[(4-chlorphenyl)imino]-

N-isopropyl-3H-1,2,4-triazol-5-amin on silica gel with heptane - tetrahydrofuran -methanol - water 30:20:2:1. Evaluation under white light.

pharmaceutical research, qualitative identification 32a

99 103 M. GU (Gu Ming)*, Z. SU (Su Zhiguo), F. OUYANG (Ouyang Fan) (*National Key Laboratory of Biochemical Engineering, Institute of Process Engineering, Chinese Academy of Sciences, P. O. Box 353, Beijing 100080, P. R. China; guming@home.ipe.ac.cn or rainbow_gm@yahoo.com): Fingerprinting of Salvia miltiorrhiza Bunge by Thin-Layer Chromatography scan compared with High Speed Countercurrent Chromatography. J. Liq. Chromatogr. Relat. Technol. 29, 1503-1514 (2006). TLC of tanshinones (cryptotanshinone, tanshinone I, tanshinone II A) on silica gel with light petroleum (60-90°C) - ethyl acetate 4:1. Evaluation and scanning densitometry at 280 nm. With TLC 8 stable components were separated in common within 48 min, respectively, from 3 crude samples of Salvia miltiorrhiza Bunge from different growth locations. With High Speed Countercurrent Chromatography (HSCCC) 12 components were separated, respectively, with good correspondence and precision within 13 h. Both TLCS and HSCCC were effective in showing the whole concentration distribution of all kinds of constituents in different samples. HSCCC showed better performance in analysis of tanshinones, which produced a fingerprint which contained more chemical information than that of TLC.

traditional medicine, densitometry 32e

99 104 Gilda GUIMARAES LEITAO*, P. A. DE SOUZA, A. A. MORAES, L. BROWN (*Núcleo de Pesquisas de Produtos Naturais (NPPN), Universidade Federal do Rio de Janeiro, Bloco H, CCS, Ilha do Fundao, 21941-590, Rio de Janeiro, RJ, Brazil: ggleitao@nppn.ufri.br): Step-gradient CCC separation of phenylpropanoid and iridoid glycosides from roots of Stachytarpheta cayennensis (Rich.) Vahl. J. Liq. Chromatogr. Relat. Technol. 28, 2053-2060 (2005). TLC of glycosylated phenylpropanoids and iridoids (martinoside, isoverbascoside, verbascoside, ipolamiide, and two more iridoid glycosides) on silica gel with the organic phase of ethyl acetate - acetone - water 25:8:5. Detection by spraying with 1 % vanillin in sulfuric acid.

herbal, qualitative identification 32e

99 105 Gilda GUIMARAES LEITAO*, S. S. Al-ADJI, W. A. LOPES DE MELO (*Núcleo de Pesquisas de Produtos Naturais, Universidade Federal do Rio de Janeiro, Bl. H, CCS, Ilha do Fundao, 21.941-590, Rio de Janeiro, RJ, Brazil: ggleitao@nppn.ufri.br): Separation of free and gycolysated flavonoids from Siparuna guianensis by gradient and isocratic CCC. J. Liq. Chromatogr. Relat. Technol. 28, 2041-2051 (2005). TLC of rhamnosyl kaempferol, rutin, and quercetin-7-O-rutinoside on silica gel with the organic phase of ethyl acetate - acetone - water 5:2:1; and butanol - acetic acid - water 4:1:5. Detection by spraying with Folin-Ciocalteus reagent.

herbal, qualitative identification 32e

99 106 X. GUO* (Guo Xiaoling), L. HAN (Han Liang), Y. FENG (Feng Yifan), X. MENG (Meng Qing) (*Guangdong Inst. Pharm., Guangzhou 510006, China): (Study of the quality standard for Fengshi Gutong tincture) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao), 29 (3), 386-389 (2007). TLC of Fengshi Gutong tincture on silica gel with 1) chloroform - methanol - ammonia 80:20:3; 2) n-hexane - ethyl acetate - 4:1; 3) benzene - ethyl acetate - methanol 6:4:1; 4) benzene - ethyl acetate - methanol - isopropanol - water 60:15:15:10:3. Detection 1) by spraying with reagent of FeCl3 - K3Fe(CN)6; 2) under UV 365 nm; 3) by spraying with 10 % H2SO4 in ethanol and heating at 105 °C.

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

99 107 Fadia H. METWALLY*, M. ABDELKAWY, I. A.NAGUIB (*Cairo University, Faculty of Pharmacy, Analytical Chemistry Department, Kasr El-Aini St, 11562, Cairo, Egypt, fadiahm@yahoo.com): Development and validation of three stability-indicating methods for determination of bisacodyl in pure form and pharmaceutical preparations J. Assoc. Off. Anal. Chem. 90, 113-127 (2007) TLC of bisacodyl [4,4'-(2-pyridylmethylene)-bisphenol diacetate], monoacetyl bisacodyl and desacetyl bisacodyl on silica gel with chloroform - acetone 9:1. Quantitative absorbance measurement at 223 nm. Concentration range for bisacodyl was 0.2 - 1.4 µg/band, mean recovery was 100.4 +/- 1.9 %.

quality control, densitometry quantitative analysis 32a

99 108 Fadia H. METWALLY*, Y. S. EL-SAHARTY, M. REFAAT, S. Z. EL-KHATEEB (*Cairo University, Faculty of Pharmacy, Analytical Chemistry Department, El-Kasr El-Aini St, ET-11562 Cairo, Egypt; fadiahm@yahoo.com): Application of derivative, derivative ratio, and multivariate spectral analysis and Thin-Layer Chromatography-densitometry for determination of a ternary mixture containing drovaterine hydrochloride, caffeine, and paracetamol. J. Assoc. Off. Anal. Chem. 90, 391-404 (2007). TLC of drotaverine hydrochloride, caffeine, and paracetamol on silica gel with ethyl acetate - chloroform - methanol 16:3:1 with chamber saturation for at least 30 min. Evaluation at UV 254 nm. Quantitative determination by absorbance measurement at 281 nm, 272 nm, and 248 nm.

quality control, densitometry, quantitative analysis 32a

99 109 B. H. PATEL*, B. N. SUHAGIA, M. M. PATEL, J. R. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Department of Pharmaceutical Chemistry, Ganpat Vidyanagar, Kherva, Mehsana-382711, Gujarat, India, bhpmph@yahoo.co.in).: Simultaneous estimation of pantoprazole and domperidone in pure powder and a pharmaceutical formulation by High -Performance Liquid Chromatography and High-Performance Thin-Layer Chromatography methods. J. Assoc. Off. Anal. Chem. 90, 142-146 (2007). HPTLC of pantoprazole [5-(difluoromethoxy)-2-[(3,4-dimethoxy-2-pyridyl)methyl-sulfinyl]1H-benzimidazole] and domperidone (5-chloro-1-[1-[3-(2,3-dihydro-2-oxo-1H-benzimidazol-1-yl)propyl]-4-piperidinyl]-1,3-dihydro-2H-benzimidazol-2-one) on silica gel with ethyl acetate - methanol 3:2 in a twin-trough chamber saturated for 30 min. Quantitative determination by absorbance measurement at 287 over the concentration range of 80 - 240 and 60 -180 ng/spot with mean recovery of 98.4 +/- 0.7 % for pantoprazole, and 98.8 +/- 0.7 % for domperidone.

quality control, HPTLC, densitometry 32a

99 110 A. HAZEKAMP*, A. PELTENBURG, R. VERPOORTE, C. GIROUD (*Division of Pharmacognosy, Institute of Biology, Leiden University, Einsteinweg 55, 2300 RA, Leiden, The Netherlands; ahazekamp@rocketmail.com).: Chromatographic and spectroscopic data of cannabinoids from Cannabis sativa L. J. Liq. Chromatogr. Relat. Technol. 28, 2361-2382 (2005). TLC of (-)-delta9-tetrahydrocannabinol, cannabinol, cannabidiol, cannabigerol, (-)-delta9-(trans)-tetrahydrocannabinolic acid A, cannabidioloc acid, and cannabigerolic acid as reference compounds on RP-18 with methanol - 5 % acetic acid 19:1; and on silica gel with chloroform - methanol 19:1. Evaluation under UV 254 nm. Detection by spraying with modified anisaldehyde - sulfuric acid reagent. For selective detection of cannabinoids, plates were sprayed with 0.5 % fast blue B salt in water, followed by spraying with 0.1 M sodium hydroxide solution.

pharmaceutical research, qualitative identification 32c

99 111 S. HUANG* (Huang Shengwu), Z. WU (Wu Zhihui), X. HU (Hu Xibo), J. LI (Li Jun) (*Zhejiang Univ. TCM, Hangzhou 310053, China).: (Study of the quality standard for Anxinkang dropping pills) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 29 (2), 217 0 221 (2007). TLC of the extracts of Anxinkang dropping pills on silica gel with 1) chloroform - methanol - ammonia

25:3:1; 2) petroleum ether (60 - 90 ºC) - ethyl acetate 20:1; 3) ethyl acetate - acetone - methanol 5:5:1. Detection 1) by spraying with 10 % potassium iodobismuthate solution; 2) by spraying with 5 % p-diethylaminobenzaldehyde in 10 % H2SO4 in ethanol, and heating at 105 °C for 10 min; 3) by spraying with 10 % H2SO4 in ethanol and heating at 105 °C. Identification by comparison with standard.

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

99 112 J. IQBAL*, A. GUPTA, A. HUSAIN (*Organic Chemistry Section, Department of Chemistry, Aligarh Muslim University, Aligarh-202002 (U. P.), India; jawaid.iqbal@lycos.com): Photochemistry of phenazopyridine hydrochloride. Pharmazie 61, 747-750 (2006). TLC of phenazopyridine and 4 major metabolites (i. a. p-methoxyaniline) on silica gel with chloroform - methanol mixtures. Also irradiation of phenazopyridine adsorbed on silica gel. The drug was dissolved in methanol and mixed with aqueous slurry of silica gel. TLC plates were prepared and wet plate photolyzed as such with a mercury lamp. The plate appeared as yellow chromatogram, which turned dark yellow within 15 min. The progress of reaction was monitored by Co-TLC of a withdrawn scratch with the starting drug.

quality control, qualitative identification, AMD 32 a

99 113 V. ISELI, O. POTTERAT, L. HAGMANN, J. EGLI, M. HAMBURGER* (*Institute of Pharmaceutical Biology, Department of Pharmaceutical Sciences, University of Basel, Klingelbergstrasse 50, CH-4056 Basel, Switzerland; Matthias.hamburger@unibas.ch). : Characterization of the pungent principles and the essential oil of Zanthoxylum schinifolium pericarp. Pharmazie 62, 396-400 (2007). TLC of the CO2 extracts of Z. schinifolium and Z. bungeanum (hydroxy-alpha-sanshool and hydoxy-beta-sanshool) on silica gel with chloroform - methanol 9:1. Detection by spraying with vanillin-sulfuric acid reagent.

qualitative identification 32e

99 114 L. J. PATEL*, B. N. SUHAGIA, P. B. SHAH, R. R. SHAH (*Shri B. M. Shah College of Pharmacy, Modasa 383315, India).: TP-HPTLC and HPTLC methods for the estimation of carvedilol in bulk drug and pharmaceutical formulations. Indian J. Pharm. Sci. 68 (6), 790-793 (2007). HPTLC of carvedilol in bulk drug and pharmaceutical formulations, on silica gel with ethyl acetate - toluene - methanol 2:8:7. Quantitative absorbance measurement at 242 nm. The hRf value of carvedilol was 65. The method was found to be linear over the concentration range of 50-300 ng/spot with recovery of 98.3-101.1%.The method was validated for accuracy and precision. Comparison with an HPLC method showed the HPTLC method to be advantageous regarding sample throughput.

