CBS 100 • March 2008

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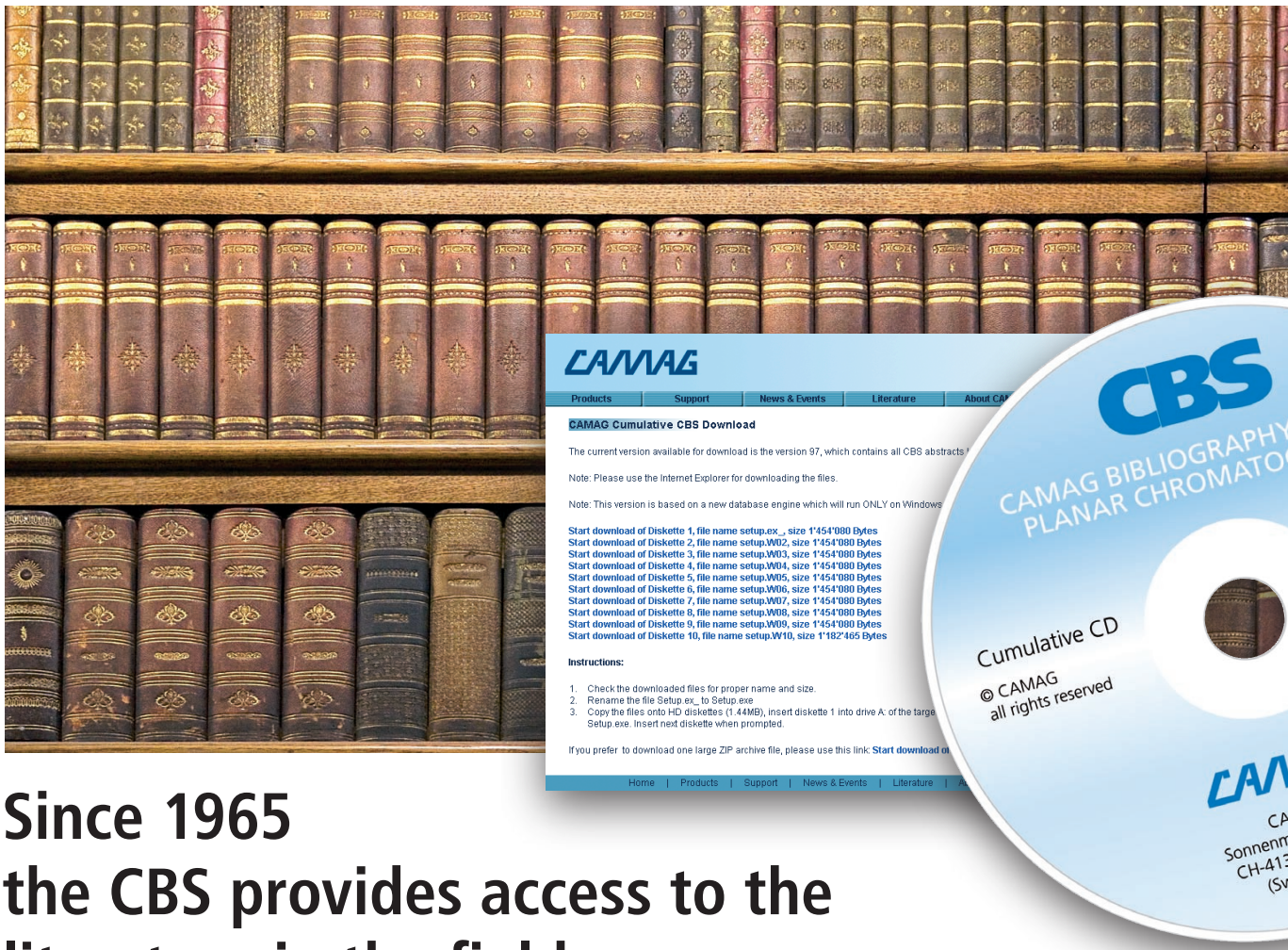

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Bio-activity based analysis of irradiated sunscreens using HPTLC and in situ detection with *Vibrio fischeri*

1

◀ *Dr. Urs Hauri, Vera Baumgartner, Dr. Christopher Hohl (left to right)*

The Kantonales Laboratorium Basel-Stadt is a state authority which supervises legal restrictions on commodities such as food, toys, cosmetics etc. The Non-Food Sector, headed by Dr. Christopher Hohl*, is specialized in analyzing additives e.g. preservatives, dye stuffs or UV filters in consumer items. Work also includes developing new methods for target compounds of toxicological concern using GC, HPLC and HPTLC. The recent introduction of toxicity tests on HPTLC plates has helped to detect peaks of special toxicological interest when screening for unknown compounds.

Introduction

Sunscreens are meant to protect human skin from damaging UVA and UVB radiation. However, in some formulations, the contained UV filters can degrade when exposed to sunlight, which was already shown in 1997 [1]. The toxicological relevance of the degradation compounds has not been investigated up to this date. The following method combines chromatography with bioactivity detection, enabling a determination of the specific bioactivity of photodegradation products in sunscreens.

The effect-specific analysis by HPTLC-bioluminescence coupling was chosen to link chemical-physical with biological detection methods using the luminescent bacterium *Vibrio fischeri*. An inhibition of bioluminescence is based on a disturbance of the bacteria's metabolism, whereas the degree of inhibition correlates with the toxicity of a compound. The *Vibrio fischeri* bacterium has been used since 1979 for ecotoxicological tests, especially in water analysis (cuvette test, DIN 38412 L34) but also for testing chemicals. To use the bacteria as a detection method for HPTLC, a special test kit called Bioluminex is available (www.chromadex.com).

The procedure in brief is as follows: The samples were applied on an HPTLC plate, separated by automated multiple development (AMD), documented under UV at 254 nm and 366 nm and then immersed in a solution of *Vibrio fischeri*. In addition, special zones of interest which were identified by *Vibrio fischeri* detection were scratched out from the HPTLC plate and

reanalyzed with HPLC-DAD and LC-MS. Peaks obtained with HPLC-DAD were then compared with our former HPLC data on the photodegradation compounds of UV filters.

Chromatogram layer

HPTLC plates LiChrospher Si 60 F_{254} (Merck), 20 x 10 cm, prewashed by developing in methanol followed by drying on a TLC plate heater at 120 °C for 30 min.

Sample preparation

For the generation of photodegradation products UV filter standards were dissolved in appropriate solvents and irradiated on microscope slides with artificial light (Atlas Suntest CPS+). Extraction from the slides was carried out with ethanol/acetone.

Sunscreen samples were either spread on microscope slides and irradiated with either artificial light or sunlight, or alternatively applied on the skin and irradiated with sunlight (30 min in the early afternoon during summertime, 47° northern latitude). Extraction from the slides and from the skin was performed with ethanol/acetone.

Sample application

Bandwise with a Linomat 5, band length 6 mm, distance from lower edge 8 mm, application volume 25 µL for UV filter standards, 10 µL for sunscreen samples (approx. 2 µg/band of UV filters on the plate)

Chromatography

UV filter standards photodegradation products: AMD 2 system with diisopropylether – n-hexane in 6 steps without pre-conditioning, drying time 2–3 min, migration distance 50 mm.

Sunscreen samples photodegradation products: AMD 2 system with t-butylmethylether – n-hexane in 7 steps with pre-conditioning, drying time 2–3 min, migration distance 50 mm.

Plates were dried for at least 30 min at 120 °C on a TLC plate heater III.

Densitometry

Multi-wavelength scan at 200–400 nm with TLC scanner 3 and WinCATS software

Detection

UV-detection at 254 nm and 366 nm with Reprostar 3, followed by biodetection via dipping the plate in the *Vibrio fischeri* solution for 1 s with an immersion device and evaluation with the BioLuminizer using an exposure time of 55 s.

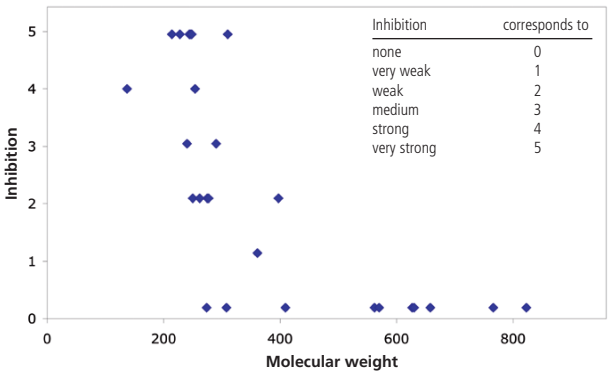
Results and Discussion

Limitations of the method, due to the application of the aqueous *Vibrio fischeri* (Vf) solution, concerned the choice of plate coatings and solvents as well as the drying of the plate. It turned out that only the polar layers silica gel and LiChrospher worked well for this aqueous bioassay regarding wettability. For the development of the separation system, only solvents could be used which were not toxic to *Vibrio fischeri* and which evaporated without leaving residues on the plate coating. The method is not suitable for the analysis of thermally labile or volatile compounds because of the heating process used for drying the HPTLC plate.

The potential of the method was clearly evident both for pure standards as well as for cosmetic samples. Detection of UV filter standards with *Vibrio fischeri* worked very well. Bioactive substances appeared as dark zones on a bright luminescent background. The inhibiting effect depended highly on the specific UV filter and ranged from a strong suppression of luminescence to no effect at all.

In comparison with conventional detection, biodetection mostly showed a different response: some zones which gave a high signal in UV could not be seen at all while other zones were much more pronounced with biodetection. Equal response was also possible in some cases. The bioactivity of UV filters correlated with their molecular weight (MW). All newer UV filters (not in use before 1998), having a MW of over 400, had no inhibiting effect on *Vibrio fischeri* at all.

Substance	Abbr.	Molecular weight	Inhibition of *Vibrio fischeri*
4-Aminobenzoic acid	PABA	137	strong
Benzophenone-1	B-1	214	very strong
Benzophenone-3	B-3	228	very strong
3-Benzylidene-camphor	3-BC	240	medium
Benzophenone-8	B-8	244	very strong
Benzophenone-2	B-2	246	very strong
Isoamylmethoxycinnamate	IMC	248	very strong
2-Ethylhexylsalicylate	EHS	250	weak
4-Methylbenzylidene Camphor	MBC	254	strong
Homosalate	HMS	262	weak
2-Phenyl-5-benzimidazole-sulfonic-acid	PBSA	274	none
Menthylanthranilat	MA	275	weak
2-Ethylhexyl-4-(dimethylamino)-benzoate	EHDP	277	weak
Ethylhexylmethoxycinnamate	EHMC	290	medium
2-Hydroxy-4-methoxybenzophenone-5-sulfonic acid	B-4/5	308	none
tert. Butylmethoxydibenzoylmethane	BMDBM	310	very strong
Octocrylene	OC	361	very weak
Diethylamino hydroxybenzoyl hexylbenzoate	DHHB	397	weak
4-(2-oxo 3-bornylidenemethyl) phenyl trimethylammonium methyl sulphate	CBMS	409	none
Terephtalydene dicamphor sulfonic acid	TDSA	562	none
Drometrizole Trisiloxane	DTS	570	none
Bis ethylhexyloxyphenol methoxyphenyl triazine	BEMT	627	none
Disodium Phenyl Dibenzimidazole Tetrasulfonate	DPDT	630	none
Methylene bis-benzotriazolyl tetramethylbutylphenol	MBBT	658	none
Diethylhexylbutamidotriazone	DEBT	766	none
Octyltriazone	EHT	823	none



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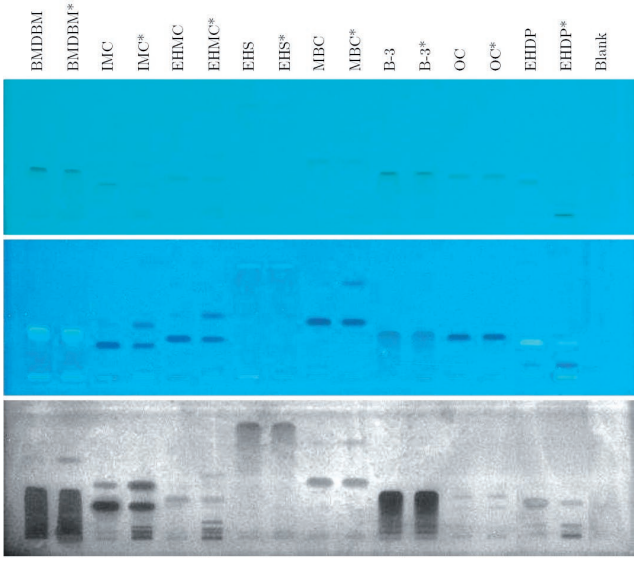
The comparison of irradiated UV filter standards with non-irradiated standards was also revealing. Photodegradation was observed for ethylhexyl-methoxycinnamate (EHMC), isoamylmethoxycinnamate (IMC) and ethylhexyldimethylaminobenzoate (EHDP). All photodegradation compounds had a stronger inhibiting effect than the UV filters themselves. It must be kept in mind, however, that the photodegradation of a UV filter strongly depends on the matrix and the combination of UV filters in which it is used.



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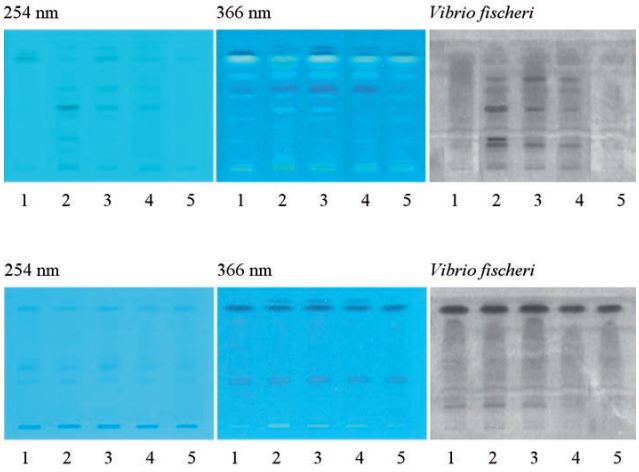
▲ *Detection of non-irradiated and irradiated (marked with*) UV filter standards at 254 nm, 366 nm and with Vibrio fischeri (top to bottom)*

Therefore, further tests were carried out on five commercially available sunscreens. The irradiated and non-irradiated sunscreen extracts were analyzed with HPTLC as well as with HPLC-DAD and LC-MS. Most sunscreens contained matrix compounds (e. g. preservatives) which also inhibited luminescence. Two of the sunscreens showed very strong degradation, which could be seen under UV but even more so with *Vibrio fischeri*. Detailed analysis was performed on spots which occurred only after irradiation indicating that these are photodegradation products. The first of the following sunscreen samples contains ethylhexylmethoxycinnamate, t-butylmethoxydibenzoylmethane and 2-ethylhexyl-4-(dimethylamino)-benzoate and shows strong degradation. Whereas the second sunscreen sample contains t-butylmethoxydibenzoylmethane, 4-methyl-

benzylidene camphor, 2-phenyl-5-benzimidazole-sulfonic acid and octyltriazone and has a stable formulation.



▲ *UV-detection versus Vibrio fischeri detection of two sunscreen samples (slide: track 1: not irradiated, track 2: artificial light, track 3: sunlight; skin: track 4: sunlight, track 5: not irradiated): sample showing strong photodegradation (top) and sample having stable formulation (bottom)*

To compare HPTLC and HPLC methods and to identify degradation compounds, sunscreen extracts were developed on an HPTLC plate and the zones of interest were scratched out and reanalyzed with HPLC-DAD and LC-MS. This procedure for identification worked very well, even though separation with HPTLC was inferior to HPLC as a zone of high bioactivity on the HPTLC plate could lead to several peaks in HPLC. Peak heights of compounds using bioluminescence detection didn't correlate with those when using conventional detectors (HPTLC-UV, HPLC-DAD, LC-MS). In one case, a highly bioactive compound would have remained undetected by the physical-chemical detectors used.

Detection with *Vibrio fischeri* can be used for two purposes: First, its characteristic detection selectivity enables the detection of previously undetected compounds. Second, the inhibition of the bacteria as an indicator of bioactivity can help single out those photodegradation compounds which need further toxicological evaluation.

The complete study titled "Bio-activity based Analysis of irradiated Sunscreens using HPTLC and in situ Detection with *Vibrio fischeri*" can be downloaded from: http://www.kantonslabor-bs.ch -> Information (Infos) -> Opinions (Themenpapiere)

or with deeplink: http://www.kantonslabor-bs.ch/content.cfm?nav=17&content=25

[1] Schwack, W., Rudolph, Th. Photoreactions of chemical UVA filters in cosmetics. GIT Laboratory Journal 1 (1997) 17–20.

* Dr. Christopher Hohl, Kantonales Laboratorium Basel-Stadt, Abteilung Non-Food, Postfach, CH-4012 Basel, Switzerland, Christopher.Hohl@bs.ch

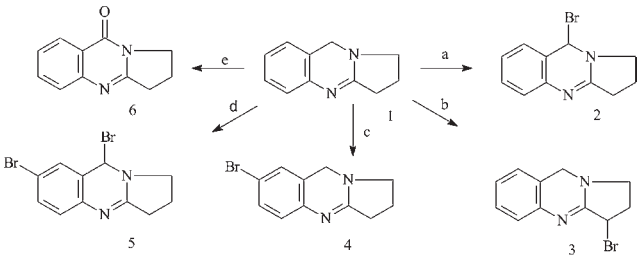
Product control: Bromination and oxidation of the alkaloid deoxypeganine

▲ *Dr. N. Mukarramov, Prof. Dr. Kh. Shakhidoyatov (left to right)*

The department of Organic Synthesis in the Institute of Chemistry of Plant Substances, headed by Professor Shakhidoyatov*, has been engaged in research in the field of synthesis and chemical modification of natural compounds including alkaloids for many years. Currently structure elucidation is performed by IR spectroscopy, H^1-/C^{13}-NMR spectroscopy, mass spectrometry and X-ray fluorescence spectroscopy. Planar chromatography is used for process control, identification and purity control.

Introduction

The alkaloid deoxypeganine (DOP) is isolated from the plant *Peganum harmala* [1] and is used as anticholine esterase preparation in medical treatment [2]. For synthesis or chemical modification of natural compounds, it is necessary to supervise the reaction process stage by stage, thereby monitoring the multiple directions of the reactions that the process may take. It is essential to know the structures of the initial compounds, the intermediates and final product(s). The process of DOP (1) bromination can undergo the following reactions:



Direction (a) includes the bromination in position 4 yielding 4-bromo-deoxypeganine (2). Direction (b) leads to α-bromo-deoxypeganine (3). Additionally, bromination also occurs at the phenyl ring in position 6 (c) with formation of 6-bromo-deoxypeganine (4). The double bromination (d) finally results in 4,6-dibromo-deoxypeganine (5). Another type of reaction in the presence of N-bromosuccinimide (NBS) is the oxidation (e) affording the alkaloid deoxyvasicinone (DOV, 6) or 6-bromo-deoxyvasicinone (7).

For control of the product reactions, planar chromatography is used because of its various advantages:

- **Analyses on different research projects can simultaneously be performed.**
- **The analysis takes not more than 10 min and does not require a special sample preparation.**
- **HPTLC is an inexpensive technique and does not need much solvent, i.e. only 5 mL per plate.**
- **20 samples can be analyzed in parallel.**
- **HPTLC allows the qualitative and quantitative separation of a difficult mixture of substances, followed by spectra identification.**

Sample preparation

1 mg of the exact weight of the compound is dissolved in 1 mL methanol.

Chromatogram layer

HPTLC plates silica gel 60 F_{254} (Merck, Germany), alternatively flexible plates TLC AL Sil G/UV (Whatman, UK) or HPTLC-AF-UV (Sorbfil, Russian Federation), all 20 × 10 cm

Sample application

Bandwise with Linomat 5, up to 20 tracks, 3 mm band length, application volume 2 and 4 µL of the sample solution, track distance 7 mm, distance from the left side 15 mm, distance from lower edge 8 mm

Chromatography

In the twin-trough chamber 20 × 10 cm with chloroform – methanol – acetone – cyclohexane 5:1:5:5 for the Merck and Whatman phases and 4:1:4:4 for the Sorbfil phases. The migration distance was 60 mm

from the lower plate edge. After development the plate was dried in a stream of cold air for 10 min.

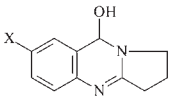
Densitometry

Absorption measurement at 254 nm with TLC Scanner 3 and winCATS software

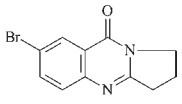
Note of the editor: It is recommended to perform the measurement at the optimum wavelength of each substance, optional with the multi-wavelength scan, to obtain the most sensitive detectability.

Results and discussion

Bromination of DOP (1) with NBS in the ratio 1:1 in chloroform gives compounds 2 and 5 which upon alkaline treatment turn into the corresponding hydroxy derivatives (8) and (9). Compound 8 is the natural alkaloid peganole (4-hydroxy-deoxypeganine), previously isolated from *Peganum harmala*. Compound 9 is 6-bromo-peganole. Bromination in the ratio 1:4 followed by processing with 5 % KOH solution leads to the formation of compound 9 and partially compound 7.



X=H (8); X=Br (9)



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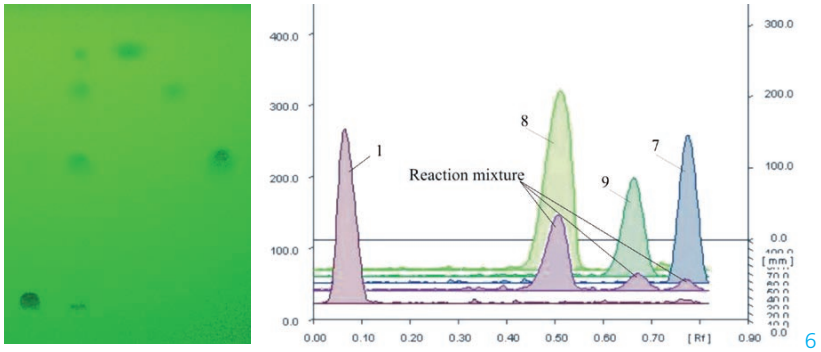
Unlike the reaction in chloroform, DOP hydrochloride dissolved in water reacts with NBS (ratio 1:1) to 6-bromo-deoxypeganine (4). Changing the ratio to 1:4 the direction of the reaction tends to the formation of 6-bromo-deoxyvasicinone (7). Oxidation DOP*HCl with potassium permanganate gives peganole (8) and partially DOV (6).

Apparently the bromination of DOP in chloroform and its hydrochloride in water, respectively, differ in terms of reaction products. Because it is difficult to isolate the products formed and to determine them gravimetrically, quantitative HPTLC was used.



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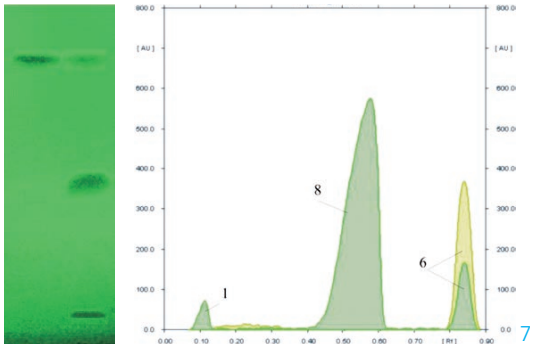
▲ *Chromatogram of DOP products in chloroform; track 1: DOP, track 2: reaction product of 1 with bromine and KOH, track 3: 6-bromo-deoxyvasicinone (7), track 4: 6-bromo-peganole (9), track 5: peganole (8)*



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▲ *Chromatogram of DOP*HCl oxidation products with $KMnO_4$; track 1: DOV (6), track 2: reaction product, containing peganole (8) besides partially DOV (6)*

The best selectivity between peganole (8) and 6-bromo-peganole (9) was found on the Merck plates.

hR_F values in the reaction mixture			
Plates / Products	Merck	Whatman	Sorbfil
1	8	7	8
8	49	51	61
9	69	69	69
7	80	78	83

[1] Khashimov Kh. Sh. et al., Chem of natural compounds 5 (1969) 456

[2] Yunusov S. Yu. et al., Pat. 605614 (USSR) 1978

Further information is available on request from the authors.