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

99 115 N. J. SHAH*, S. J. SHAH, D. M. PATEL, N. M. PATEL (*Shri b.M.Shah College of Pharmaceutical Education and Research, Modasa 383315, India): Development and Validation of HPTLC method for the estimation of Etoricoxib. Indian J. Pharm. Sci. 68 (6),788-789 (2007). HPTLC of etorocoxib in dosage forms on silica gel with chloroform - methanol - toulene 2:1:2. The plate was scanned at 289 nm for quantitative evaluation. The hRf value of etorocoxib was 58. The method was linear in the range of 100 - 600 ng/spot.The method was validated for accuracy, precision and repeatability. It was found suitable for routine quality control of formulations.

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

99 116 G. JUERGENLIEMK, F. PETEREIT, A. NAHRSTEDT* (*Institute of Pharmaceutical Biology and Phytochemistry of the Westf. Wilhelms-University, Hittorfstr. 56, D-48149 Münster, Germany; nahrste@uni-muenster.de): Flavan-3-ols and procyanidins from the bark of Salix purpurea L. Pharmazie 62, 231-234 (2007). TLC of flavan-3-ols and dimeric and trimeric procyanidins on silica gel with ethyl acetate - formic acid - water 18:1:1. Detection by spraying with natural products reagent, vanillin-hydrochloric acid, and anisaldehyde-sulfuric acid.

herbal, qualitative identification 32e

99 117 H. KALÁSZ*, A. HUNYADI, M. BÀTHORI (*Department of Pharmacology & Therapeutics, Faculty of Medicine and Health Sciences, United Arab Emirates University, P. O. Box 17666, Al Aim, United Arab Emirates; huba@kalasz.com): Novel results of two-dimensional Thin-Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 28, 2489-2491 (2005). 2-D TLC of 4 ecdysteroids and several flavonoids on cyano phase with toluene - acetone - ethanol - 25 % ammonia 100:140:32:9 and ethyl acetate - ethanol - water 16:2:1. Detection under UV 254 nm, and under white light and UV 366 nm after spraying with vanillin-sulfuric acid reagent followed by heating. TLC of L-deprenyl and 14C-L-deprenyl((-)-N-methyl-N-propynyl(2-phenyl-1-methyl)ethylammonium hydrochloride) on silica gel with chloroform - methanol - water 7:5:1, and dichloromethane - triethanolamine 19:1 for elution and displacement, that is for the first and second dimensional developments, respectively.

qualitative identification 32e

99 118 U. KIJKOWSKA-MURAK, D. MATOSIUK, A. HAWRYL, Monika WAKSMUNDZKA-HAJNOS*, B. KURAN, J. KOSSAKOWSKI (*Department of Inorganic Chemistry, Faculty of Pharmacy, Medical University, 6 Staszica, 20081 Lublin, Poland; monika.hajnos@am.lublin.pl): Use of RP-HPTLC systems for the determination of lipophilicity of 3,5-dioxo-4-azatricyclo[5.2.2.02, 6]undecanes - 5-HT 1A antagonists. J. Liq. Chromatogr. Relat. Technol. 29, 2019-2033 (2006). HPTLC of twelve 3,5-dioxo-4-azatricyclo[5.2.2.02,6]undecanes on RP-18 W and RP-18 in horizontal chambers. Mobile phases were prepared by mixing the respective amounts of water and polar modifiers (methanol, dioxane, acetone) in the range from 50 - 75 or 90 % for RP, and 40, 50 - 65, or 75 % for RP W-plates. Evaluation under UV 254 nm.

pharmaceutical research, HPTLC 32a

99 119 J. KOCHANA*, A. PARCZEWSKI, J. WILAMOWSKI (*Department of Analytical Chemistry, Faculty of Chemistry, Jagiellonian University, Ingardena 3, Cracow 30-060, Poland; kochana@chemia.uj.edu.pl): SPE/TLC profiling of the impurities of MDMA: The influence of an agglutinant, diluents, and adulterants. J. Liq. Chromatogr. Relat. Technol. 29, 1247-1256 (2006). TLC of MDMA (3,4-methyledioxymethamphetamine) and additives (magnesium stearate, aspirin, paracetamol, caffeine, glucose, citric acid) with acetonitrile - chloroform 1:1, chloroform - methanol 9:1 (best separation), acetonitrile - chloroform - ammonia 2:8:1, chloroform - methanol - ammonia 9:1:1, and chloroform - acetone - methanol - ammonia 10:8:1:1. Detection under UV 254 and 366 nm.

toxicology, qualitative identification 32c

99 120 Jolanta KOCHANA*, A. ZAKRZEWSKA, A. PARCZEWSKI, J. WILAMOWSKI (*Department of Analytical Chemistry, Jagiellonian University, Ingardena 3, 30-060 Cracow, Poland; kochana@chemia.uj.esu.pl): TLC screening method for identification of active components of "ecstasy" tablets. Influence of diluents and adulterants. J. Liq. Chromatogr. Relat. Technol. 28, 2875-2886 (2005). TLC of the active components of "ecstasy" (MDMA, PMA, PMMA, and ephedrine) on silica gel with 10 mobile phases. The simplex method has been employed to find the optimum composition of the eluent chloroform - dioxane - methanol - ammonia - acetonitrile 7:30:4:3:30. Detection under UV light at 254 nm.

toxicology, qualitative identification 32c

99 121 U. KOLAK*, A. TUERKEKUL, F. OEZGOEKCE, A. ULUBELEN (*Faculty of Pharmacy, Department of General Chemistry and Analytical Chemistry, Istanbul University, 34116, Istanbul, Turkey; ufukkolak@yahoo.com): Two new diterpenoid alkaloids from Aconitum cochleare. Pharmazie 60, 953-955 (2005). TLC of cochleareine, acoleareine, 14-acetyltalatisamine, and talatisamine on silica gel. Detection under UV light at 254 nm. Also co-chromatography with standards. Using a Chromatotron apparatus the crude alkaloidal mixture was separated on alumina radial plates and eluted with a gradient of petroleum ether, chloroform, and methanol.

herbal, pharmaceutical research, qualitative identification, preparative TLC 32e

99 122 T. KOOBKOKKRUAD, A. CHOCHAI, C. KERDMANEE, W. DE-EKNAMKUL* (*Department of Pharmacognosy, Faculty of Pharmaceutical Sciences, Chulalongkorn University, Thailand, dwanchai@chula.ac.th): TLC-Densitometric analysis of artemisinin for the rapid screening of high-producing plantlets of Artemisia annua L. Phytochem Anal. 18, 229-234 (2007). HPTLC of Artemisia annua leaves on silica gel with hexane - ethyl acetate - acetone 16:1:1. Detection by exposing to ammonia vapor at 100ºC for 2 hours. Quantitative determination by absorbance measurement at 320 nm. Linearity is between 0.5 and 12 µg/mL and the limit of detection is 0.5 µg/mL. The method is as sensitive and accurate as the HPLC-UV method involving a pre-column reaction.

herbal, HPTLC, quantitative analysis, comparison of methods, densitometry 32e

99 123 Dorota KOWALCZUK*, M. B. WAWRZYCKA, A. H. MAJ (*Department of Medicinal Chemistry, Medical University, 4, Jaczewskiego Str., 20-090 Lublin, Poland; dorota.kowalczuk@am.lublin.pl): Application of an HPTLC densitometric method for the quantification and identification of nifedipine. J. Liq. Chromatogr. & Relat. Technol. 29, 2863-2873 (2006). HPTLC of nifedipine (1,4-dihydro-2,6-dimethyl-4-(2-nitro-phenyl)-3,5-pyridinedicarboxylic acid dimethyl ester) on silica gel in horizontal chamber with n-hexane - ethyl acetate - acetone 6:3:2. Quantitative determination by absorbance masurement at 335 nm.

quality control, quantitative analysis, HPTLC 32a

99 124 J. KRZEK*, U. HUBICKA, J. SZCZEPANCZYK, A. KWIECIEN, W. RZESZUTKO (*Department of Inorganic and Analytical Chemistry, Jagiellonian University, Collegium Medicum, Medyczna 9, 30-688 Krakòw, Poland; jankrzek@cm-uj.krakow.pl): Simultaneous determination of fusidic acid, m- and p-hydroxybenzoates and butylhydroxyanisol by TLC with densitometric detection in UV. J. Liq. Chromatogr. & Relat. Technol. 29, 2129-2139 (2006). TLC of fusidic acid on silica gel with n-hexane - ethyl acetate - glacial acetic acid 6:3:1. To detect the spots on chromatograms, densitometric measurements at three different wavelengths were carried out, i. e., 240 nm (FA), 260 nm (MHB, PHB), and 290 nm (BHA), leading to increased selectivity and decreased interferences of the peaks.

quality control, qualitative identification, quantitative analysis, densitometry 32a

99 125 A. KUMAR*, A. KUMAR, A. J. BAXI (*Department of Pharmaceutical Sciences, Birla Institute of Technology, Mesra, Ranchi 835215, abhishekkumar_78@rediff.com): Standardization of ayurvedic medicated oil and the effect of Moorchhan on the amount of marker in the oil. Indian Drug 44 (2), 122-127 (2007). HPTLC of ayurvedic medicated oil on silica gel in a twin-trough chamber with ethyl acetate - toulene 1:9 for fingerprint analysis, and ethyl acetate - methanol - water 200:27:20 for quantitative determination of colchicine.

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

99 126 K. L. KRISHNA*, M. PARIDHAVI, S. S. AGARWA (*Department of Pharmacology, Shree Dhanvantary Pharmacy College and Pharamceutical Analysis & Research Centre, Kim Surat Dt.

Gujarat, Krishpharm@rediffmail.com): Physico-chemical standardization of sufoof-e-suzak qawi an unani polyherbomineral formulation. Indian Drugs 44 (3), 220-223 (2007). TLC of the volatile oil of Sufoof-E-Suzak Qawi, an Unani medicine, on silica gel with different mobile phases. Detection under UV 254 and 366 nm, and with vanillin sulphuric acid reagent, iodine vapour, and 5 % ethanol sulphuric acid.

traditional medicine, HPTLC 32a

99 127 V. L. SURYAVANSHI*, P. A. SATHE, M. M. BAING, G. R. SINGH, S. N. LAKSHMI (* Department of Chemistry, S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai, 400 019, India): Determination of Rutin in Amaranthus spinosus Linn. Whole Plant Powder by HPTLC. Chromatographia 65 (11-12), 767-769 (2006). Description of a simple, precise and accurate HPTLC method for the determination of rutin in the whole plant powder of Amaranthus spinosus Linn, which has been reported to have anti-diabetic, anti-thrombotic, anti-inflammatory and anti-carcinogenic activity. TLC of a methanol extract of the whole plant powder on silica gel with ethyl acetate - formic acid - methanol - water 100:9:11:17. Quantitative determination by densitometric measurement in absorbance mode at 363 nm. Linearity was between 10 and 60 µg/mL.

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

99 128 F. L. YAN (Yan Fu-Lin), A. X. WANG (Wang Ai-Xia), Z. J. JIA (Jia Zhong-Jian)* (*College of Chemistry and Chemical Engineering, National Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou, Gansu 730000 People's Republic of China; Jiazj@lzu.edu.cn): Three new polymeric isoprenyl benzofurans from Ligularia stenocephala. Pharmazie 60, 155-159 (2005). Preparative TLC of stenocephalin A, stenocephalin B, and 5,6-dimethoxy-2-isopropenylbenzofuran on silica gel with petroleum ether - acetone 5:2, and chloroform - acetone 40:1 and 20:1. Detection by spraying with 5 % sulfuric acid in ethanol followed by heating. Evaluation under UV light.

traditional medicine herbal, qualitative identification, preparative TLC 32e

99 129 X. LI (Li Xin), J. PAN (Pan Jing), K. GAO (Gao Kun)* (*State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou, 730000, P. R. China; npchem@lzu.edu.cn): y-Pyranone derivatives abd other constituents from Erigeron annuus. Pharmazie 61, 474-477 (2006). Analytical and preparative TLC of 3-hydroxyl-y-pyranones (3-O-beta-D-(6'-O-linolenic)glucopyranosyl-y-pyranone and erigeside) on silica gel using chloroform - methanol 8:1 and ethyl acetate - methanol - water 12:2:1. Detection under UV light at 254 nm or by spraying with 5 % sulfuric acid in ethanol followed by heating.