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CAMAG under new leadership


8

Mr. Rolf Rolli joined CAMAG 1. September 2007 as CEO. He brings extensive management experience from his associations with several similar companies. He worked in research & development for Sandoz in Basel and in the USA. He was Head of Development & Product Management of Knoll Pharma. Since 1992 Mr. Rolli has worked in the position of CEO for several life science companies, such as Life Technologies (Invitrogen)/Switzerland, SOTAX, Zymark Switzerland and Germany. Before he joined CAMAG he was CEO of Merck Biosciences, a daughter company of Merck Darmstadt. In all of his affiliations he has been in close contact with customers and distributors worldwide. He has organized workshops in Switzerland and abroad and has made numerous presentations at international symposia.

Since Mr. Rolli joined CAMAG, the winds of change have blown through our company and to our international distributors, who appreciate his support. In the few months Mr. Rolli has been with CAMAG, he has already made extensive business trips, to the United States, to India and to the Middle East. Upon his initiative CAMAG started a new campaign to exploit the cosmetics industry as a (potentially) lucrative field of application for planar chromatography, which is reflected in several contributions to this CBS.

From Mr. Rolli's activities as CEO you may expect a leap forward for CAMAG and for planar chromatography!

(Dieter Jänchen)

CBS

CAMAG BIBLIOGRAPHY SERVICE

A database devoted to planar chromatography

The CAMAG Bibliography Service is designed to serve the requirements of TLC/HPTLC users. It is distinguished from other databases in that it contains information oriented towards practice, for example detailed information on the separation systems or detection methods used.

A CBS abstract contains – if quoted in the original publication:

- Name(s) of author(s)
- Address of the author to whom correspondence should be directed
- Original title, if in one of the common Western hemisphere languages
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- Brief abstract of the TLC/HPTLC related content with particular reference to separation systems, detection methods, quantification, results, etc.
- Key words

Thus the database search informs analysts about the existence of TLC/HPTLC papers that might be helpful for solving their particular analytical question.

Surely the CBS is not intended to free the user from studying the original publication.

Since 1997 the most comprehensive compilation of literature in the field of Planar Chromatography (TLC/HPTLC) is available as database. It contains all abstracts of CBS issues beginning with CBS 51 (May 1983). The database is regularly updated and currently includes more than 8000 abstracts of publications between 1982 and today.

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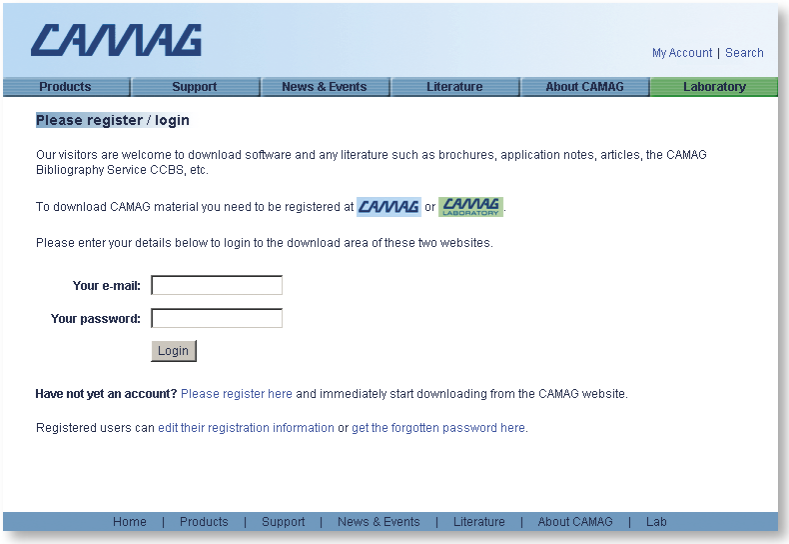

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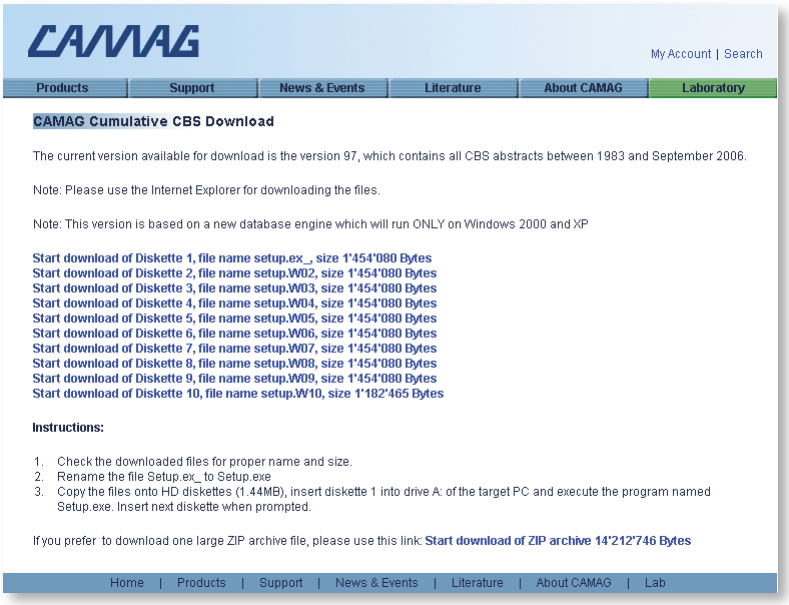

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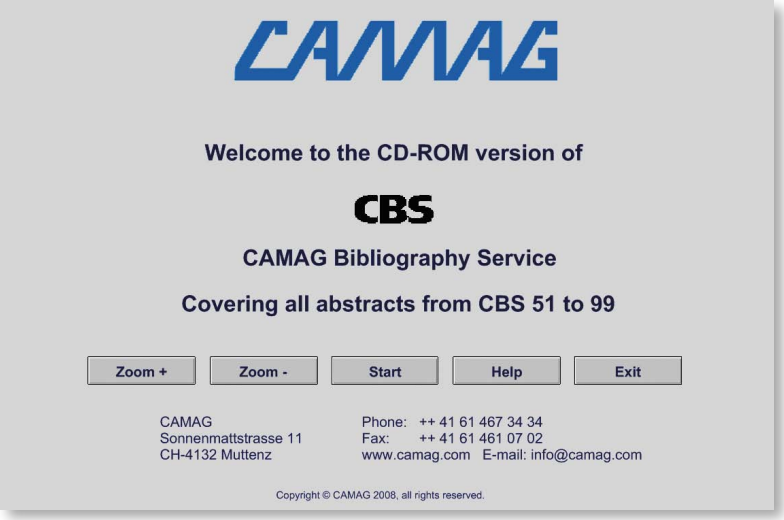
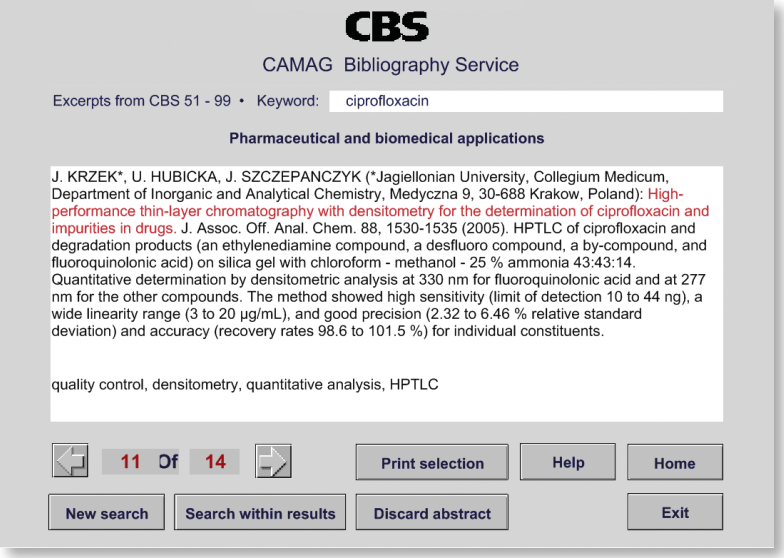
For example a search for 'ciprofloxacin' retrieved 14 hits in the CCBS database of which the 11th is viewed. All abstracts in which the key word occurs appear separately and in sequence on the screen together with an indication of the total number of abstracts (14), in which the search word was found, and the currently viewed abstract entry (11). Now you can browse the found abstracts by clicking the left and right arrows displayed. You can eliminate abstracts from the search results using the DISCARD ABSTRACT button or refine your search by using the SEARCH WITHIN RESULTS button. Click PRINT to print out the selected abstracts. Select NEW SEARCH to start again with a new search. Press HELP to obtain further information regarding the search options. HOME directs you to the welcome page and EXIT closes the database.



Click the START button in the welcome page and enter your search term.

Start the search by clicking START SEARCH or by pressing ENTER on your keyboard. For example the search term may be a substance name, a technique, a reagent or an author's name. For detailed information on available search options click the HELP button. Using ZOOM+ and ZOOM- varies the view display.



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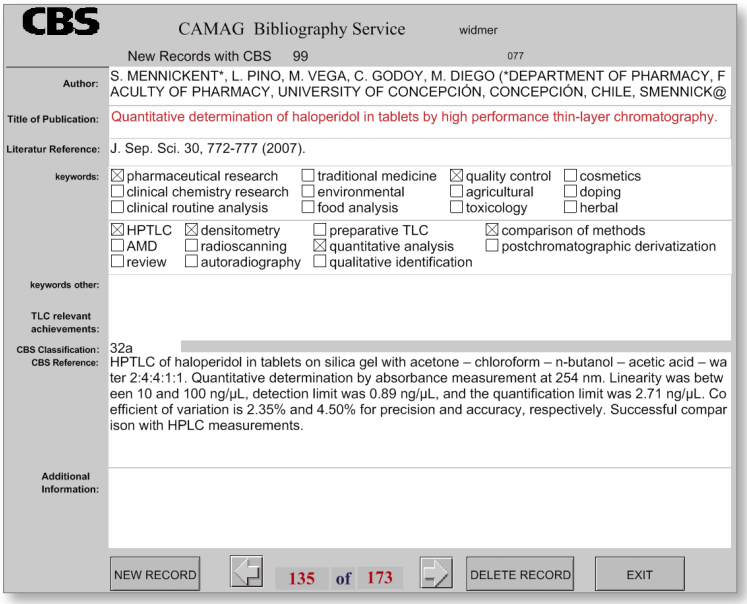
Composing the literature abstracts of the CAMAG Bibliography Service ("yellow pages" of the CBS) once and today

The CBS was established by Dr. Dieter Jänchen in 1965 at the suggestion of some business friends who agreed to provide the editor with reports on relevant TLC publications that they came across. In order to keep these reports as consistent as possible, a reporting form was created in which the referees entered the details they considered essential – partly written by hand, partly with a typewriter. From these reports the editor composed the individual CBS abstracts, one by one. This was the practice until the end of the 80's.

The circle of referees changed over the years as did the reporting format, which had been refined over the years, providing the editor the option to compose the CBS abstract from the referee's report with modest changes, omissions, etc. During the 90's the quality of the reports had improved to an extent that drafting the abstracts could be delegated to a knowledgeable CAMAG person and the editor needed to make corrections only here and there.



▲ *CBS report entry mask showing an example*

In 2003 Dr. Gerda Morlock took over CBS editorship. She introduced an efficient electronic reporting system for the referees. In the present system (since January 2005) the referees, using the previous format enter their reports directly into a database and email these directly to CAMAG. Here they are collected by Ms Valeria Widmer who adds nominal corrections in order to make them consistent with other reports. She also checks whether all required methodological details are contained and, if necessary, requests clarification. The complete collection of abstracts, arranged already according to the CBS classification system, is then emailed to Dr. Morlock. After her final approval the collection of abstracts is mailed to the printers. At the same time they are electronically incorporated in the current CCBS database, resulting in the CCBS database being updated with each issue and readily available for download from the CAMAG website.

Feedback from our customers shows us that this unique TLC/HPTLC bibliography with its convenient and efficient search routine is highly appreciated.

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PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

Es ist mir eine große Freude, Ihnen die 100. Ausgabe des CBS vorzulegen und das zudem im 70. Jubiläumsjahr der Planar-Chromatographie!

Die Geburtsstunde der Planar-Chromatographie wird auf das Jahr 1938 zurückgeführt, als N. A. Izmailov und M. S. Shraiber im Pharmazeutischen Institut in Kharkov, Ukraine, als erste ein zirkulares Dünnschicht-Chromatogram erzeugten.

Die Geburtsstunde des CBS war 1965, als Dieter Jänchen die überaus weitsichtige Idee hatte, Kunden regelmäßig mit Kurzreferaten der neuesten Publikationen über Dünnschicht-Chromatographie zu versorgen und sie darüber hinaus über Entwicklungen in diesem Fachbereich zu informieren. Er war über die Jahre der kraftvolle Motor des CBS und führte das Journal zu weltweit hohem Bekanntheitsgrad.

In den letzten 40 Jahren wandelte sich der CBS im Erscheinungsbild (siehe erste Inlayseite), und die sogenannten weissen Seiten fokussieren bereits seit einigen Jahren verstärkt praktische Anwendungsbeispiele.

In dieser CBS-Jubiläumsausgabe reflektiert eine spezielle Einlage zu den gelben Seiten die Entwicklung und den Fortschritt dieses Bibliographie-Dienstes – eine einzigartige und von den Kunden hochgeschätzte Datenbank. Anlass genug, die aktuelle Datenbank-Version kostenfrei unter **www.camag.com** herunterzuladen.

Herzlichst Ihre

Gerda Morlock

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Dear friends

It is a great honor to publish the 100th CBS issue, especially this year, in the year when planar chromatography celebrates its 70th birthday!

Planar chromatography dates back to the year 1938 when, N. A. Izmailov and M. S. Shraiber at the Pharmaceutical Institute in Kharkov, Ukraine first made a circular thin-layer chromatogram, called at that time, spread layer or spot chromatography.

The CBS dates back to 1965 when Dieter Jänchen had the outstanding, forward-looking idea to support customers regularly by providing abstracts about recent thin-layer chromatographic publications in addition to information on the latest developments in the field. He has been the force and guiding light behind CBS over the years, which has resulted in the journal having worldwide recognition.

During its 40 years of existence the CBS has undergone several changes of appearance, readily apparent from page 1 of the inlay to its white pages, which have increasingly focused on reports of planar chromatography in practice from all fields of application.

In this jubilee CBS issue, a special inlay on the yellow pages of the CBS reports on the progress of this bibliography service over the years, which has become a unique (re)search tool, highly appreciated by the customers. Take the opportunity and download free of charge the latest database version from **www.camag.com**.

Sincerely,

Gerda Morlock

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CAMAG

MÄRZ
MARCH
2008

100

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

100 002 Gertrud MORLOCK*, W. SCHWACK (*University of Hohenheim, Institute of Food Chemistry, Garbenstr. 28, 70599 Stuttgart, Germany; gmorlock@uni-hohenheim.de): The contribution of planar chromatography to food analysis. J. Planar Chromatogr. 20, 399-406 (2007). General aspects of food analysis using planar chromatography as an optimum tool for national and international standards to keep analysis economical. Contents: 1. The changing situation as a challenge; 2. TLC and HPTLC applications in food analysis and rapidly growing topics; 2.1 Topics in the past twenty years; 2.2 Rapidly growing topics in the future; 3. Is HPTLC a reliable quantitative method in food analysis; 3.3 Performance key data; 3.2 Method comparison; 3.3 Separating power; 4. Obstacles and benefits of planar chromatography; 4.1 Obstacles; 4.2 Benefits; 5. Future potential of HPTLC in food analysis; 5.1 Simplified sample preparation; 5.2 Simultaneous determination of analytes with different detection principles or analytes difficult to detect in general; 5.3 Digital evaluation of plate images; 5.4 Bioactivity-based detection; 5.5 Mass-selective information on demand; 5.6 Cost-effectiveness; 6. Conclusions. Planar chromatography for simple solution of difficult problems, reduced sample preparation, selective derivatization, quantitative and sensitive determinations using appropriate instrumentation, compliance with regulated environments, e. g. cGMP and cGLP, validation fulfilling requirements for reliable analysis, reduced costs, high throughput and comparable results.

food analysis, review, HPTLC quantitative analysis, qualitative identificatio, comparison of methods 1b

100 003 E. REICH*, Anne SCHIBLI (*CAMAG Laboratory, Sonnenmattstr. 11, 4132 Muttenz, Switzerland; eike.reich@camag.com): High-Performance Thin-Layer Chromatography for the Analysis of Medicinal Plants. Thieme Medical Publishers Inc., New York (2006). This book presents the theoretical and technical information needed to perform reliable and reproducible high-performance thin-layer chromatography (HPTLC) to establish the identity, purity, quality, and stability of raw materials, extracts, and finished botanical products. The text provides a complete overview of the techniques and common applications of HPTLC in herbal analysis. Chapters covered are theoretical concepts (stationary phase, mobile phase, TLC results, densitometry), practical aspects of modern TLC (sample preparation, selecting the stationary phase, sample application, chromatogram development, derivatization, documentation, reporting and record keeping, TLC software, standardization), typical applications in herbal analysis, method development, and validation of qualitative and quantitative HPTLC methods.

herbal traditional medicine, quality control, review, HPTLC, quantitative analysis, qualitative identification, densitometry 1a

2. Fundamentals, theory and general

100 004 Tatjana DJAKOVIC-SEKULIC*, N. PERISIC-JANJIC, C. SARBU, Z. LOZANOV-CRVENKOVIC (*Department of Chemistry, Faculty of Sciences, University of Novi Sad, Trg D. Obradovica 3, 21000 Novi Sad, Serbia; tanja@ih.ns.ac.yu): Partial least-squares study of the effects of organic modifier and physicochemical properties on the retention of some thiazoles. J. Planar Chromatogr. 20, 251-257 (2007). TLC with aqueous ammonia-organic modifier (acetonitrile, dioxane, acetone) mobile phases has been used to study the effect on retention of the chromatographic system and the physicochemical properties of twelve 2,4-dioxotetrahydro-1,3-thiazoles. Principal-component analysis and partial least-squares regression were used to determine the molecular properties with the greatest effect on retention for each modifier. Good correlation was obtained between experimental and calculated retention data. HPTLC on RP-18 without chamber saturation. Detection under UV 254 nm.

HPTLC qualitative identification 2c

100 005 Malgorzata JANICKA (Faculty of Chemistry, Department of Physical Chemistry, Maria Curie-Sklodowska University, M. Curie-Sklodowska Sq. 3, 20-031 Lublin, Poland; mjanicka@hermes.umcs.lublin.pl): Use of thin-layer and over-pressured-layer chromatography to study the hydro-

phobicity of homologous s-triazines. J. Planar Chromatogr. 20, 267-274 (2007). Comparison of retention factors in pure water, log kw, determined by linear extrapolation and by a numerical method based on Oscik's equation, and calculated values of log P as hydrophobicity indices for nine homologues s-triazines. The effect of mobile phase pH on solute retention was investigated as well as the effect of mobile and stationary phase properties on chromatographic behavior; 3 different organic modifiers (dioxane, acetonitrile, and tetrahydrofuran) and two stationary phases (RP-8 and RP-18) were used. Correlations of calculated log P values and log kw with carbon number confirm the usefulness of chromatographic techniques for studying the hydrophobicity of organic compounds. TLC on RP-18 with buffer - methanol mixtures in saturated sandwich chambers. Detection under UV light at 254 nm.

pharmaceutical research, qualitative identification 2d

100 006 L. KOMSTA*, W. MARKOWSKI, G. MISZTAL (*Department of Medicinal Chemistry, Skubiszewski Medical University, Jaczewskiego 4, 20-090 Lublin, Poland; lukasz.komsta@am.lublin.pl): A proposal for new Rf equal-spread criteria with stable distribution as a random variable. J. Planar Chromatogr. 20, 27-37 (2007). The retention factor Rf is used in several criteria generally known as chromatographic response functions. In TLC and HPTLC most of these are based on differences between the retention factors of two substances, which are summed or multiplied. There are also other functions, e. g. the multispot response function which has a clearly defined range (0 to 1), but its distribution is unstable. Here two new independent coefficients: Ru (retention uniformity) and Rd (retention distance) are proposed; these always have values between the range 0 to 1 and stable density, irrespective of the number of compounds separated. An example is given of their use in the separation of fibrate-type antihyperlipidemic drugs by normal and RP-TLC (114 systems).

2d

100 007 Marzena PODGÓRNA (Institute of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland; marzenapodgorna@wp.pl): Effects of the composition of two-component mobile phases on the separation of selected porphyrins by partition thin-layer chromatography. J. Planar Chromatogr. 20, 259-260 (2007). Investigation of the effects of two-component mobile phases (carbon tetrachloride-methanol, chloroform-methanol, dichloromethane-methanol and chlorobenzene-methanol) on the separation of porphyrins. The results were characterized by determination of the Rf values. TLC of five porphyrins (porphin, 5,10,15,20-tetra(4-methoxyphenyl)porphyrin, 5,10,15,20-tetra-(4-pentyloxyphenyl)porphyrin, 5,10,15,20-tetra(4-decyloxyphenyl)porphyrin), and5,10,15,20-tetra-4-hexadecyloxyphenylporphyrin) on RP-18. Optimum separations were obtained by use of 5:5 mixtures.
qualitative identification 2c

100 008 B. SPANGENBERG*, R. E. KAISER (*University of Applied Sciences, 77652 Offenburg, Badstrasse 24, Germany; spangenberg@fh-offenburg.de): The water content of stationary phases. J. Planar Chromatogr. 20, 307-308 (2007). The water content of stationary phases as well as controlling the water content are very important for obtaining reliable separation results in TLC. Dimroth's salt (today widely known as Reichardt's dye), 4-(2,4,6-triphenylpyridinium) 2,6-diphenylphenoxide, can not only be used to measure the water content of solvents; it was found to be suitable for easily checking of the water content of TLC layers: A solution of 2,6-diphenyl-4-(2,4,6-triphenyl-1-pyridino)phenolate hydrate in acetone (1.65 mg/mL) was applied to the plate as spot or as a band. The plate was stored in an oven at 120 °C for 30 min then left for 20 min over sulfuric acid of different concentrations which resulted in relative humidity from 9 to 72 %. From the spot spectra measured directly between 400 to 900 nm absorption spectra can be calculated; an inverse linear relationship exists between the absorption at 500 nm and water content. Spots of Dimroth's salt are suitable for checking the water content of TLC plates. Simply measuring the dye absorption at 500 nm and comparison with a calibration plot reveals the water content of the layer.

2d

3. General techniques

100 009 R. BHUSHAN*, H. BRÜCKNER, V. KUMAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee 247 667, India): Indirect resolution of enantiomers of penicillamine by TLC and HPLC using Marfey‘s reagent and its variants. Biomed. Chromatogr. 21 (10), 1064-1068 (2007). Indirect chiral separation of penicillamine (3,3-dimethylcysteine) enantiomers after derivatization with Marfey‘s reagent (FDNP-Ala-NH2) and two of its structural variants, FDNP-Phe-NH2 and FDNP-Val-NH2, with phenol - water 3:1 and solvent combinations of acetonitrile and triethylamine phosphate buffer in normal and reversed-phase TLC, respectively. Also separation of the diastereomers on a reversed-phase HPLC column with gradient elution of acetonitrile and 0.01 m trifluoroacetic acid. Comparison of the results due to these three reagents. Successful application of the method for checking the enantiomeric impurity of l-penicillamine in d-penicillamine and to check the enantiomeric purity of pharmaceutical formulations of d-penicillamine.

quality control, quantitative analysis, qualitative identification, HPTLC comparison of methods 3d

100 010 T. DJAKOVIC-SEKULIC, Nada PERISIC-JANJIC* (*Department of Chemistry, Faculty of Science, University of Novi Sad, Trg D. Obradovica 3, 21000 Novi Sad, Serbia; pnada@ih.ns.ac.yu): Study of the characteristics and separating power of unconventional TLC supports. II. Principal-Components analysis. J. Planar Chromatogr. 20, 7-11 (2007). Study of chromatographic retention data for the 3,5-dinitrobenzoic acid esters of a homologous series of aliphatic C1 - C20 linear alcohols on five unconventional TLC stationary phases - rice starch, microcrystalline cellulose, aminoplast, talc, and paraffin oil-impregnated silica gel. The stationary phases were characterized by means of retention scores obtained by principal-components analysis.

qualitative identification 3b

100 011 R. JOHNSSON*, G. TRÄFF, M. SUNDEN, U. ELLERVIK (*Organic Chemistry, Lund University, P.O. Box 124, SE-221 00 Lund, Sweden): Evaluation of quantitative thin layer chromatography using staining reagents. J. Chromatogr. A 1164 (1-2), 298-305 (2007). TLC using staining reagents is fast, versatile and sometimes the only viable method method for analyzing organic compounds without chromophores. Investigation of quantitative TLC using staining reagents in combination with modern image analysis software showed that it is possible to get reliable measurements, suitable for high-throughput screening or physical organic investigations. Illustration of the range of detection and the errors for the different parts of the process, which are largely due to the staining process but can be diminished by measuring ratios of compounds.
quality control, quantitative analysis

qualitative identification, HPTLC postchromatographic derivatization 3e

100 012 Elena MATEOS, V.L. CEBOLLA*, L. MEMBRADO, J. VELA, Eva GALVEZ, Muriel MATT, F.P. COSSIO (*Instituto de Carboquímica, CSIC, P.O. Box 549, 50080 Zaragoza, Spain; vcebolla@carbon.icb.csic.es): Coralyne cation, a fluorescent probe for general detection in planar chromatography. J. Chromatogr. A 1046 (2), 251-257 (2007). Fluorescence scanning densitometry of various analytes on HPTLC silica gel plates impregnated with a solution of coralyne cation, based on the increase or decrease, that the corresponding analyte induces on native coralyne emission at a given excitation wavelength. Compared to a procedure previously described for berberine cation, and Reichardt‘s dye probes, the sensitivity of coralyne in HPTLC detection of non-fluorescent, structurally different analytes (e.g. long-chain alkanes, alcohols, alkylbromides, neutral lipids) is superior.