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

99 130 J. LI* (Li Juan), X. HUANG (Huang Xiaodan), B. LU (Lu Bingwan), Y. XIAN (Xian Yanfang), J. CHEN (Chen Jiannan) (*Guangzhou Univ. TCM, Guangzhou 510405, China): (Determination of taurine in preparation of Hedan pills by thin-layer chromatography) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao), 29 (2), suppl. 1-3 (2007). TLC of taurine in Hedan pills on silica gel with 1) n-butanol - nitrile 20:1; and 2) chloroform - methanol 10:1. Detection 1) by spraying with 5 % vanillin in H_2SO_4 and heating at 105°C. Identification by fingerprint techniques.

pharmaceutical research, quality control, traditional medicine, quantitative analysis, HPTLC, puerarin 32c

99 131 Hanna LISKIEWICS*, M. W. KOWALSKA, M. RUTKOWSKA, H. GLINIAK (*Department of Drugs Technology, Wroclaw Medical University, Nankiera 1 SQ. 50-140 Wroclaw, Poland; hanna@bf.uni.wroc.pl): Synthesis and anxiolytic activity of 1-phenyl-2-(4-aryl-1,3,4,5-tetrahy-

dropyrido[2,3-b][1,4]diazepin-2-ylidene)-ethanone. Pharmazie 61, 517-521 (2006). TLC of 1-phenyl-2-(4-aryl-1,3,4,5-tetrahydropyrido[2,3-b][1,4]diazepin-2-ylidene)-ethanone on silica gel with diethyl ether - ethanol 5:1; detection under UV light.

pharmaceutical research, organic synthesis 32a

99 132 M. M. BAING*, V. V. VAIDYA, P. A. CHAMPANERKAR, W. SHAH (*Dept. of Chemistry, S.P. Mandali's Ramnarain Ruia College, Matunga, mumbai 400019, vaidya_vikas@yahoo.com): Simultaneous HPTLC determination of Frusemide and Spironolactone from pharmaceutical formulation. Indian Drugs 44 (3), 205-208 (2007). HPTLC of frusemide (= furosemide) and spironolactone on silica gel with toluene - acetonitrile - glacial acetic acid 70:30:2, with chamber saturation for 15 min at room temperature. Development over 8 cm, followed by air drying. Quantitative determination by densitometry at 254 nm. Linearity was between 8 - 32 ng/µL and 20 - 80 ng/µL for frusemide and spironolactone respectively. The method was validated for accuracy and precision. The limit of detection and quantification for frusemide was 3 ng/µL and 8 ng/µL respectively, and for spironolactone 2 ng/µL and 6 ng/µL, respectively. Recovery by standard addition was 99.4-101% for both compounds.

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

99 133 K. M. ROSENBLATT, H. BUNJES, A, SEELING, H, OELSCHLAEGER* (*Institute of Pharmacy, Philosophenweg 13, D-07743 Jena, Germany): Investigations on the thermal behavior of omeprazole and other sulfoxides. Pharmazie 60, 503-507 (2005). TLC of omeprazole (5-methoxy-2-[[(4-methoxy-3,5-dimethyl-2-pyridinyl)methyl]sulfinyl]-1H-benzimidazole) and more than 30 degradation products on silica gel with ethyl acetate. Evaluation in white light and under UV 254 and 366 nm.

quality control, qualitative identification 32a

99 134 I. MASTEROVÁ, D. GRANCAI*, Z. GRANCAIOVÁ, M. POUR, K. UBIK (*Department of Pharmacognosy and Botany, Faculty of Pharmacy, Odbojarov 10, 832 32 Bratislava, Slovak Republic; grancai@fpharm.uniba.sk): A new flavonoid: tinctosid from Anthemis tinctoria L. Pharmazie 60, 956-957 (2005). TLC of caffeic acid, patuletin, and patulitrin on silica gel by two fold development with benzene - ethanol - acetone 7:2:1; and ethyl acetate - iso-propanol - n-butanol - acetic acid - water 50:30:17.5:17.5:15 for sugars. Detection under UV 254 and 366 nm and by spraying with natural products reagent (for flavonoids) and p-anisidine followed by heating for 5 min (for sugars).

herbal, qualitative identification 32e

99 135 S. MENNICKENT*, L. PINO, M. VEGA, C. GODOY, M. DIEGO (*Department of Pharmacy, Faculty of Pharmacy, University of Concepción, Concepción, Chile, smennick@udec.cl): Quantitative determination of haloperidol in tablets by high performance thin-layer chromatography. J. Sep. Sci. 30, 772-777 (2007). HPTLC of haloperidol in tablets on silica gel with acetone - chloroform - n-butanol - acetic acid - water 2:4:4:1:1. Quantitative determination by absorbance measurement at 254 nm. Linearity was between 10 and 100 ng/µL, detection limit was 0.89 ng/µL, and the quantification limit was 2.71 ng/µL. Coefficient of variation is 2.35% and 4.50% for precision and accuracy, respectively. Successful comparison with HPLC measurements.

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

99 136 A. MIRZAIE*, A. JAMSHIDI, S. WAQIF-HUSAIN (Faculty of Food Science and Technology, Science and Research Branch, Islamic Azad University, P.O. Box 14515-775, Tehran, Iran): Fast Chromatographic Separation of Plasticizers on Thin Layers of an Inorganic Ion-Exchanger:

Quantitative Determination of Di(2-ethylhexyl)phthalate. Chromatographia 65 (3-4), 245-248 (2007). TLC of dimethyl phthalate, diethyl phthalate, dibutyl phthalate, di(2-ethylhexyl)phthalate (DEHP), benzyl butyl phthalate, diisodecyl phthalate, dimethyl adipate, diethyl adipate, di(2-ethylhexyl)adipate, triethyl citrate, tributyl citrate, tributyl acetyl citrate and n-butyl stearate on inorganic ion-exchanger stannic silicate with toluene - ethyl acetate 10:1 over 12 cm (25 min). Quantification of DEHP by densitometry at 280 nm. Limit of quantitation for DEHP was 500 ng/zone and limit of detection 50 ng/zone.

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

99 137 N. MISHRA, A. P. GUPTA, B. SINGH, V. K. KAUL*, P. S. AHUJA (*Department of Natural Plant Products , Institute of Himalayan Bioresource Technology, Box No. 6, Palampur 176061 (HP), India; vkaul2002@yahoo.co.in): A rapid determination of podophyllotoxin in Podophyllum hexandrum by reverse phase High Performance Thin Layer Chromatography. J. Liq. Chromatogr. Relat. Technol. 28, 677-691 (2005). HPTLC of podophyllotoxin and podophyllin on RP-18 in a twin trough chamber with acetonitrile - water 2:3. Densitometric measurement of lignans in absorption mode at 217 nm.

HPTLC, quantitative analysis 32e

99 138 H. MOEHRLE*, C. ROHN, G. WESTLE (*Institut für Pharmazeutische Chemie, Universitätsstrasse 1, D-40225 Düsseldorf, Germany; h.moehrle@uni-duesseldorf.de): Indolspaltung bei Mebhydrolin durch Natriumperjodat - 2. Mitt. Mechanismus der Dilactam-Bildung. / Indole cleavage with mebhydroline by sodium periodate - Part 2. Mechanism of the dilactam formation (German). Pharmazie 61, 391-399 (2006). TLC of 5-benzyl-2,3,4,5-tetrahydro-2-dimethyl-1H-pyrido[4,3-b]indol-2-ium-bromid, 2,5-dimethyl-2,3,4,5-tetrahydro-1H-pyrido[4,3-b]indol and 15 other compounds on alumina with chloroform - ethanol- ammonia 13:6:1; and benzene - ethyl acetate 3:2. Also TLC on silica gel with chloroform - acetone - ethanol - ammonia 90:10:10:1, diisopropyl ether, and chloroform - ethyl acetate 7:3. Detection under UV light at 254 and 366 nm, Dragendorff reagent (and spraying with 10% sulfuric acid), and Ehrlich reagent.

pharmaceutical research, qualitative identification 32a

99 139 M. MONFORTE-GONZÁLES, F. MEDINA-LARA, G. GUTIÉRREZ-CARBAJAL, F. VÁZQUEZ-FLOTA* (* nidad de Bioquímica y Biología Molecular de Plantas, Centro de Investigación Científica de Yucatán, Calle 43 No.130 Chuburná 97200, Mérida Yucatán México; felipe@cicy.mx): Capsaicinoid quantitation by in situ densitometry of Thin Layer Chromatography plates. J. Liq. Chromatogr. & Relat. Technol. 30, 1697-1704 (2007). TLC of capsaicinoids from chili peppers with e. g. capsaicin, dihydrocapsaicin, coumaric acid, vanillin, ferulic acid, and cinnamic acid as standards, on silica gel by two fold development with cyclohexane - chloroform - acetic acid 7:2:1; chloroform - methanol - acetic acid 95:1:5; and cyclohexane - acetone 4:5. Visualization under UV light at 254 nm. Quantitation by densitometry at 254 nm.

herbal, food analysis, quantitative analysis, densitometry 32e

99 140 B. MORAK, M. NOWAK, Krystina PLUTA* (*Department of Organic Chemistry, The Medical University of Silesia, Jagiellonska 4, 41-200, Sosnowiec, Poland; pluta@slam.katowice.pl): Determination of the lipophilicity parameters R(MO) and Log P of new azaphenothiazines by reversed-phase Thin-Layer Chromatography. J. Liq. Chromatogr. & Relat. Technol. 30, 1845-1854 (2007). TLC of three types of azaphenothiazines (10H- and 10-alkyldipyrido-1,4-thiazines, 6H- and 6-alkyldiquino-1,4-thiazines and 14H- and 14-alkyldiquino-1,4-thiazines) on RP-18 with acetone (concentration ranged from 50 to 85 % in 5 % increments) and aqueous Tris buffer pH 7.4, with chamber saturation. Detection under UV 254 nm.

pharmaceutical research, qualitative identification 32a

99 141 M. MORSCH, L. G. J. GIRARDI, V. CECHINEL-FILHO, C. MEYRE-SILVA, C. A. RODRIGUES* (*Núcleo de Investigacoes Químico-Farmaceuticas (NIQFAR), Curso de Farmácia/CCS, Universidade de Vale do Itajaí (UNIVALI), CEP 88.302-202, Itajaí, SC, Brasil; crodrigues@univali.br): The use of chitosan modified with glutaraldehyde and glyoxal as chromatographic support for the separation of flavonoids from Aleurites moluccana leaves. Pharmazie 61, 670-672 (2006). TLC of swertisin and 2”-O-rhamnosylswertisin on silica gel with chloroform - methanol 7:3 and 17:3. Detection under UV light at 254 nm or by spraying with 2 % iron(III) chloride solution in ethanol. The compounds were identified by direct comparison with authentic samples.

herbal, traditional medicine, qualitative identification 32e

99 142 K. NU* (Nu Kewen), J. ZHAO (Zhao Jianping), Y. MENG (Meng Youchu), ZH. LIANG (Liang Zhuli) (*Guangxi Nafang Wanshida Pharm. Com., Guangxi Nanning 530003, China).: (Method improving for TLC identification and determination of Liandan Xiaoyanpian tablets) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 29 (3), suppl. 3-4 (2007). TLC of Liandan Xiaoyanpian tablet extracts on silica gel with 1) toluene - chloroform - acetone 4:4:1; and 2) chloroform - methanol - ammonia 36:4:1. Detection 1) by spraying with 10 % H_2SO_4 in ethanol and heating at 105 °C.