HPTLC quantitative analysis, qualitative identification, comparison of methods, postchromatographic derivatization 3e

100 013 Gertrud MORLOCK*, Y. UEDA (*Institute of Food Chemistry, University of Hohenheim, Garbenstrasse 28, D-70599 Stuttgart, Germany): New coupling of planar chromatography with di-

rect analysis in real time mass spectrometry. J. Chromatogr. A 1043 (1-2), 243-251 (2007). Presentation of the coupling of planar chromatography with direct analysis in real time time-of-flight mass spectrometry (DART-TOF-MS) for the first time. By cutting the plate within a track led to substance zones positioned on the plate edge, the interested zones were directly introduced into the DART gas stream to obtain the mass signals instantaneously within seconds, giving the detectability in the very low ng/zone-range on the example of isopropylthioxanthone. The coupling was perfectly suited for identification and qualitative purposes, but for quantification of results the analytical response and the repeatability were strongly dependent from proper manual positioning of the HPTLC plate into the excited-state gas stream of the ion source. By using stable isotope-labeled standards the drawback can be overcome demonstrated with the example of caffeine, and the analytical response (R2 of 0.9892) and repeatability (RSD < ±5.4%, n = 6) were improved to a high extent. The spatial resolution by an in-house-built plate holder system was shown to be better than 3 mm; the decay of the signal was observed. Comparison of the efficacy of this new coupling to a plunger-based extraction device for HPTLC/electrospray ionization-MS. The detectability of latter showed to be down to the pg/zone-range, e.g. the limit of quantification for isopropylthioxanthone to be 100 pg/zone. The repeatability was comparable (RSD ± 6.7 %), however, without the need of internal standard correction, and the analytical response slightly better (R2 of 0.9983). The spatial resolution was 2 mm or 4 mm depending on the plunger head used.

quality control, HPTLC quantitative analysis, qualitative identification, comparison of methods 3f

100 014 V. PANCHAGNULA, A. MIKULSKIS, L. SONG, Y. WANG, M. WANG, Tanya KNUBOVETS, Elaine SCRIVENER, Eva GOLENKO, Ira KRULL, M. SCHULZ, H.E. HAUCK, W.F. PATTON* (*Biochemistry Department, PerkinElmer Life and Analytical Sciences, Waltham, MA 02451, USA; wayne.patton@perkinelmer.com): 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 (2D PEC/TLC). It was found that phosphopeptides migrate more slowly 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 distinguishing phosphopeptides based upon hydrophilicity in the second dimension, which permits a restricted region of the plate to be directly interrogated for the presence of phosphopeptides by MS. Discussion of peptide sequencing and identification of phosphopeptide from the plates by matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF)-MS and tandem MS.

quality control, HPTLC densitometry, quantitative analysis, qualitative identification, planar electrochromatography 3

100 015 P.W. PLOCHARZ*, T.H. DZIDO, P. SLAZAK, G.W. JOZWIAK, A. TORBICZ (*Department of Physical Chemistry, Medical University of Lublin, Lublin, Poland, 2 Department of Inorganic Chemistry, Medical University of Lublin, Lublin, Poland): Influence of sample application mode on performance of pressurized planar electrochromatography in completely closed system. J. Chromatogr. A 1170 (1-2), 91-100 (2007). Use of three modes of sample application on the chromatographic plate at present investigations of pressurized planar electrochromatography (PPEC) systems taking into special attention their influence on performance of the separating system: 1) application directly with microsyringe, 2) deposition of sample solution on scrap of adsorbent layer followed by location of this scrap on the chromatographic plate, 3) application with a commercially available aerosol applicator. All three modes were combined with prewetting of the chromatographic plates in order to accomplish equilibration of the stationary phase - mobile phase system. Best results were obtained when the plate was prewetted and when application was performed with commercially available aerosol applicator.

Pressurized planar electrochromatography 3c

100 016 S. WANGTHONG*, I. TONSIRIPAKDEE, T. MONHAPHOL, R. NONTHABENJAWAN, S. PATTANAARGSON WANICHWECHARUNGRUANG (* Department of Chemistry, Faculty of Science, Chulalongkorn University, Payatai, Bangkok 10330, Thailand): Post TLC developing technique for tyrosinase inhibitor detection. Biomed. Chromatogr. 21 (1), 94-100 (2006). Presentation of a post TLC developing technique to detect substances which can inhibit tyrosinase activity. The TLC plate is sprayed with tyrosinase and l-tyrosine solutions successively. A positive result is detected as white zone against a brownish-purple background. The method is suitable as a quick screening procedure for tyrosinase inhibitor detection, and as a guiding procedure for the isolation of tyrosinase inhibitors from mixtures or natural product extracts.

quantitative analysis, qualitative identification, postchromatographic derivatization 3e

4. Special techniques

100 017 K. DREISEWERD, J. MUETHING* (*Institut für Medizinische Physik und Biophysik, Westfälische Wilhelms-Universität Münster, Rbert-Koch-Str. 31, 48149, Germany; jm@uni-muenster.de): Structural characterization of gangliosides by HPTLC/IR-MALDI-o-TOF. CBS 97, 2-5 (2006). HPTLC of gangliosides on silica gel with chloroform - methanol - water 24:17:4 and addition of 2 mM CaCl2, after chamber saturation with filter paper for 3 h, over 80 mm, followed by drying for 5 min at room temperature. Detection by dipping in orcin solution (0.3 % (w/v) in 3 M H2SO4) followed by heating at 100 °C for 3 min. Alternative detection of GM3-bands by derivatization with primulin (0.02 % (w/v) in aceton - water 4:1). Quantitative determination by direct IR-MALDI-o-TOF-analysis. The limit of detection for GM3 was about 50 ng/zone.

pharmaceutical research, HPTLC 4e

100 018 H. LUFTMANN, M. ARANDA, Gertrud MORLOCK* (*Institute of Food Chemistry, University of Hohenheim, Stuttgart, Germany, gmorlock@uni-hohenheim.de): Automated interface for hyphenation of planar chromatography with mass spectrometry. Rapid. Commun. Mass. Spectrom. 21, 3772-3776 (2007). A new fully automated online interface to couple HPTLC with ESI-MS/MS is presented for the first time. Among the major features of this interface are the time required for analysis, precision, suited for normal and reversed-phase layers and all plate sizes and carriers, no post-chromatographic process is required, it can be coupled universally with all LC-MS ion sources without any adjustment or mass spectrometer modification, and the quantitative analysis can be performed without any internal standard with a given detectability at the low-nanogram and even picogram level. The validation results for caffeine quantification in energy drinks and pharmaceutical samples, without internal standard, proved the reliability of the interface and its usefulness for quantitative analysis with comparable results to those obtained by validated HPTLC-UV methods.

HPTLC quantitative analysis, comparison of methods 4e

100 019 A. ORINAK*, I. TALIAN, E.V. EFREMOV, F. ARIESE, Renata ORINAKOVA (*Institute of Chemistry Sciences, Department of Physical Chemistry, University of P. J. Safarik, Moyzesova 11, 041 54 Kosice, Slovak Republic): Diterpenoic acids analysis using a coupled TLC-surface-enhanced Raman spectroscopy system. Chromatographia 67 (3-4), 315-313 (2008). Investigation of two different chromatographic substrates and one interface for coupling surface-enhanced Raman spectroscopy (SERS) with TLC. A chromatographic thin layer, specially produced for RS measurements, and a monolithic silica thin layer were used. A typical TLC plate with a modified aluminium backplate foil on one side was used as an interface. As test analytes three biologically active diterpenes (gibberellic acid, abietic acid, and kaurenoic acid) were applied directly onto the surface, followed by the addition of silver colloid and measurements by SERS. The strongest signal (excitation at 514.5 nm) was obtained for gibberellic acid using a Raman treated thin layer where the enhancement factor value was determined to be 102. No useful SERS signals were observed when the monolithic silica layer was used. Similar SERS spectra on modified aluminium backplate were obtained for abietic acid and gibberellic acid and no SERS spectrum was obtained for kaurenoic acid.

HPTLC quantitative analysis 4e

5. Hydrocarbons and halogen derivatives

100 020 H. HEGEWALD (Lacrome Lda, Rua Cesar Batista 6 D, 7000 715 Evora, Portugal; lacrome@clix.pt): Chlorine-free mobile phase for determination of PAH in water extracts. CBS 98, 9-11 (2007). Quantitative HPTLC of polycyclic aromatic hydrocarbons (PAH) from water samples, on caffeine-impregnated silica gel, with isopropyl acetate in a precooled (-20 °C, 30 min) twin-trough chamber without chamber saturation over 70 mm at -20°C. After application the dry plate was first equilibrated in the solvent-free trough for 10 min at -20 °C. Qualitative HPTLC at room temperature in the horizontal developing chamber with isopropyl acetate - n-hexane 3:1 over 50 mm. Detection by dipping in paraffin - toluene 1:1 (for fluorescence enhancement). Quantitative determination by fluorescence measurement at UV 366/>400 nm. Qualitative evaluation under UV 366 nm. The method is based on the German standard DIN 38407-7 for quantitative determination of 6 PAH but uses isopropyl acetate as a chlorine-free solvent instead of dichloromethane.

environmental quality control, qualitative identification, quantitative analysis, HPTLC densitometry 5b

100 021 Beata JANOSZKA (Medical University of Silesia, Faculty of Medicine, Department of Chemistry, Jordana 19, 41-808 Zabrze, Poland; rokchemm@infomed.slam.katowice.pl): Densitometric TLC analysis of azaarenes in grilled meat. J. Planar Chromatogr. 20, 221-26 (2007). TLC of seven azaarenes, acridine, benzo(h)quinoline, benzo(a)acridine, benzo(c)acridine, dibenzo(a,c)acridine, dibenzo(a,j)acridine, and dibenzo(a,h)acridine, on RP-18 in a horizontal chamber with dichloromethane - n-hexane - 2-propanol 60:40:1. After drying visualization under UV light at 254 and 366 nm. Quantification by densitometric fluorescence measurement at 380 nm. Limits of determination were from 0.04 to 0.30 ng/zone.

food analysis, densitometry, quantitative analysis 5b

8. Substances containing heterocyclic oxygen

100 022 Magdalena BARTNIK*, K. GLOWNIAK, A. GROMEK (*Department of Pharmacognosy, Medical Plant Laboratory, Skubiszewski Medical University, Chodzki 1, 20-093 Lublin, Poland; mbartnik@pharmacognosy.org): TLC and HPLC analysis of the flavonoid glycosides in the aerial parts of Peucedanum tauricum Bieb. J. Planar Chromatogr. 20, 127-130 (2007). TLC of isorhamnetin-3-glucoside, isorhamnetin-3-rutinoside, kaempferol-3-rhamnosidoglucoside, isoquercitrin, rutoside, hyperoside on silica gel in horizontal chamber with ethyl acetate - methyl ethyl ketone - formic acid - water 5:3:1:1 or ethyl acetate - formic acid - water 9:1:1. Quantitation by scanning at 366 nm; detection by spraying with a 1 % methanolic solution of Natural Product Reagent A followed by a 4 % methanolic solution of polyethylene glycol 400.

herbal traditional medicine, qualitative identification, densitometry, quantitative analysis 8a

100 023 Agnieszka BAZYLKO*, A.K. KISS, J. KOWALSKI (*Department of Pharmacognosy, Faculty of Pharmacy, Medical University of Warsaw, 1 Banach Street, 02-097 Warsaw, Poland; oklyzab@farm.amwaw.edu.pl): Densitometric determination of flavonoids in methanolic and aqueous extracts of Epilobii angustifolii herba by use of HPTLC. J. Planar Chromatogr. 20, 53-56 (2007). HPTLC of flavonoids (quercetin glucuronide, hyperoside, isoquercitrin, quercetin galloyl galactoside, quercitrin) on silica gel with ethyl acetate - formic acid - water 136:5:6 in a horizontal chamber. Densitometric quantitfication of flavonoids at 350 nm.

traditional medicine herbal, HPTLC quantitative analysis, densitometry 8a

100 024 Josipa CVEK*, M. MEDIC-SARIC, I. JASPRICA, A. MORNAR (*Agency for Medicinal Products nd Medical Devices, Ksaverska cesta 4, 10000 Zagreb, Croatia; bebamms@pharma.hr): High-Performance Thin-Layer chromatographic analysis of the phenolic acid and flavonoid content of Croatian propolis samples . J. Planar Chromatogr. 20, 429-435 (2007). HPTLC of 3 phenolic acids (caffeic acid, p-coumaric acid, isoferulic acid) and 4 flavonoids (pinocembrin, pino-

cembrin-7-methyl ether, chrysin, tectochrysin) on silica gel with chloroform - methanol - formic acid 88:7:5 with chamber saturation. Detection by spraying with 1 % ethanolic aluminium chloride solution. Quantification by scanning densitometry in absorbance mode.

food analysis, HPTLC densitometry, quantitative analysis 8a

100 025 Renata NOWAK (Department of Pharmaceutical Botany, Medical University, 1 Chodzki Street, 20-093 Lublin, Poland; renata.nowak@am.lublin.pl): TLC fingerprinting analysis of the European dog rose. J. Planar Chromatogr. 20, 43-48 (2007). Two dimensional TLC of flavonoids (with quercetin 3-rhamnoside, quercetin 3-glucoside, quercetin 3-rutinoside, catechin, and gallic acid as markers) on cellulose with n-butanol - acetic acid - water 6:4:1 in the first direction and 15 % acetic acid in the second direction; TLC of phenolic acids on cellulose with toluene - methanol - acetic acid - acetonitrile 16:2:1:1 in the first direction and sodium formate - formic acid - water 10:1:200 in the second direction. Flavanols were separated on silica gel with chloroform - methanol - water 13:7:2 in the first direction and ethyl acetate - formic acid - acetic acid - water 75:3:2:20 in the second direction. Chromatograms were developed in a horizontal chamber after saturation for 10 min. Detection after drying by UV light at 254 and 366 nm. Detection also by spraying with 5 % aluminium chloride in methanol for flavonoids, with aqueous 5 % iron(III) chloride for gallic acid, 1 % diazosulfanilamide in acetone and 1 % vanillin in hydrochloric acid for flavanols. After spraying with vanillin solution plates were heated at 110 °C for 5 min and viewed in white light and, after 30 min, under UV light at 366 nm. Also TLC of flavonoids (astragalin, quercetin 3-galloylglucoside, rutin, hyperoside, quercitrin, kaempferol 3-rhamnoside on silica gel with ethyl acetate - methanol - formic acid - acetic acid - water 80:10:1:1:8. For an identity test natural product reagent, 0.5% diphenylborinic acid 2-aminoethylester in ethyl acetate, was used. After develoment the plates were heated at 100 °C for 3 min and immediately immersed in the NP reagent, then viewed under UV light at 366 nm and in white light.

pharmaceutical research, quality control, herbal, qualitative identification 8a

100 026 L. POBLOCKA-OLECH, Miroslawa KRAUZE-BARANOWSKA*, M. WIWART (*Department of Pharmacognosy, Medical University of Gdansk, Gen. J. Hallera 107 st., 80-416 Gdansk, Poland; krauze@amg.gda.pl): HPTLC determination of catechins in different clones of the genus Salix. J. Planar Chromatogr. 20, 61-64 (2007). HPTLC of flavonoids (catechin, epicatechin, gallocatechin, catechin gallate as standards) on RP-18 with acetonitrile - water - formic acid 10:40:3. The best separation of catechin and epicatechin was achieved by multiple gradient development with increasing concentrations of acetonitrile (from 20 to 22 %) in the water - formic acid mixture. UV detection at 282 and 500 nm (after derivatization with vanillin-phosphoric acid) for estimation of catechin content.

herbal traditional medicine, HPTLC densitometry quantitative analysis 8a

100 027 S. RASTOGI*, M.M. PANDEY, A.K.S. RAWAT (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Lucknow 226001, India; subharastogi1@ rediffmail.com): A new, convenient method for determination of mangiferin, an anti-diabetic compound, in Mangifera indica L.. J. Planar Chromatogr. 20, 317-320 (2007). HPTLC of mangiferin (a C-glucosyl xanthone) on silica gel with ethyl acetate - methanol - water - formic acid 20:2:2:1. Detection and quantification were performed densitometrically at 270 nm.

traditional medicine herbal, food analysis, densitometry, quantitative analysis, HPTLC 8a

10. Carbohydrates

100 028 Gertrud MORLOCK*, M. A. VEGA-HERRERRA (*University of Hohenheim, Institute of Food Chemistry, Garbenstrasse 28, 70599 Stuttgart, Germany; g.morlock@uni-hohenheim.de): Two new derivatization reagents for planar chromatographic quantification of sucralose in dietetic products. J. Planar Chromatogr. 20, 411-417 (2007). HPTLC of sucralose in dietetic products on

silica gel impregnated with 0.1 M dipotassium hydrogen phosphate solution, and on amino phase with acetonitrile - water 17:3. Also a mixture of sucralose, sucrose, glucose, fructose was separated on amino phases with acetonitrile - water 3:1. Detection by dipping in 2-naphthol sulfuric acid reagent and aniline diphenylamine ortho-phosphoric acid reagent, followed by heating at 120 °C. Post-chromatographic derivatization on aluminium-backed amino phases was performed by heating the plate 190 °C for 20 min. Evaluation under UV light at 366 nm. For fluorescence enhancement the amino phase was dipped into a 1:2 solution of paraffin in n-hexane. Densitometric evaluation by fluorescence measurement at 500 and 405 nm.

food analysis, densitometry, HPTLC quantitative analysis 10a

100 029 T. RANGANATHAN, P. KULKARNI* (*Food and Fermentation Technology Division, Mumbai University of Chemical Technology, Mumbai, India): A simple method for the analysis of trehalose using HPTLC. Food Chem. 77, 263-265 (2002). HPTLC of trehalose on silica gel, impregnated with phosphotungstic acid of pH 2.5, with n-butanol - pyridine - water 8:4:3. Detection by spraying with a solution of 6.5 mM N-(1-naphthyl)-ethylenediamine dihydrochloride in methanol, containing 3 % sulfuric acid. The hRf values of raffinose, trehalose, maltose, sucrose, glucose, and fructose were 30, 41, 46, 53, 55, and 59, respectively.

food analysis, toxicology, HPTLC quantitative analysis, densitometry 10a

100 030 Katarína REIFFOVÁ*, J. PODOLONOVICOVÁ, L. ONOFREJOVÁ, J. PREISLER, R. NEMCOVÁ (*Pavol Jozef Safárik University, Faculty of Natural Sciences, Institute of Chemistry, Department of Analytical Chemistry, Moyzesova 11, 041 54 Kosice, Slovak Republic; reiffova@kosice.upjs.sk): Thin-Layer Chromatography and matrix-assisted laser desorption/ionization mass spectrometric analysis of oligosaccharides in biological samples. J. Planar Chromatogr. 20, 19-25 (2007). TLC of fructooligosaccharides with raftilose as standard on silica gel impregnated with sodium acetate with butanol - acetic acid - water 2:2:1 in a saturated vertical twin-trough chamber with. Visualization with diphenylamine-aniline-phosphoric acid reagent (in acetone). The blue-pink spots were also detected by reflectance densitometry at 370 nm. MALDI-MS was used for analysis of fructooligosaccharides.

food analysis, clinical chemistry research, densitometry, quantitative analysis 10a

11. Organic acids and lipids

100 031 Fatma HELMY*, F. ROTHENBACHER, L. NOSAVANH, J. LOWERY, A. JURACKA (*Biology Department, Delaware State University, 1200 N. Dupont Highway, Dover DE 19901, USA; fhelmy@desu.edu): A comparative study of the phospholipid profiles of guinea pig cardiac muscle and bullfrog cardiac and thigh skeletal muscle, and their in-vitro differential deacylation by endogenous phospholipases. Thin layer chromatographic and densitometric analysis. J. Planar Chromatogr. 20, 209-215 (2007). TLC of phospholipids (with cardiolipin, phosphatidyl ethanolamine plasmologen and phosphatidyl cholin plasmologen as standards) on silica gel, prewashed with chloroform - methanol 2:1 and acetone, using one-dimensional TLC with 1-propanol - chloroform - ethyl acetate - methanol - water 50:50:50:21:18 and two-dimensional TLC with 1-propanol - chloroform - ethyl acetate - methanol - water 50:50:50:21:18 in the first direction and hexane - diethyl ether 1:1 in the second direction after hydrolysis with 1 % hydrochloric acid to reveal alkenylphospholipids. Detection by staining with thionine reagent resp. with leucofuchsin reagent. Densitometric scanning at 600 nm (for thionine) and at 560 nm (for leucofuchsin).
clinical chemistry research, densitometry

quantitative analysis, qualitative identification 11c

100 032 A. MIRZAIE, A. JAMSHIDI, S. W. HUSAIN* (*Chemistry Department, Faculty of Science, Science and Research Branch, Islamic Azad University, P. O. Box 14515-775, Poonak-Hesarak, Tehran, Iran; syedwhusain@yahoo.com): TLC quantification of methylparaben on an inorganic ion-exchanger in the presence of other food additives. J. Planar Chromatogr. 20, 141-143 (2007). TLC of methyl, ethyl, propyl p-hydroxybenzoate, p-hydroxybenzoic acid, benzoic acid,

sodium benzoate, butylated hydroxyanisol, and butylated hydroxytoluene on the inorganic ion exchanger stannic silicate in a twin-trough chamber with n-hexane - ethyl methyl ketone - acetic acid 80:20:3. Quantitation by scanning densitometry at 260 nm.

food analysis, qualitative identification, quantitative analysis, densitometry 11a

100 033 Magdalena WOJCIAK-KOSIORA (Department of Chemistry, Laboratory of Planar Chromatography, Medical University, Staszica 6, 20-081 Lublin, Poland): Separation and determination of closely related triterpenic acids by high performance thin-layer chromatography after iodine derivatization. J. Pharm. Biomed Anal. 45(2), 337-340 (2007). HPTLC of oleanolic acid and ursolic acid on silica gel impregnated with 1 % iodine solution in chloroform after sample application. Development with petroleum ether - ethyl acetate - acetone 82:18:1. Detection by spraying with a solution of 10 % sulfuric acid in ethanol followed by heating at 120 °C for 3 min. Quantification by densitometry in absorbance mode at 530 nm.

pharmaceutical research, HPTLC densitometry, quantitative analysis, qualitative identification 11