pharmaceutical research, quality control, traditional medicine, quantitative analysis, HPTLC 32c

99 143 J. P. FAN (Fan Jie-Ping), C. H. HE (He Chao-Hong)* (*Department of Chemical Engineering, Zhejiang University, Hangzhou 310027, P. R. China; chhezju@zju.edu.cn).: Single-step preparative separation of barbinervic acid and its epimer (rotungenic acid), along with two other pentacyclic triterpene acids from the leaves of Diospyros kaki using HSCCC. J. Liq. Chromatogr. Relat. Technol. 29, 815-827 (2006). TLC of barbinervic acid, rotungenic acid, 24-hydroxy ursolic acid, and ursolic acid on silica gel with n-hexane - acetone - ethyl acetate 4:2:1. Detection under UV light.

qualitative identification 32e

99 144 B. PATEL*, M. PATEL, J. PATEL, B. SUHAGIA (*B-16, Surjit Soc., NR, Hari OM Soc., India Colony, Bapunagar, Ahmedabad, Gujarat-380 024, India; bhpmph@yahoo.co.in).: Simultaneous determination of omeprazole and domperidone in capsules by RP-HPLC and densitometric HPTLC. J. Liq. Chromatogr. & Relat. Technol. 30, 1749-1762 (2007). HPTLC of omeprazole (5-methoxy-2-[[(4-methoxy-3,5-dimethyl-2-pyridinyl)methyl]sulfinyl]-1H-benzimidazole) and domperidone (5-chloro-1-[1-[3-(2,3-dihydro-2-oxo-1H-benzimidazol-1-yl)propyl]-4-piperidinyl]-1,3-dihydro-2H-benzimidazol-2-one) on silica gel with ethyl acetate - methanol - benzene 2:1:2. Quantitative determination by absorbance measurement at 295 nm.

quality control, quantitative analysis, densitometry, HPTLC 32a

99 145 V. PATHANIA, A. P. GUPTA, B. SINGH* (*Division of Natural Plant Products, Institute of Himalayan Bioresource Technology, P. Box No. 6, Palampur 176 061 (HP), India; bikram_npp@rediffmail.com). : Improved HPTLC method for determination of curcuminoids from Curcuma longa. J. Liq. Chromatogr. Relat. Technol. 29, 877-887 (2006). HPTLC of curcuminoids (curcumin, demethoxycurcumin, and bis-demethoxycurcumin) on spherical silica gel with chloroform - methanol 49:1 with chamber saturation. Densitometry at 366 nm in adsorption-reflection mode.

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

99 146 O. POZHARITSKAYA, S. IVANOVA, A. SHIKOV*, V. MAKAROV (*Interregional Center “Ad-

aptogen", Piskarevsky prosp., St.-Petersburg, Russia, alexs79@mail.ru).: Separation and evaluation of free radical-scavenging activity of phenol components of Emblica officinalis extract by using an HPTLC-DPPH method. J. Sep. Sci. 30, 1250-1254 (2007). HPTLC of Emblica officinalis extract on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 10:1:1:2. Primary detection under UV 280 nm. Antiradical activity of individual components was estimated on intensity of disappearance of violet/purple background of plate after dipping in DPPH* solution (0.5 mM in methanol) at room temperature for 90 s and that 30 s at 60 °C. Quantitative determination by absorbance measurement at 517 nm as negative peak. DPPH* scavenging activity of emblicanins A and B was 7.9 and 11.2 times more active than that of ascorbic acid and 1.3 and 1.8 times more active than gallic acid, respectively.

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

99 147 O. POZHARITSKAYA, V. KOSMAN, A. SHIKOV*, D. DEMCHENKO, A. ESCHENKO, V. MAKAROV (*Interregional Center "Adaptogen", St. Petersburg, Russia, alexs79@mail.ru).: Comparison between HPLC and HPTLC densitometry for the determination of icariin from Epimedium koreanum extracts. J. Sep. Sci. 30, 708-712 (2007). HPTLC of icariin in the aerial part of Epimedium koreanum Nakai on silica gel with ethyl acetate - glacial acetic acid - formic acid - water 10:1:1:2. Quantitative determination by absorbance measurement at 270 nm. The LOD and LOQ for icariin were 66 and 215 ng/band, respectively. Results did not show statistical significance between HPLC and HPTLC.

herbal, HPTLC, quantitative analysis, densitometry, comparison of methods 32e

99 148 Alina PYKA*, M. BABUSKA, A. DZIADEK, D. GURAK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, PL-41-200, Sosnowiec, Poland; alinapyka@wp.pl or apyka@slam.katowice.pl).: Comparison of spectrodensitograms of the selected drugs on different chromatographic sorbents. J. Liq. Chromatogr. & Relat. Technol. 30, 1385-1400 (2007). TLC of alpha-tocopherol acetate, alpha-tocopherol, cholecalciferol, estradiol, testosterone, and hydrocortisone on silica gel, silica gel and kieselguhr, and aluminium oxide after prewashing with methanol; TLC of lipophilic vitamins on silica gel with toluene, and on RP-18 with methanol both with chamber saturation. Densitometric measurement at UV 254 nm. The resulting densitograms of the compounds studied indicate that applied sorbents have an influence on the wavelength of the obtained fundamental absorption band and the additional absorption bands, as well as on their intensity values.

pharmaceutical research, densitometry, quantitative analysis 32a

99 149 S. Q. DE OLIVEIRA, G. BARBON, Grace GOSMANN*, S. BORDIGNON (*Faculdade de Farmácia, Universidade Federal do Rio Grande do Sul (UFRGS), Av. Ipiranga, 2752 , Porto Alegre, RS 90610-000, Brazil; grace.gosmann@ufrgs.br). : Differentiation of South Brazilian Baccharis species by TLC. J. Liq. Chromatogr. & Relat. Technol. 29, 2603-2609 (2006). TLC of phenolic and terpenoid compounds with 4'-O-beta-D-glucopyranosyl-3',5'-dimethoxybenzyl-caffeate as standard on silica gel with chloroform - ethanol - acetic acid 30:20:3. Detection by spraying with anisaldehyde - sulfuric acid reagent and heating to 100 °C, and 1% methanolic diphenylboryloxyethylamine, followed by PEG 400. Evaluation under visible and UV light at 366 nm.

herbal, traditional medicine, qualitative identification 32e

99 150 G. R. SINGH*, V. V. VAIDYA, S. SHAILAJAN, M. M. BAING, P. A. CHAMPANERKAR (Analytical Chemistry Laboratory, S. P. mandali's, Ramnarain Ruia College, Matunga, Mumbai 400019, vaidya_vikas@yahoo.com): Quantity of lupeol in vernonia cinereal whole plant powder by high performance thin-layer chromatography. Indian Drugs 43 (12), 989-982 (2006). HPTLC of lupeol in a methanolic extract of powdered Vernonia cinerea L. on silica gel with dichlorome-

thane - toluene - acetone - methanol 30:50:5:3. Detection by spraying with anisaldehyde reagent. Quantitative determination by absorbance measurement at 581 nm. Lupeol response was linear over the range 50 µg/mL. The concentration of lupeol in Vernonia cinerea L. was found to be 2.01 µg. The method was validated and can be used for routine quality control of Vernonia cinerea L. including quantitation of lupeol.

pharmaceutical research, traditional medicine, herbal, HPTLC, quantitative analysis, densitometry 32a

99 015 E. REICH et al., see section 8

99 151 A. RUEBE, S. KLEIN, K. MAEDER* (*Department of Pharmacy, Institute of Pharmaceutics and Biopharmaceutics, Martin Luther University Halle-Wittenberg, Halle/Saale, Germany, maeder@pharmazie.uni-halle.de).: Monitoring of in vitro fat digestion by electron paramagnetic resonance spectroscopy. Pharm. Res. 23, 2024-2029 (2006). HPTLC of the chloroform extract of a lipophilic model drug (tempol benzoate) into a long-chain triacylglyceride (olive oil) after 0, 5, 20 and 45 min of digestion with pancreatin on silica gel, by AMD using an 11 step gradient based on hexane and ethyl acetate. Detection by spraying with an aqueous copper sulfate solution (10 % copper sulfate, 8 % phosphoric acid, 5 % methanol), followed by heating at 150 °C. Quantitative determination by absorbance measurement at 675 nm. Lipid recovery was between 104 and 119 %.

pharmaceutical research, HPTLC, densitometry, AMD 32b

99 152 M. S. Y. KHAN*, M. AKHTER (*Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Jamia Hamdard (Hamdard University), New Delhi, 110 062 India; msykhan@hotmail.com).: Glyceride derivatives as potential prodrugs: synthesis, biological activity, and kinetic studies of glyceride derivatives of mefenamic acid. Pharmazie 60, 110-114 (2005). TLC of two glyceride derivatives of mefenamic acid (“3a and 3b”) on silica gel with hexane - ethyl acetate 5:1. Detection by exposure to iodine vapors.

pharmaceutical research, qualitative identification 32a

99 153 M. SAJEWICZ, R. PIETKA, A. PIENIAK, Teresa KOWALSKA* (*Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland; kowalska@us.edu.pl).: Application of Thin-Layer Chromatography to the investigation of oscillatory instability of selected profen enantiomers in physiological salt. J. Liq. Chromatogr. Relat. Technol. 29, 2059-2069 (2006). TLC of S-(+)-ibuprofen, S-(+)-naproxen, and R,S-(+/-)-2-phenylpropionic acid on silica gel (prewashed with methanol - water 9:1 and impregnated with a 0.03 mol/L solution of L-arginine in methanol by dipping for 2 s at 22 +/- 2 °C) with acetonitrile - methanol - water 10:2:3 for naproxen and 20:4:3 for 2-phenylpropionic acid. Both mobile phases contained several drops of acetic acid to fix the pH at 4.8. Densitometric evaluation at 210 nm.

densitometry 32a

99 154 M. SAJEWICZ, R. PIETKA, G. DRABIK, Teresa KOWALSKA* (*Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland; kowalska@us.edu.pl).: On the mechanism of oscillatory changes of the retardation factor (RF) and the specific rotation [alpha]D with selected solutions of S-(+)-naproxen. J. Liq. Chromatogr. Relat. Technol. 29, 2071-2082 (2006). TLC of S-(+)-naproxen on silica gel (prewashed with methanol - water 9:1 and impregnated with a 0.03 mol/L solution of L-arginine in methanol by dipping for 2 s at 22 +/- 2 °C) with acetonitrile - methanol - water 5:1:1.5 containing several drops of acetic acid to fix the pH at 4.8; and two-dimensional development with acetontrile - methanol - water 10:2:3. Densitometric evaluation at 235 nm.

densitometry 32a

99 155 C. SANTOS ROSA, M. D. GARCÍA GIMENEZ, M. T. SAENZ RODRIGUEZ, R. De LA PUERTA VAZQUEZ* (*Pharmacology Department, Faculty of Pharmacy, University of Seville, Spain. C/Profesor García Gonzales n° 2, 41012-Sevilla, Espana; puerta@us.es).: Antihistaminic and antieicosanoid effects of oleanolic and ursolic acid fraction from Helichrysum picardii. Pharmazie 62, 459-462 (2007). TLC of oleanolic and ursolic acid on silica gel with n-hexane - diethyl ether 7:3; or dichloromethane - ethyl acetate 7:3. Detection with oleum reagent.