13. Steroids

100 034 L. AFINISHA, D. SOBAN, A. SUNDARESAN, C. ARUMUGHAN* (*National Institute for Interdisciplinary Science and Technology, Kerala, India, carumughan@yahoo.com): A new method for simultaneous estimation of unsaponifiable constituents of rice bran oil using HPTLC. J. Sep. Sci. 30, 2786-2793 (2007). HPTLC of unsaponifiable constituents of rice bran oil on silica gel in two stage separation: First separation with benzene - chloroform 12:1 for sterols, oryzanols, and tocols. Quantitative determination by absorbance measurement at 206 nm for sterols (1), 325 nm for oryzanols (2), and 297 nm for tocols (3). Second separation with petroleum ether - diethyl ether 50:1 for steryl esters (4), wax (5), and squalene (6). Detection by dipping in 5 % methanolic sulphuric acid followed by heating at 110 °C for 1 hour. Quantitative determination by absorbance measurement at 439 nm. The hRf values were 12 for (1), 21 for (2), 39 for (3), 36 for (4), 46 for (5), and 74 for (6). Linearity was between 150 and 1200 ng/zone for the first separation and between 400 and 1200 ng/zone the second separation. The limits of detection and quantification were 6 and 20 ng/zone for (1), 1 and 4 ng/zone for (2), 11 and 38 ng/zone for (3), 22 and 73 ng/zone for (4), 19 and 65 ng/zone for (5), and 3 and 10 ng/zone for (6), respectively. Intra-assay precision was between 0.52 and 1.94 % and inter-assay precision was between 0.87 and 2.27 %. Recoveries ranged from 93.5 to 101.9 %.

food analysis, HPTLC, quantitative analysis, densitometry 13c

100 035 L. JÄNTSCHI, S. HODISAN, C. CIMPOIU, A. HOSU, E. DARVASI, T. HODISAN* (*'Babes-Bolyai' University, Faculty of Chemistry and Chemical Engineering, 11 Arany Janos, 400028 Cluj-Napoca, Romania; thodisan@chem.ubbcluj.ro): modeling of thin-layer chromatographic separation of androstane isomers. J. Planar Chromatogr. 20, 91-94 (2007). Descripton of the modeling of TLC separation of androstane isomers to find the optimum mobile phase. A mathematical model was developed and tested. The model takes into account the interaction between solvents and uses a complex function for modeling. The proposed mathematical model gives results similar to those obtained by use of other optimization models, e. g. the Simplex and Prisma methods. TLC of 5alpha-androstan-3beta-ol, 5alpha-androstan-3alpha-ol, 5alpha-androstan-17beta-ol, 5beta-androstan-3alpha,17beta-diol, 5alpha-androstan-3beta,17beta-diol on silica gel in a saturated chamber using different mixtures of chloroform, acetone, and petroleum ether resulting in an optimum mobile phase composition of 55:19:26. Detection by spraying with 5 % ammonium molybdate and 5 % sulfuric acid in water and heating to 80 °C.

qualitative identification 13a

100 036 K. SHANKER, S.C. SINGH, S. PANT, P. SRIVASTAVA, A.K. YADAV, R. PANDEY, R.K. VERMA, M.M. GUPTA* (*Analytical Chemistry Division, Central Institute of Medicinal and Aromatic Plants, Lucknow, 226015, India): Quantitative TLC analysis of sterol (24β-ethylcholesta-5,22 E,25-triene-3β-ol) in Agnimantha (Clerodendrum phlomidis Linn). Chromatographia 67

(3-4), 268-274 (2008). HPTLC of 24β-ethylcholesta-5,22E,25-triene-3β-ol (ECTO) in the aerial part of Clerodendrum phlomidis (used as a chemical marker for the standardization of C. phlomidis plant extracts) on silica gel with chloroform - methanol 197:3. Detection by spraying with anisaldehyde reagent. Quantitative determination by densitometry in absorption mode at 650 nm. Linearity was between 150 and 400 ng/band with good correlation (r2 = 0.996).

quantitative analysis, qualitative identification, HPTLC densitometry 13c

100 037 Malgorzata STAREK*, J. KRZEK, S. MICHNIK (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Cracow, Poland; mstarek@interia.pl): TLC-densitometric analysis of β-sitosterol in pumpkin seed oil. J. Planar Chromatogr. 20, 327-330 (2007). TLC of β-sitosterol on silica gel with toluene - ethyl acetate - glacial acetic acid 15:4:1 with chamber saturation for 30 min. Visualization by spraying with anisaldehyde reagent and heating at 90 °C for 5 min. Densitometric quantitation at 525 nm.
food analysis, densitometry

quantitative analysis 13c

14. Steroid glycosides, saponins and other terpenoid glycosides

100 038 G. JANICSÁK*, E. TÓTH, I. MÁTHÉ (*Institute of Ecology and Botany of the Hungarian Academy of Sciences, Vácrátót, Alkotmány út, H-2163, Hungary; janicsak@botanika.hu): TLC-densitometric investigations of phenylpropanoid glycosides in black horehound (Ballota nigra L.). J. Planar Chromatogr. 20, 443-446 (2007). TLC of caffeoylmalic acid, forsythoside, and verbascoside on silica gel in an unsaturated chamber with formic acid - acetic acid - water - ethyl acetate 15:15:36:134. Detection by dipping into a 1 % methanolic solution of natural products reagent and heating for 10 min at 40 °C. The dried plates were subsequently dipped into a 5 % methanolic solution of polyethyleneglycol 400 and then heated as before. Quantitation by densitometry at 395 nm.

herbal, traditional medicine, densitometry, quantitative analysis 14

15. Terpenes and other volatile plant ingredients

100 039 Silvia GONZALEZ, J. GEISSER, Hannelore HEGER, D. LACHENMEIER* (*Chemisches und Veterinäruntersuchungsamt (CVUA) Karlsruhe, Weissenburgerstr. 3, 76187 Karlsruhe, Germany; Lachenmeier@web.de): Assessing the authenticity of absinthe. CBS 97, 6-7 (2006). HPTLC of absinthe (a beverage of the wormwood plant, Artemisia absinthium) on silica gel with acetone - acetic acid (98 %) - toluene - dichloromethane 1:1:3:5 over 70 mm. Detection by dipping in a solution of acetic anhydride - sulphuric acid - ethanol 1:1:10 followed by heating at 104 °C for 5 min. Quantitative determination by absorbance measurement at 554 nm. The hRf value of absinthin was 64 and selectivity regarding matrix was given. Linearity was between 0.1 and 10 g/L. The limit of detection and quantification for absinthin was 0.05 and 0.11 g/L, respectively. The precisions were better than 13.5 % (intraday) and 15.8 % (interday).

food analysis, quality control, herbal, HPTLC densitometry
quantitative analysis 15a

100 040 D. LACHENMEIER (Chemisches und Veterinaeruntersuchungsamt, Karlsruhe, Germany, lachenmeier@web.de): Assessing the authenticity of absinthe using sensory evaluation and HPTLC analysis of the bitter principle absinthin. Food Res. Int. 40, 167-175 (2007). HPTLC of absinthin in absinthe beverage (from the wormwood plant Artemisia absinthium L.) on silica gel with acetone - acetic acid (98 %) - toluene - dichloromethane 1:1:3:5. Detection by dipping into a solution of acetic anhydride - sulphuric acid - ethanol 1:1:10, followed by heating for 5 min at 104 °C. Quantitative determination by absorbance measurement at 554 nm. The hRf value of absinthin was 64 and selectivity regarding matrix was given. Linearity was between 0.1 and 10 g/L. The precision was better than 13.5 % (intraday) and 15.8 % (interday). The limit of detection and quantification for absinthin was 0.05 and 0.11 g/L, respectively.

toxicology, food analysis, HPTLC densitometry, quantitative analysis 15a

17. Amines, amides and related nitrogen compounds

100 041 H.A. KHAN (Department of Biochemistry, College of Science, Bld 5, King Saud University, P. O. Box 2455, Riyadh 11451, Saudi Arabia; khan_haseeb@yahoo. com): Thin-Layer Chromatographic separation of cadaverine and ornithine, and spectrophotometric quantification . J. Planar Chromatogr. 20, 231-233 (2007). TLC of cadaverine and ornithine on calcium sulfate (and silica gel) with methanol. Detection by spraying with 0.2 % ethanolic solution of ninhydrin and then heating the plates at 110 °C for 15 min. Quantitation by scraping the spot from the plate and measuring the absorbance at 550 nm. The lower limit of detection was found to be 0.75 µg/zone of ornithine.

comparison of methods, quantitative analysis 17a

100 042 B. MUSZYNSKA, A. MASLANKA, K. SULKOWSKA-ZIAJA, J. KRZEK* (*Department of Inorganic and Analytical Chemistry, Collegium Medicum, Jagiellonian University, 9 Medyczna Str. , 30-688 Kraków, Poland; jankrzek@cm-uj.krakow.pl): TLC-UV analysis of indole compounds and other nitrogen-containing bases in the fruiting bodies of Lactarius deterrimus. J. Planar Chromatogr. 20, 57-60 (2007). TLC of 5-methylcytosine, tryptamine, melatonin, tryptophan, indole-3-acetic acid, and indole on silica gel with 1-butanol - glacial acetic acid - water 12:3:5 and isopropanol - 25 % ammonia - water 8:1:1. Densitometry at 280 nm.

food analysis, densitometry, quantitative analysis, preparative TLC 17a

18. Amino acids and peptides, chemical structure of proteins

100 043 R. BHUSHAN*, H. BRÜCKNER, V. KUMAR, D. GUPTA (*Department of Chemistry, Indian Institute of Technology, Roorkee247 667, India; rbushfcy@iitr.ernet.in): Indirekt TLC resolution of amino acid enantiomers after derivatization with Marfey‘s reagent and its chiral variants. J. Planar Chromatogr. 20, 165-171 (2007). TLC of 17 DL amino acids derivatized with1-fluoro-2,4-dinitrophenyl-5-L-alaninamide, 1-fluoro-2,4-dinitrophenyl-5-L-phenylalaninamide, or 1-fluoro-2,4-dinitrophenyl-5-L-valinamide on silica gel with phenol - water 3:1 or on RP-18 with mobile phases containing acetonitrile and triethylamine-phosphate buffer (50 mM, pH 5.5) with saturation for 10-15 min.

qualitative identification 18a

100 044 A. MOHAMMAD*, S. LAEEQ (*Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh Muslim University, Aligarh 202002, India; mohammadali4u@rediffmail. com): Mixed surfactants enable separation of lysine from other essential amino acids in TLC on silica gel. J. Planar Chromatogr. 20, 423-427 (2007). TLC of 8 essential amino acids (L-lysine, L-valine, L-isoleucine, DL-threonine, L-methionine, L-leucine, DL-phenylalanine, DL-tryptophan) on silica gel with 17 mobile phases, of which the mixed aqueous surfactant solution Triton X-100 (0.001 mM) - sodium dodecyl sulfate, 0.081 mM - acetone 1:1:5 was identified as the best mobile phase for specific separation of lysine. Visualization by spraying with 0.3 % ninhydrin in acetone. Limit of detection was 0.5 µg/zone.

quality control, qualitative identification, biochemistry 18a

100 045 Iva REZIC*, T. REZIC, L. BOKIC (*Laboratory of Analytical Chemistry, Department of Applied Chemistry, Faculty of Textile Technology, University of Zagreb, Croatia; iva_rezic@net. hr): Optimization of the TLC separation of seven amino acids. J. Planar Chromatogr. 20, 173-177 (2007). TLC of seven amino acids (alanine, asparagine, cysteine, leucine, phenylalanine, serine, threonine) on microcrystalline cellulose with butanol - glacial acetic acid - water 60:19:21. Detection by spraying with 3 % ethanolic ninhydrin solution and heating. The performance of this mobile phase was confirmed experimentally. Optimization by use of the experimental design software packages Design-Expert 6 and Statistica.

qualitative identification 18a

22. Alkaloids

100 046 V. DIGHE, R.T. SANE, G. PAREKH*, V. GOKARN, O. DHOTRE (*Department of Chemistry, Ramnarain Ruia College, Matunga (East), Mumbai-400 019, India; gaurangparekh80@yahoo.co.in): HPTLC quantitation of camptothecin in Nothapodytes foetida (Wight) Sleumer stem powder. J. Planar Chromatogr. 20, 131-133 (2007). HPTLC of camptothecin (4-ethyl-4-hydroxy-1H-pyrano[3‘,4‘:6,7]indolizino[1,2b]quinoline-3,14(4H,12H)dione) on silica gel with toluene - acetonitrile - glacial acetic acid 65:35:1. Detection and quantitation were performed by densitometric scanning in fluorescence mode at 370 nm.