herbal, qualitative identification 32e

99 156 K. SCHAEFER, P. WINTERHALTER* (*Institute of Food Chemistry, Technical University of Braunschweig, Schleinitzstrasse 20, D-38106, Braunschweig, Germany; p.winterhalter@tu-bs.de).: Application of high speed countercurrent chromatography (HSCCC) to the isolation of kavalactones. J. Liq. Chromatogr. Relat. Technol. 28, 1703-1716 (2005). TLC of kavalactones (kavain, 7,8-dihydrokavain, methysticin, 7,8-dihydromethysticin, yangonin, and demethoxyyangonin) on silica gel with the organic layer of n-hexane - ethyl acetate - methanol - water 6:5:6:5. Detection by spraying with anisaldehyde - sulfuric acid followed by heating.

herbal, food analysis, qualitative identification 32e

99 157 M. SCHMIDT, F. BRACHER* (*Department Pharmazie - Zentrum für Pharmaforschung, Ludwigs-Maximilians-Universität München, Butenandtstr. 5-13, D-81377 München, Germany; Franz.Bracher@cup.uni-muenchen.de).: A convenient TLC method for the identification of local anesthetics. Pharmazie 61, 15-17 (2006). TLC of seven local anesthetics (benzocaine, procaine, tetracaine, lidocaine, prilocaine, bupivacaine, articaine) and the related antiarrhythmic drug procainamide on silica gel with ethyl acetate - methanol - 32 % ammonia 96:2:3 with chamber saturation for 15 min. Detection a) under UV light at 254 nm; b) spraying with cobalt(II) thiocyanate solution; c) by subsequent spraying with Ehrlich's reagent. Except for articaine/prilocaine all drugs could be distinguished. However, articaine could be distinguished from prilocaine and other local anesthetics by a colour reaction with copper(II) sulfate solution.

toxicology, qualitative identification 32c

99 158 O. SHIROTA*, K. NAGAMATSU, S. SEKITA (*Laboratory of Pharmacognosy and Natural Products Chemistry, Faculty of Pharmaceutical Sciences at Kagawa Campus, Tokushima Bunri University, 1314-1 Shido, Kagawa, 769-2193, Japan; shirota@kph.bunri-u.ac.jp).: Simple preparative isolation of salvinorin A from the hallucinogenic sage, Salvia divinorum, by centrifugal partition chromatography. J. Liq. Chromatogr. & Relat. Technol. 30, 1105-1114 (2007). TLC of salvinorin A, a potent naturally occuring kappa-opioid selective agonist, on silica gel and on RP-18 with n-hexane - ethyl acetate 1:1. Detection by spraying with vanillin - phosphoric acid reagent followed by heating.

traditional medicine, herbal, toxicology, qualitative identification 32e

99 159 N. SINGH, A. P. GUPTA, B. SINGH, V. K. KAUL* (*Department of Natural Plant Products, Institute of Himalayan Bioresource Technology, P. O. Box No. 6, Palampur 176061 (HP), India; vkaul2002@yahoo.co.in). : Quantification of picroside-I and picroside II in Picrorhiza kurroa by HPTLC. J. Liq. Chromatogr. Relat. Technol. 28, 1679-1691 (2005). HPTLC of the iridoid gycoside picroside I and picroside II on silica gel with chloroform - methanol 41:9 in a saturated twin-trough chamber. Quantitation by absorbance measurement at 290 nm.

herbal, traditional medicine, qualitative identification, quantitative analysis, HPTLC 32e

99 160 R. SKIBINSKI, Genowefa MISZTAL* (*Department of Medicinal Chemistry, Medical University of Lublin, 6 Chodzki Str, 20-093, Lublin, Poland; kzchl@asklepios.am.lublin.pl).: Determination of citalopram in tablets by HPLC, densitometric HPTLC, and videodensitometric HPTLC methods. J. Liq. Chromatogr. Relat. Technol. 28, 313-324 (2005). HPTLC of citalopram (1-[3-

(dimethylamino)propyl]-1-(4-fluorophenyl)-1,3-dihydro-5-isobenzofurancarbonitrile) and moclobemide (as standard) on silica gel with benzene - acetone - ethanol - 25 % ammonia 9:8:2:1. Quantitative determination by densitometry at 226 nm and by video densitometry at UV 254 nm.

quality control, densitometry, quantitative analysis, HPTLC 32a

99 161 J. SMITH*, D. TUCKER, K. WATSON, G. JONES (*School of Biological, Biomedical and Molecular Sciences, University of New England, Armidale NSW 2351, Australia, jsmith38@une.edu.au).: Identification of antibacterial constituents from the indigenous Australian medicinal plant Eremophila duttonii F. Muell. (Myoporaceae). J. Ethnopharmacol. 112, 386-393 (2007). TLC of two serrulatane diterpenes in aerial parts of Eremophila duttonii F. Muell. (Myoporaceae) with ethyl acetate - hexane 3:1. Detection by spraying with 0.5 % p-anisaldehyde in 5 % sulphuric acid, and 5 % glacial acetic acid in methanol. Bioautography over developed plates to examine regions of growth inhibition. Structures of separated compounds with antibacterial activity against Staphylococcus aureus were identified by NMR spectroscopy as: serrulat-14-en-7,8,20-triol (hRf 57) and serrulat-14-en-3,7,8,20-tetraol (hRf 31).

traditional medicine, herbal, qualitative identification 32e

99 162 G. SONG (Song Guangda), J. PAN (Pan Jinhuo), Y. YUAN (Yuan Yanfang), (Nanjing Univ. TCM, Coll. Pharm., Nanjing 210029, China): (Study of the quality standard for Huoxue zhitong Babu ointment) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao), 29 (2), 235-237 (2007) TLC of the extracts of the title Chinese traditional patent medicine on silica gel with 1) toluene - ethyl acetate 15:1; 2) chloroform - methanol - concentrated ammonia water 20:5:0.5. Detection 1) under UV 365 nm; 2) 5% nynhydrin solution and heating at 105 ºC till the spots visualized. Identification by standard comparison, also by GC fingerprint techniques. Quantification of strychnine by HPLC. Discussion of application of the procedures for the quality control of the medicine.

pharmaceutical research, traditional medicine, quality control, HPTLC, quantitative analysis 2c, 4d

99 163 D. SRIRAM*, P. YOGEESWARI, K. MEENA (*Medicinal Chemistry Research Laboratory, Pharmacy Group, Birla Institute of Technology and Science, Pilani - 333031, India; dsriram@bits-pilani.ac.in).: Synthesis, anti-HIV and antitubercular activities of isatin derivatives. Pharmazie 61, 274-277 (2006). TLC of twelve isatin analogues (derivatives of 3-{[(4,6-dimethylpyrimidin-2-yl)benzenesulfonamido-4'-yl]imino}-5-fluoro-1,3-dihydro-2H-indol-2-one) on silica gel with chloroform - methanol 9:1. Visualization by iodine vapor.

pharmaceutical research, qualitative identification 32a

99 164 B. TIPERCIUC, C. SARBU* (*Babes-Bolyai University, Faculty of Chemistry and Chemical Engineering, Arany Janos 11, 400028 Cluj-Napoca, Romania; csarbu@chem.ubbcluj.ro).: Prediction of the chromatographic retention (lipophilicity) of some new methyl-thiazole-oxadiazoline derivatives by multivariate regression methods. J. Liq. Chromatogr. & Relat. Technol. 29, 2257-2270 (2006). HPTLC of 20 methyl-thiazole-oxadiazoline derivatives on RP 18 with mixtures of methanol - water with varying contents from 45 - 70 % in 5 % steps. Examination after drying under UV light at 254 nm.

pharmaceutical research, qualitative identification, HPTLC 32a

99 165 V. V. DIGHE*, R. T. SANE, S. MENON, V. G. GOKARN, A. A. GURSALE (*TDM Laboratory, Plot No.194, Scheme no.6, Road No.15, Sion(E), Koliwada, Mumbai 400022, vijay.g12@rediffmail.com).: High Performance Thin Layer Chromatographic Method for quantitative determination of rutine in leaf powder of morus Alba Linn. Indian Drugs 44 (2), 117-121 (2007). HPTLC of rutine in methanolic extracts of powdered and dried Morus alba Linn. leaves, on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 16:1:1:2. Detection and quantification of rutine by densitometric scanning at 254 nm. The method was validated for its preci-

sion. The accuracy of the method was checked by conducting recovery studies at three different levels of rutin and the average percentage recovery of rutin was found to be 96.9%. The proposed HPTLC method provided a good resolution of rutine from other constituents and can be used for quantitation of rutine present in the leaves of Morus alba Linn.

pharmaceutical research, traditional medicine, HPTLC, densitometry, quantitative analysis 32a

99 166 D. V. MHASKE, D. S. R. DHANESHWAR* (* Department of Quality Assurance Techniques and Pharm. Chem., Bharati Vidyapeeth University, Centre for Advanced Pharmaceutical Research, Erandwane, Pune, 411038, Maharashtra, India).: Novel TLC Densitometric Method for Quantification Of Solasodine in Various Solanum Species, Market Samples and Stability Indicating HPTLC and LC Determination of Dasatinib in Pharmaceutical Dosage Formulations. Chromatographia, 66 (1-2), 95-102 (2007). Description of two sensitive and reproducible methods for the quantitative determination of dasatinib in the presence of its degradation products. HPTLC on silica gel with toluene - chloroform 7:3, followed by densitometric measurement at 280 nm. Validation of both separation methods according to ICH guidelines, no interference from the tablet excipients was found. Discussion of application of the methods as the stability indication because the proposed analytical methods could effectively separate the drug from its degradation products, which was subjected to acid-alkali hydrolysis, oxidation, dry heat, wet heat and photodegradation.

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

99 167 G. WU (Wu Gang), D. Q. FEI (Fei Dong-Qing), K. GAO (Gao Kun)* (*State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, P. R. China; npchem@lzu.edu.cn).: Aromadendrane-type sesquiterpene derivatives and other constituents from Erigeron acer. Pharmazie 62, 312-315 (2007). Analytical and preparative TLC of 4alpha,10beta-alloaromadendranediol, 4beta,10beta-aromadendranediol, ent-manool-13-O-alpha-L-4'-acetylarabinopyranoside, and ergost-6,22-diene-5alpha,8alpha-epidioxy-3beta-ol on silica gel with choroform - acetone 10:1, 3:1, and 2:1. Detection under UV 254 nm or by spraying with 5 % sulfuric acid in ethanol followed by heating.

preparative TLC, qualitative identification 32e

99 168 C. X. YANG (Yang Cai-Xia), Q. ZHANG (Zhang Qi), Z. J. JIA (Jia Zhong-Jian)* (*Department of Chemistry, Lanzhou University, Lanzhou, Gansu 730000, P. R. China; jiazj@lzu.edu.cn).: Diterpene glycosides from Aster homochlamydeus. Pharmazie 60, 461-463 (2005). Preparative TLC of ent-manool-13-O-beta-D-4'-acetylxylopyranoside and ent-manool-13-O-beta-D-3'-actylxylopyranoside on silica gel by three fold development with chloroform - ethyl acetate 30:1. Detection by spraying with 5 % sulfuric acid in ethanol or 5 % iron(III) chloride in ethanol, followed by heating. Evaluation under UV light.

traditional medicine, herbal, preparative TLC 32e

99 169 M. YANG (Yang Min), J. X. LI (Li Ji-Xin), X. Li (Li Xin), Z. J. JIA (Jia Zhong-Jian)* (*College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou, Gansu 730000, P. R. China; jiazj@lzu.edu.cn).: Sesquiterpenes and other constituents from Achillea wilsoniana. Pharmazie 60, 554-558 (2005). TLC of three new compounds (4E,10E-9beta-hydroxy-3-(2-methylbutyroyloxy)germacra-4,10(1)-diene-12,6alpha-olide, 4E,10E-3-(2-methylbutyroyloxy)germacra-4,10(1)-diene-12,6alpha-olide, and 1beta,6alpha-dihydroxy-10beta-methyl-5alphaH,7alphaH-eudesm-4-one and numerous known compounds on silica gel with petroleum ether (60-90°C) - ethyl acetate 2:1; and 5:1; and petroleum ether (60-90°C) - acetone 6:1. Detection under UV light or by spraying with 5 % sulfuric acid in ethanol followed by heating.

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

99 170 J. YU* (Yu Jiaqi), Z. YANG (Yang Zhonglan), H. JIAN (Jian Hongjun), Y. ZHANG (Zhang Yongping), L WEI (Wei Ling) (*Guizhou Zunyi Hosp., Zhunyi, Guizhou 563000, China).: (Quality control of Shajun Zhiyang lotion) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao), 27 (8), 900 - 903 (2005). TLC of Shajun Zhiyang lotion on silica gel with 1) benzene - ethyl acetate - isopropanol - methanol - ammonia 12:6:3:3:1; 2) benzene - ethyl acetate 30:1; 3) ethyl acetate. Detection 1) under UV 365 nm; 2) by spraying with 5% vanillin in H_2SO_4 solution and heating at 105 °C. Identification by fingerprint techniques. Quantification of matrine by HPLC.

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

33. Inorganic substances

99 171 M. Loredana SORAN*, M. CURTUI, C. MARUTOIU (*National Institute of Research and Development for Isotopic and Molecular Technology, 72-103 Donath Street, RO-400293 Cluj-Napoca, Romania; loredana-soran@yahoo.com).: Separation of U(VI) and Th(IV) from some rare earths by Thin Layer Chromatography with di-(2-ethylhexyl)-dithiophosphoric acid on silica gel. J. Liq. Chromatogr. Relat. Technol. 28, 2515-2524 (2005). TLC of U(VI), Th(IV), and some rare earths on silica gel impregnated by development with 2.5 M aqueous ammonium nitrate solution with ethyl methylketone - tetrahydrofuran 2:1 containing 1 M di-(2-ethylhexyl)-dithiophosphoric acid in unsaturated chambers. Detection by spraying with 0.05 % aqueous Arsenazo III solution.

qualitative identification 33a

37. Environmental analysis

99 172 M. Y. Z. ABOUL EISH, MARTHA J. M. WELLS* (*Center for the Management, Utilization, and Protection of Water Resources, and Department of Chemistry, Tennessee Technological University, Box 5033, Cookeville, TN 38505, USA).: Assessing the trihalomethane formation potential of aquatic fulvic and humic acids fractionated using thin-layer chromatography. J. Chromatogr. A 1116 (1-2), 272-276 (2006). Application of TLC to fractionate well-characterized aquatic humic materials coupled with the novel evaluation of the trihalomethane formation potential of the fractionated materials. HPTLC on silica gel with methanol - ethyl acetate 2:1. Identification of three common fractions based on retention factor (Rf) in all substances examined.

qualitative identification, HPTLC, disinfection by-products, drinking water treatment 37c

38. Chiral separation

99 173 M. SAJEWICZ, R. PIETKA, Teresa KOWALSKA* (*Institute of Chemistry, Silesian University, 9 Szkolna Street, Katowice, Poland; kowalska@us.edu.pl): Chiral Separations of ibuprofen and propanolol by TLC. A study of the mechanism and thermodynamics of retention. J. Liq. Chromatogr. Relat. Technol. 28, 2499-2513 (2005). TLC of R,S-(+/-)-ibuprofen and S-(+)-ibuprofen on silica gel prewashed with methanol - water 9:1 and impregnated with a 0.03 mol/L methanolic solution of L-arginine by dipping. Separation with acetonitrile - methanol - water 5:1:1 and several drops of acetic acid to adjust the pH to 4.8. Two dimensional development with the same mobile phase in the first direction, followed by drying and application of the S-(+)-enantiomer and development in the second direction. Densitometric evaluation at 210 nm. Chiral separation of propanolol with acetonitrile - methanol 15:4 containing ammonia for one and two dimensional separation.

qualitative identification 38

International Symposium for High Performance Thin-Layer Chromatography Helsinki, 11th–13th June 2008

City of Helsinki, Picture Bank/Photo Niko Soveri

We are pleased to announce that the an International Symposium on High-Performance Thin Layer Chromatography will be held on 11th–13th June 2008, onboard a luxury cruising ship on route Helsinki-Stockholm-Helsinki. This magnificent chromatography cruise will include parallel workshops, a symposium, and a manufacturers session. The participation fee is very attractive: 600 € normal rate, 500 € reduced rate for administrations and universities, 300 € special rate for students and unemployed persons. This participation fee includes the full scientific programme, and added to that, lunches, coffee breaks, dinners, breakfasts, and two-night accommodation in nice 11 m^2 1–2 person cabins with a bathroom and window.

The scientific symposium program will feature invited keynote speakers, selected submitted lectures and poster presentations. Contributions are invited from all areas of thin-layer chromatography, but especially from colleagues working in the pharmaceutical, food, environmental and medical fields. Papers on theory, method development, validation, instrumental methods, hyphenated techniques, and quantitative applications in all areas of chemistry would be most welcome.
Colleagues wishing to participate in the scientific program should submit a brief abstract to the scientific committee (committee@hptlc.com), stating whether they wish to present an oral or poster paper. Abstract should be no more than 250 words and 2 pictures in a MS Word file (presentation in arial, 12, justified). The abstract should indicate the title, the authors names (with the presenting author underlined), affiliation (with e-mail address) and a brief description of the work to be presented.

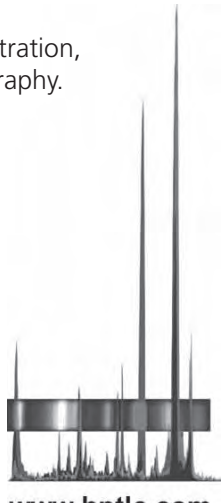
Deadlines are: March 15th 2008 for the abstracts submission, April 30th 2008 for last registration, June 30th 2008 for full paper submission to a spezial issue of Journal of Planar Chromatography.

info@hptlc.com for general information,
committee@hptlc.com for all abstracts and scientific matters, and
registration@hptlc.com for registration concerns.

Rates all included:

Workshop 200 €
Symposium 400 €
Full program 600 €
Admin./Univ. 500 €
Students 300 €

Special -100 € when membership



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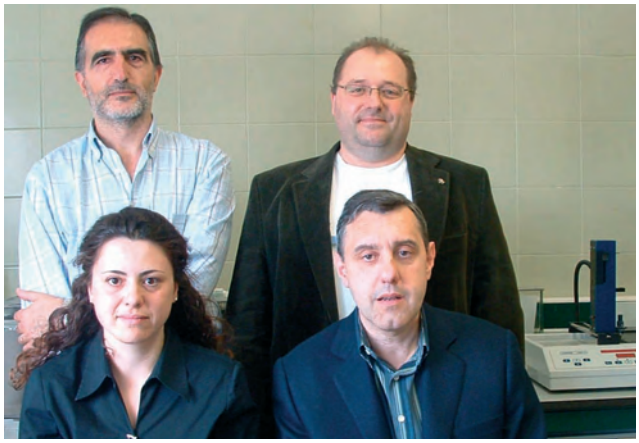
A general detection technique for HPTLC based on changes in fluorescence



▲ *Dr. Elena Mateos, Dr. Vicente L. Cebolla*, Dr. Luis Membrado, Dr. Jesús Vela (from left front anti-clockwise)*

The activity of the Separation and Detection Technology group of the Instituto de Carboquímica (CSIC) in Zaragoza, Spain, has traditionally been focused on the development of original analytical techniques for characterizing very complex mixtures, mostly derived from fossil fuel conversion processes. These consist of a structurally wide variety of high-molecular weight and/or high-boiling molecules.

Mixtures also include saturated hydrocarbons, which have neither ultraviolet nor fluorescent response under the usual analytical working conditions. Determination of the hydrocarbon type is a classical analysis in petroleum industry which presents considerable technical limitations. Therefore one of the group's research lines is to contribute to the development of universal systems to detect and quantify compounds which lack chromophores or are difficult to analyze using conventional optical detectors, e.g. saturated hydrocarbons and lipids.

Introduction

HPTLC was chosen because it is a well-adapted technique for analyzing all the constituents of complex, dirty samples, including those that do not migrate. This is an advantage over column-based techniques in which some heavy and/or polar hydrocarbons may be irreversibly adsorbed on the stationary phase. For HPTLC a general detection technique was developed, based on changes in emission of certain fluorescent substances which are induced through non-covalent interactions established towards the analyte [1, 2]. Therefore, it is neither a derivatization nor a destructive method and thus of special interest for the detection of non-fluorescent analytes.

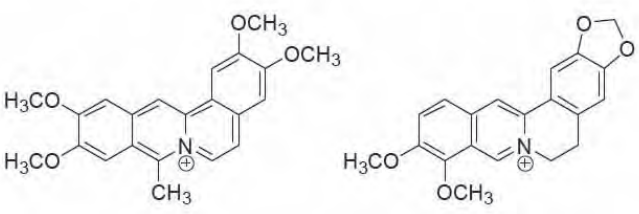
It is based on the fact that virtually any analytes induce changes in the fluorescence spectra of certain fluorescent substances, e.g. berberine and coralyne cations. Their emission intensities are exclusively affected by increase in emission (positive peaks) or fluorescence quenching (negative peaks).

HPTLC provides a solvent-free medium for studying the intermolecular photophysical processes. Magnitude and sign of the emission depend on the balance between non-specific and specific interactions, in addition to the dielectric permittivity of the medium. This creates a microenvironment that isolates the fluorophor and prevents non-fluorescence decay mechanisms [3, 4]. We refer to non-specific interactions as those being electrostatic in nature (i.e. ion-dipole induced interactions in the case of the studied cationic fluorophores), and to specific interactions as those involving directional interactions, such as electron-donor acceptor, H-bonding, etc.

It has been shown that polar compounds, e.g. antibiotics and amino acids, give negative peaks which correspond to fluorescence quenching [1, 2]. This quenching may be attributed to net specific interactions which increase the rate constant of non-radiative decay processes, thereby producing a decrease in quantum yield.

Chromatogram layer

HPTLC plate silica gel 60 (Merck), 10×10 cm



▲ *Structure formulae of coralyne (left) and berberine (right) cations*

Plate impregnation

Silica gel plates are pre- or post-chromatographically impregnated with berberine or coralyne solutions depending on their compatibility with developing solvents used. For example pre-impregnation is performed by dipping the plate in methanolic coralyne (6 or 12 mg/L) and berberine (60 mg/L) solutions followed by drying at 40 °C overnight.

Sample application

Bandwise with Automatic TLC Sampler 4, 7 tracks per plate, band length 2 mm, track distance 10 mm, distance from lower edge 10 mm, application volume 1–10 µL depending on sample concentration. On each plate, one track is left blank.

Chromatography

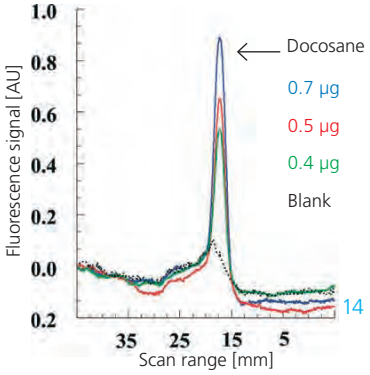
In the Horizontal Developing Chamber: Saturated hydrocarbons with dichloromethane, heavy gas oil samples with n-hexane and cholesterol with petroleum ether – diethyl ether – acetic acid 80:20:1. The migration time is 5 min.

Densitometry

Fluorescence measurement of berberine at 365/>450 and coralyne at 410/>450 nm.

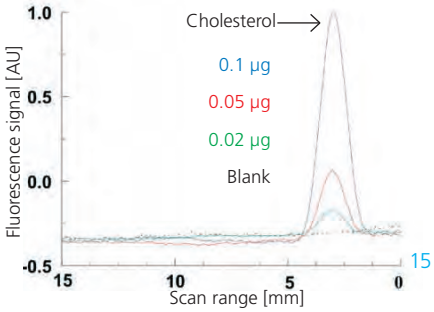
Results and discussion

On silica gel layers impregnated with berberine or coralyne solutions, the presence of paraffinic compounds produces an increase in fluorescence emission. The signal depends on alkane concentration and chain length for a given system [3–5].



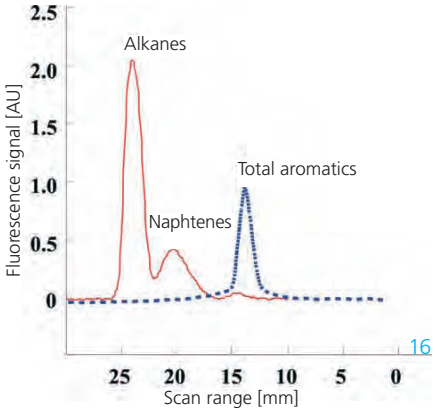
▲ *Fluorescent response of non-fluorescent docosane (n-C22H46) on a plate pre-impregnated with coralyne solution (6 mg/L).*

This phenomenon is not restricted to saturated hydrocarbons. Other apolar compounds, for example cholesterol, give positive peaks depending on sample load. In all cases the detection sensitivity can be tailored through impregnation concentration [5].



▲ *Fluorescent response of cholesterol on a plate pre-impregnated with coralyne solution (6 mg/L)*

The detection technique has been applied to petrochemical samples. The linear range for alkanes and naphthenes ranged between 0.05–1.5 µg, and 0.6–2.4 µg, respectively. Results obtained have been in a good agreement with those obtained from other techniques used in petrochemical industry [5, 6].



▲ *Analysis of a heavy gas oil sample taken from a refinery: Overlay of absorbance measurement at UV 254 nm (blue: on silica gel with acetone) and fluorescence measurement (red: on berberine-impregnated silica gel with n-hexane).*

[1] E. Gálvez et al. Anal. Chem. 78, 3699, 2006
[2] E. Mateos et al. J. Chromatogr. A 1146, 251, 2007
[3] F. Cossio et al. Anal. Chem. 72, 1759, 2000
[4] F. Cossio et al. Org. Lett. 2, 2311, 2000
[5] V. Cebolla et al. J. Chromatogr. Sci. 37, 219, 1999
[6] M. Matt et al. J. Sep. Sci. 26, 1665, 2003

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Further information is available on request from the author.

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Bioluminex™: An effective yet simple tool for screening mixtures

▲ *New Developments Group: Ms. Larissa Ikenouye, Ms Sarah Hickey, Dr. Sheryl Verbitski, Mr. Gerald Gourdin (from left to right)*

Dr. Verbitski*, New Developments Manager at ChromaDex (www.chromadex.com) in Boulder, Colorado and her research group, employ chromatographic separation techniques to analyze dietary supplements, plant biomass, pharmaceuticals, foods, and beverages. A main focus for the group is the advancement of the Bioluminex assay (www.bioluminex.com), which was recently launched into the international market.

Introduction

ChromaDex has developed a rapid-screening kit capable of identifying single compounds with biological activity in complex mixtures. Additionally this technology can be used as a bioassay guided fractionation tool. For complex mixtures like foods, food additives, and dietary supplements, standard bioactivity tests only establish overall activity. In such samples the identification of the active compound/s requires the tedious isolation of single components followed by assays to determine biological effects.

Alternatively, the Bioluminex™ assay involves the direct coupling of bioluminescence to HPTLC providing a unique, rapid, and effective way of monitoring toxicity or biological activity in complex mixtures. After chromatography, the HPTLC plate is coated with bioluminescent bacteria employing a simple dipping procedure. Single compounds that inhibit the bacteria are selectively identified as dark zones on a luminescent background. Results occur within seconds and can be documented by video imaging. This rapid-screening assay is particularly suitable to screen food, beverage, and dietary supplement related complex mixtures for the presence of non-traditional chemical and toxic adulterants. It is also a very useful tool for identifying compounds with potential biological activity. This technology is kit compatible thus providing a rapid and inexpensive analysis of many samples.

The bioluminescent marine bacterium *Vibrio fischeri* was chosen for this application. As *V. fischeri* cells reach a critical cellular density, respiring cells expel excess free energy as detectable blue-green light. This observed bioluminescence reflects the metabolic status of the cell and will decrease for cells exposed to toxic substances. Thus, a reduction in light emission is a measure of toxicity and can be selectively viewed and quantitated on HPTLC chromatograms.

Chromatogram layer

Merck or Bioluminex™ HPTLC plates silica gel 60 F_{254} (10 x 10 cm) pre-washed with methanol (chromatography) and dried at 100 °C for 15 min.

Sample application

Bandwise with Automatic TLC Sampler 4, band length 6 mm, application volumes 1–10 µL depending on sample and sample concentration

Chromatography

In a twin-trough chamber (10 x 10 cm, pre-saturated for 30 min, lined with filter paper) with different solvents mentioned below. The migration distance was 60 mm from the lower edge. The developed plates were dried at 40 °C for 2 h prior to detection.

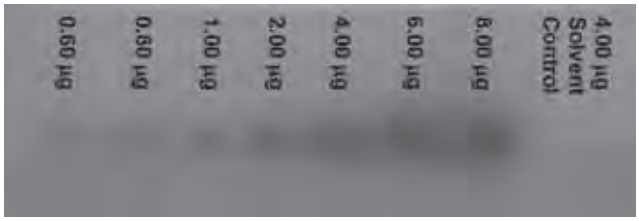
Bioluminex™ detection

Bioluminex bacteria were grown overnight (30 h) in a 200 mL batch culture in complex Bioluminex™ medium at 120 rpm and 28 °C under atmospheric conditions in an incubator. Directly before the assay Bioluminex™ buffer was added to the fully luminescent bacteria and dissolved at 120 rpm and 28 °C under atmospheric conditions. The developed HPTLC plate was coated with buffered luminescent bacteria using the TLC Plate Immersion Device III. To

improve data quality excess bacteria were removed from the plate using a squeegee device and images were immediately recorded over a 10 min span.

Results and discussion

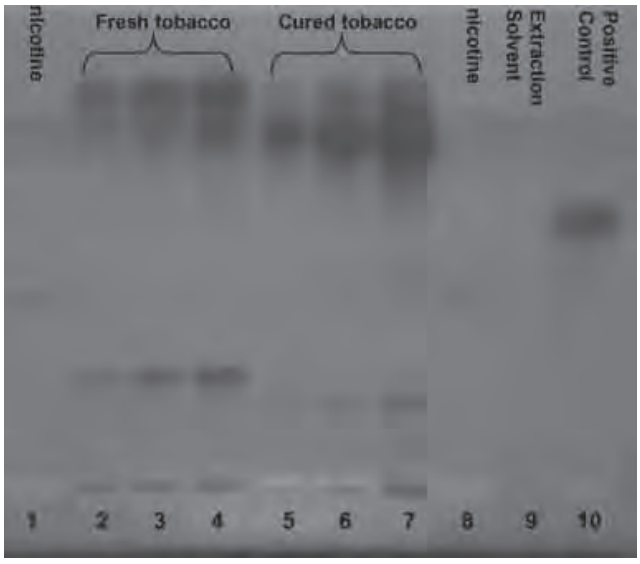
The Bioluminex assay can be used to quickly determine the activity of a substance. For example, various amounts of melamine were applied to the HPTLC plate and activity toward *Vibrio fischeri* was evaluated. In USA melamine found its way into pet food as illicit bulking agent that killed dogs and cats. The bioactivity of melamine can be detected by *V. fischeri*.



18

▲ *Detection of melamine*

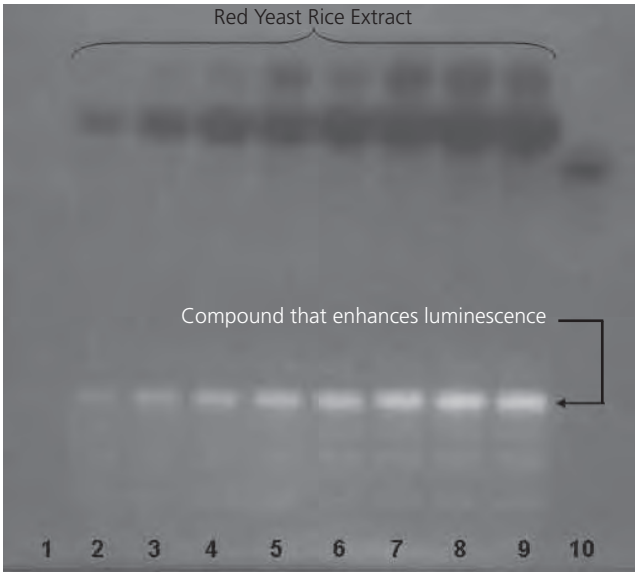
This assay provides a characteristic fingerprint that can be used to compare different products such as fresh versus cured tobacco. In the provided example, fresh and cured tobacco products were extracted with methanol, separated with chloroform – methanol – ammonium hydroxide 9:1:0.05 and then analyzed using the Bioluminex assay. The two extracts exhibit a unique chemical and biological fingerprint.



19

▲ *Detection of fresh versus cured tobacco extracts*

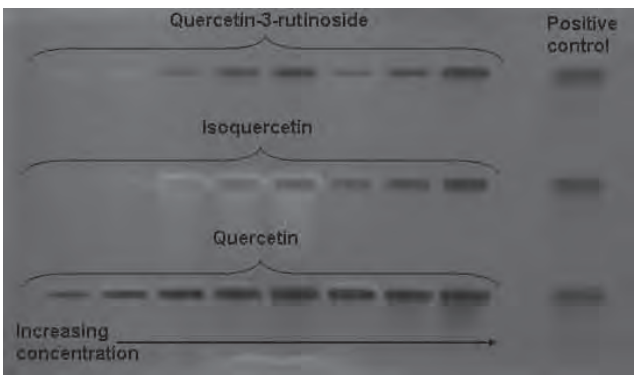
Complex mixtures such as a red yeast rice extract, which is a natural bacteriostatic and cholesterol-lowering agent and used in Asia as remedy for infects, can be screened for compounds with interesting biological activity. For instance, red yeast rice was extracted with methanol, separated using chloroform – ethyl formate – formic acid – methanol 5:5:2:2 and analyzed with the Bioluminex assay. Two compounds at R_f 82 and 91 inhibit *V. fischeri* bioluminescence while a compound at R_f 25 enhances bioluminescence.



20

▲ *Detection of red yeast rice extracts (track 2–9)*

The Bioluminex assay is a quick and easy tool to study structure-activity relationships (SAR). Presented here is a SAR analysis of 3 structurally similar compounds, quercetin, isoquercetin, and quercetin-3-rutinoside, applied to an HPTLC plate (without chromatography).



21

▲ *SAR analysis of 3 structurally similar compounds*

Up to now appliciability of this new bioactivity-based detection was investigated for samples in the field of food, forensic and environmental analysis. For example *V. Fischeri* is detecting ochratoxin in canned corn, aflatoxin B1 in honey, digoxin in milk, benzopyrene in celery seed, capsaicin in cayenne pepper, strychnine or monofluoroacetic acid in various drinks, domoic acid in soda, and patulin in apple juice. Thereby most interesting is the elucidation of further bioactive compounds which are first visible in some samples. Therefore, in a next step the employment of online mass spectrometry is helpful to get information about bioactive unknowns.

Further information is available on request from the author.

*Dr. Sheryl Verbitski, New Developments Department, Chroma-Dex Analytics, 2830 Wilderness Place, Boulder, CO 80301, USA, SherylV@chromadex.com

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CAMAG BioLuminizer

Combining the separation power of planar chromatography with bioluminescence detection enables the identification of single biological active compounds. All substances which are generating this distinct effect are detected in complex mixtures in the picomol range, e.g. in waste water or forensic samples. The comprehensive detection of bioactive compounds in samples includes for example also unknown metabolites and is quite different from the general target analysis using reference substances.

BioLuminizer is a compact, user-friendly detection system for bioluminescence imaging, which shows an exceptional image quality and a high resolution for a short exposure time.

All the consumables necessary for this detection are supplied by CAMAG as BioLuminex standard kit (No. 022.9765).

Determination of acrylamide in drinking water

▲ *Alexander Alpmann, doctorial candidate in the research group supervised by Dr. G. Morlock*

The research group of Professor Dr. Wolfgang Schwack*, Institute of Food Chemistry, University of Hohenheim, Stuttgart, is working in the field of planar chromatography as well as in other research fields (see CBS 93). The flexibility of the technique is impressive time after time, particularly for solving difficult problems in a simple way.

Introduction

Polyacrylamide is used e.g. in the paper, cosmetic, textile and construction industries as well as a flocculating agent in the treatment of drinking water. Due to its high solubility in water, the monomer acrylamide (AA) can be found in ground and drinking water. The AA concentration maximal allowed is stated at 0.1 µg/L in the EU directive 98/83/EC due to its cancerogenity.

The employment of HPLC-MS/MS according to DIN 38413-6, however, is non-profitable for smaler laboratories due to the high instrumental costs involved. Besides this, the recording of the protonated AA molecule (72 Da) can be interferred by matrix fragments due to the very low molecular mass. Additionally AA can not be detected in traces (ng/L) in the UV range. Hence, a cost-effective and selective alternative for routine analysis is based on the derivatization of AA with a fluorophor. This is performed prechromatographically at the starting zone of the HPTLC plate.

Sample preparation

Water samples (500 mL) are spiked with 250 µL N,N-dimethylacrylamide (1 ng/µL in methanol) as internal standard (IS), extracted by solid phase extraction with spherical activated carbon (Bakerbond Carbon) and 5 times eluted with 2 mL methanol – acetonitril 1:1 each. The combined eluate is reduced to ca. 1 mL in a rotary evaporator and subsequently under a gentle stream of nitrogen.

Standard and derivatization solution

Ultrapure water (500 mL each) is spiked with 50 to 200 µL AA solution (1 ng/µL in methanol) and 250 µL IS solution and treated as described above. For the blank, just ultrapure water is used.

The derivatization reagent dansulfinic acid which is not commercially available can be readily synthesized according to Scully et al. [1] and was used as 3.2 µg/µL methanolic solution.

Layer

HPTLC plate silica gel 60 (Merck) 10 x 10 cm

Sample application

As 6 x 3 mm area using the Automatic TLC Sampler 4 equipped with the heated spray nozzle (40 °C), application volume 100 µL for samples and standards, 8 tracks, track distance 10 mm, first and lower application position 12 and 8 mm, respectively, application speed 350 nL/s, overspraying of the starting zones with 20 µL derivatization solution

Derivatization

On the TLC Plate Heater at 120 °C for 1 h.

Chromatograpy

In the twin trough chamber with ethyl acetate after focussing with methanol (migration distance 70 mm (lower plate edge), migration time 15 min). After 2 min drying the plate is dipped in a polypropylene glycol solution (25 % in *n*-hexane) for fluorescence enhancement using the Chromatogram Immersion Device III (dipping time 1 s, dipping speed 5 cm/s).

Documentation

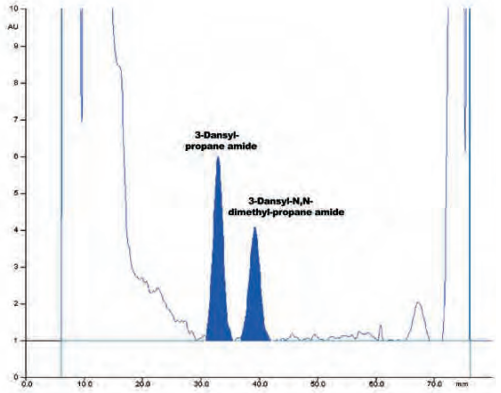
With DigiStore 2 System at UV 366/>400 nm

Densitometric evaluation

TLC Scanner 3 in fluorescence mode at UV 366/ >400 nm, linear calibration via peak area

Results and discussion

AA can selectively be detected without interfering matrix after derivatization with dansulfinic acid to 3-dansylpropane amide (DPA). The performance of sample preparation is ascertained by correction with IS (derivatized to 3-dansyl-N,N-dimethyl-propane amide).



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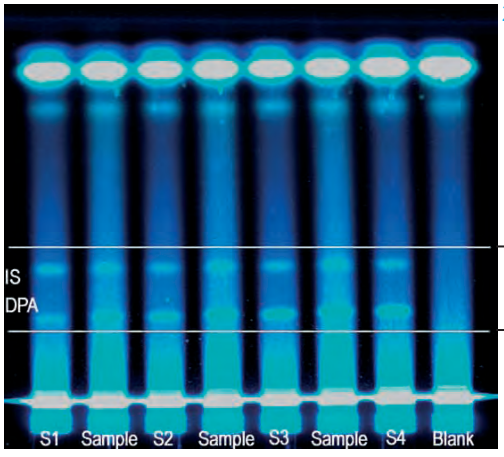
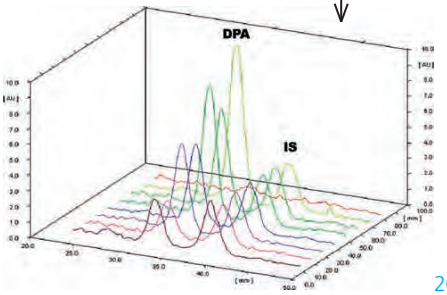
▲ *Fluorescence scan of an ultrapure water sample spiked with AA (0.2 µg/L)*

The mean within-run precision (*RSD*, *n* = 3 at 3 different concentration levels each) were established to be 4.8 % and the mean recovery over 3 concentration levels (0.1, 0.2 and 0.3 µg/L) was 96 % (corrected by the IS).



25

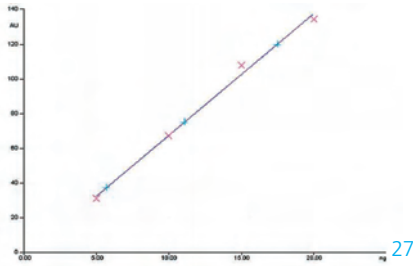
▲ *Plate image of ultra-trace analysis of AA (as DPA) in drinking water*



26

Fluorescence scan in the region marked of standards (0.1–0.4 µg/L, S1-S4), spiked blank samples (0.1–0.3 µg/L) and blank sample ▲

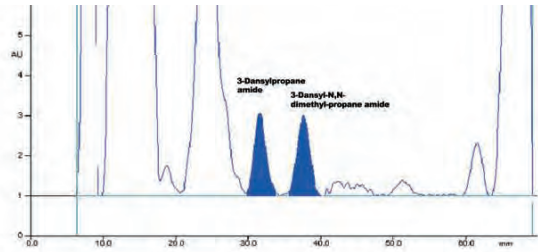
The regression in the working range of 0.1 to 0.4 µg/L was linear showing a relative standard deviation of ± 5.2 % (r = 0.9957).



27

▲ *Linear calibration of DPA (5–20 ng/zone or 0.1–0.4 µg/L)*

LOQ was calculated to be 0.08 µg/L AA in drinking water and thus enables a reliable control of the limit value at 0.1 µg/L according to 98/83/EC.



28

▲ *Densitogram of a sample spiked below the LOQ at 0.05 µg/L*

The method comparison with HPLC-MS/MS showed comparable results for the ultra-trace analysis of AA in ground water and proves the efficiency of the new method at the ultra-trace level:

Method comparison	HPLC-MS/MS AA [µg/L]	HPTLC-FLD AA [µg/L]
Ground water sample 1	<0.05	<0.05
Ground water sample 2	0.07	0.09
Ground water sample 3	0.18	0.24
Ground water sample 4	0.59	0.60

When relevant, additionally mass spectra can be recorded by online extraction (ca. 1 min/zone). Due to derivatization the protonated molecule of a higher mass (*m/z* 307) is highly advantageous because it can be detected with less interference compared to AA at *m/z* 72 and with a simple MS system (instead of the MS/MS).

[1] F. Scully et al. Environ. Sci. Technol. 18, 787, 1984

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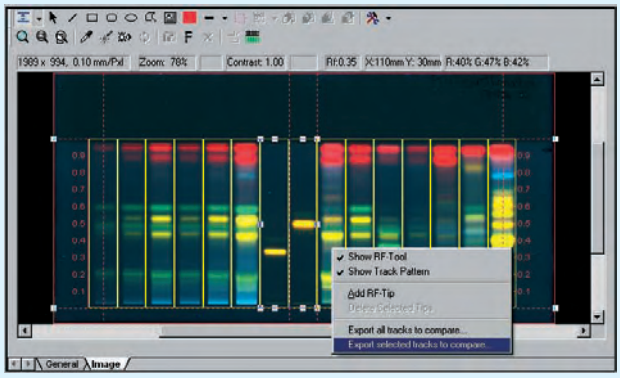
*www.ilc.uni-hohenheim.de

Visual comparison of multiple samples made easy!

An important characteristic and advantage of planar chromatography is its visual nature. TLC/HPTLC is the only chromatographic method offering the option of presenting the result as an image.

Image Comparison Viewer – a new software option running under winCATS 1.4.3 – allows comparing tracks from different TLC/HPTLC plates directly with each other on the same screen:



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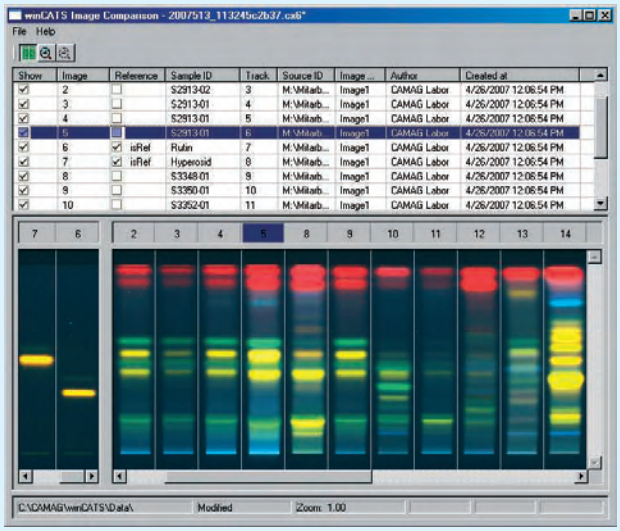
▲ *All available tracks are automatically marked and can be selected for transfer to the Image Comparison Viewer.*



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▲ *Image Comparison View of Passiflora incarnata (Passion flower) with other Passiflora species: Standards rutin (track 7) and hyperoside (8), various application volumes of Passiflora incarnata S2913 (0–5), Passiflora incarnata S3350 (9), Passiflora incarnata S3352 (10).*

- Simultaneous display of tracks from (groups of) samples or easy side by side comparison of reference tracks from different plates
- Automatic transfer of track information such as position, width, length, ID data to the viewer
- Differentiating between reference and sample tracks
- Reporting of the generated comparison as well as additional information
- Storage of data in individual archives for appropriate batch, lot number, etc.
- Trace-back of all generated data to the original analyses.

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