herbal, traditional medicine, densitometry, quantitative analysis, qualitative identification HPTLC 22

100 047 P. GHOSH, M. REDDY, R. SASHIDHAR* (*Department of Biochemistry, University College of Science, Osmania University, Hyderabad, India, sashi_rao@yahoo.com): Quantitative evaluation of sanguinarine as an index of argemone oil adulteration in edible mustard oil by high performance thin layer chromatography. Food Chem. 91, 757-764 (2005). HPTLC of dihydrosanguinarine (1), after its conversion to sanguinarine (2) as an index of argemone oil adulteration in edible mustard oil, on silica gel with hexane - acetone - methanol 16:3:1. The plate was irradiated under long wave UV light for 15 min to oxidize (1) to (2). Quantitative determination by absorbance measurement at 366 nm. The hRf values for (1) and (2) were 82 and 36, respectively. Linearity was between 5 and 300 ng/zone for (2). The limit of detection and quantification was 1 and 3 ng/zone. Recovery was between 79 and 82 %.

food analysis, toxicology, quantitative analysis, HPTLC densitometry 22

100 048 L.S. NAIR, S.N. MENON*, S. SHAILAJAN, M.M. BAING, R.T. SANE (*Therapeutic Drug Monitoring Laboratory, 194, Scheme No. 6, Road No. 15, Sion Koliwada, Sion (East), Mumbai-400 022 India; tdmlab@vsnl.net): Reversed-phase High-Performance Thin-Layer Chromatographic quantitation of mimosine from whole plant of Mimosa pudica L. J. Planar Chromatogr. 20, 49-51 (2007). HPTLC of mimosine (alpha-amino-3-hydroxy-4-oxo-1(4H)-pyridinepropanoic acid) on RP-18 with ethyl acetate - glacial acetic acid - water 60:10:17. Quantitation by densitometric scanning at 282 nm in absorbance mode. Limit of detection was 20 mg/g in the whole plant powder.

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

23. Other substances containing heterocyclic nitrogen

100 049 Tatjana DJAKOVIC-SEKULIC*, N. PERISIC-JANJIC (*Department of Chemistry, Faculty of Sciences, University of Novi Sad, Trg D. Obradovica 3, 21000 Novi Sad, Serbia; tanja@ih.ns.ac.yu): Comparative study of the retention of s-triazines on octadecylsilica, cyano, and amino HPTLC plates by use of a QSPR model. J. Planar Chromatogr. 20, 365-371(2007). Estimation of retention data by use of correlation equations and physicochemical properties is a useful tool in liquid chromatography. HPTLC of an homologous series of 9 s-triazines on RP-18, cyano-, and amino phase in unsaturated chambers with aqueous solutions of the organic solvents acetonitrile, tetrahydrofuran, and dioxane. After development the dried plates were examined under UV light at 254 nm.

HPTLC, qualitative identification 23e

100 050 Anamaria REVERDITO*, M.H. GARCÍA, A. SALERNO, O.A. LOCANI, I.A. PERILLO (*Department of Organic Chemistry, Faculty of Pharmacy and Biochemistry, University of Buenos Aires, 956 Junin St, 1113 Buenos Aires, Argentina; iperillo@ffyb.uba.ar): Detection of a covalent-ionic carbinolamine intermediate in aqueous media by SDTLC on silica gel plates. J. Planar

Chromatogr. 20, 227-230 (2007). When the components of a reaction mixture cannot be quantified by UV-visible spectrophotometry because of overlapping of their absorption bands, the components can be separated and quantified by spectrodensitometric thin-layer chromatography (SDTLC). As example serves an aminolysis reaction mixture. TLC of imidazolidine and 1-(p-chlorophenyl)-2-phenyl-3-methylimidazoline on silica gel in a twin-trough chamber saturated for 5 min with chloroform - methanol 4:1 for the first development to a distance 55 mm, and after drying development with benzene to a distance of 65 mm. Densitometric scanning at 260 nm in absorption mode.

qualitative identification, densitometry 23e

100 051 Marta STEFANIAK (Institute of Chemistry, Silesian University, 9 Szkolna St, 40-006 Katowice, Poland; m_stefaniak@op.pl): Lipophilic and physicochemical properties of metalloporphyrins separated by RP-TLC. J. Planar Chromatogr. 20, 361-364 (2007). TLC of metalloporphyrins with Zn(II), Cu(II), and Ni(II) cations on RP-18 with ethanol or ethanol - water 9:1 with chamber saturation for 30 min. Visual evaluation.

qualitative identification 23a

100 052 U. MALLAVADHANI*, A. SUDHAKAR, K. SATYANARAYANA, A. MAHAPATRA, W. LI, R. BREEMEN. (*Center for Herbal Drugs, Regional Research Laboratory, Orissa, India, uvmavadani@yahoo.com): Chemical and analytical screening of some edible mushrooms. Food Chem. 95, 58-64 (2006). HPTLC of nicotinic acid (1) and pyrazole-3(5)-carboxylic acid (2) of Volvariella volvacea on silica gel with chloroform - methanol 17:3 with one drop of formic acid added. Quantitative determination by absorbance measurement at 190 nm for (1) and 262 nm for (2). The hRf values for (1) and (2) were 30 and 40, respectively. Linearity was between 400 and 7000 ng/zone (1) and 200 and 2500 ng/zone for (2). The limits of detection and quantification were 50 and 400 ng/zone for (1) and 20 and 200 ng/zone for (2). Recoveries of both compounds were between 96 and 102 %.

food analysis, HPTLC quantitative analysis, densitometry 23e

27. Vitamins and various growth regulators

100 053 Anna NIESTROJ (Silesian University, Institute of Chemistry, 9 Szkolna Street, PL-40-006 Katowice, Poland; annaniestroj@wp.pl): Comparison of methods for calculation of the partition coefficients of selected tocopherols. J. Planar Chromatogr. 20, 483-486 (2007). New method for determination of log P for selected tocopherols, which makes use of Rf, topological indices, and log P according to Rekker. HPTLC of α-, β-, γ-, and δ-tocopherols on RP-18 with ethanol or ethanol - water 19:1. Detection by spraying with a mixture of equal volumes of solutions of dipyridyl in methanol (0.5%) and iron(III) chloride in methanol (0.2 %).

HPTLC, qualitative identification 27

28. Antibiotics, Mycotoxins

100 054 Shruti CHOPRA, S. MOTWANI* (*Department of Pharmaceutics, Faculty of Pharmacy, Jamia Hamdard, Hamdard Nagar, New Dehli 110 062, India; sanjay_bcp@rediffmail.com): Stability testing of gatifloxacin and analysis in polymeric nanoparticles. CBS 98, 5-7 (2007). HPTLC of gatifloxacin on silica gel in a saturated twin-trough chamber with n-propanol - methanol - ammonia 25% 50:10:9 over 80 mm. Quantitative determination by absorbance measurement at 292 nm. The hRf value of gatifloxacin was 60 and selectivity regardnig matrix was given. Linearity was between 400 and 1200 ng/band. The intraday and interday precision both were below 0.03 %. The limit of detection and quantification was 2.7 and 8.3 ng/zone, respectively. Recovery was between 99.2 and 101.9 %. The HPTLC method was suited to study gatifloxacin stability under different stress conditions according to ICH guidelines (acid, base, heat, oxidation, photostability).
pharmaceutical research, HPTLC

densitometry, quantitative analysis 28a

100 055 K. GRZYWNOWICZ*, M. NOWICKA (*University of Maria Curie-Sklodowska, Dept. of Biochemistry, Pl. M. C. Sklodowskiej 3, 20-031 Lublin, Poland; grzyw@hermes.umcs.lublin.pl): TLC identification of occupationally relevant mycotoxins. J. Planar Chromatogr. 20, 69-71 (2007). TLC of mycotoxins (aflatoxin B1, B2, G1, ochratoxin A, sterigmatocystin, chaetomins, roquefortine C, penicillic acid, trichothecenes) on silica gel, prewashed with methanol, with chloroform - xylene - acetone 6:3:1. Detection under UV and by spraying with 0.5 % anisaldehyde in sulfuric acid followed by heating at 110 °C for 10 min.

toxicology, environmental, qualitative identification 28b

100 056 S. NAGY, B. KOCSIS, T. KÖSZEGI, L. BOTZ* (*Pharmaceutical Institute and Central Pharmacy, Faculty of General Chemistry, University of Pécs, Honvéd u. 3., Pécs H-7624, Hungary; lajos.botz@aok.ptc.hu): Optimization of growth conditions for test fungus cultures ued in direct bioautographic TLC detection. 3. Test fungus: Candida albicans. J. Planar Chromatogr. 20, 385-389 (2007). Optimum conditions have been established for culture of the fungus Candida albicans for microbial detection of zones in direct bioautographic TLC. On the basis of the results with Candida albicans it can be differentiated between microbiostatic (bacteriostatic or fungistatic) and microbiocidal (bactericidal or fungicidal) effects on TLC plates. Aqueous solutions of amphotericin B and fluconazole were spotted on TLC plates coated with silica gel. After drying the plates were immersed in microbial suspensions with different optical densities. The viability and metabolic activity were evaluated by use of a bioluminescence ATP assay modified by Köszegi.

qualitative identification 28a

29. Pesticides and other agrochemicals

100 057 Sandra BABIC*, A. J. M. HORVAT, D. MUTAVDZIC, D. CAVIC, M. KASTELAN-MACAN (*Faculty of Chemical Engineering and Technology, Laboratory of Analytical Chemistry, Marulicev trg 19, 10000 Zagreb, Croatia; sandra.babic@fkit.hr): Sample preparation for TLC - genetic algorithm-based optimization of microwave-assisted extraction. J. Planar Chromatogr. 20, 95-99 (2007). TLC of atrazine and simazine on silica gel with hexane - chloroform - acetone 12:5:3 with chamber saturation. Detection under UV light at 254 nm. Also quantitative evaluation. The genetic algorithm proved to be an optimization procedure which can be successfully applied to optimization of microwave-assisted extraction experiments. Application of recovery experiments from spiked soil.

agricultural, quantitative analysis, qualitative identification 29d

100 058 H. CAO (Cao Haiqun), Y. YUE* (Yue Yongde), R. HUA (Hua Rimao), F. TANG (Tang Feng) Y. SHI (Shi Yanhong), X. WU (Wu Xiangwei), R. ZHANG (Zhang Rong), M. XIE (Xie Mengxing) (*International Center for Bamboo and Rattan, 100102 Beijing, China; yueyd@icbr.ac.cn): HPTLC analysis of octachlorodipropyl ether in insecticide formulations. J. Planar Chromatogr. 20, 341-345 (2007). HPTLC of octachlorodipropyl ether on silica gel prewashed with chloroform - methanol 1:1 in an unsaturated twin-trough chamber with toluene - acetic acid - water 20:20:1. Detection by spraying with silver nitrate - 2 M ethanolic potassium hydroxide, followed by heating for 30 min at 120 °C, overspraying with 1 % silver nitrate in 30 % nitric acid, and exposure to UV light for approximately 15 min. Densitometric evaluation of absorbance at 399 nm.
agricultural

toxicology, HPTLC, densitometry, quantitative analysis 29a

100 059 B.B. DAUNDKHAR, R.R. MAVLE*, M.K. MALVE, R. KRISHNAMURTHY (*Directorate of Forensic Science Laboratory, State of Maharashtra, Hans Bhugra Marg, Kalina, Vidyanagari, Santa Cruz (E), Mumbai 400 098, India; rajendramavle@gmail.com): Spectrophotometric and TLC detection reagent for the insecticides dichlorvos (DDVP) and diptrex (trichlorfon), and their metabolites, in biological tissue. J. Planar Chromatogr. 20, 217-219 (2007). TLC of dichlorvos and diptrex on silica gel with hexane - acetone 4:1. Detection by spraying with a reagent prepared from strong alkali, for example 10 % sodium hydroxide, and 0.5% aqueous sodium sulfide solution. The sensitivity is approx. 20 μg for both dichlorvos and diptrex.

toxicology, qualitative identification 29a

100 060 W. FAN (Fan Wei), Y. YUE* (Yue Yongde), F. TANG (Tang Feng), H. CAO (Cao Haiqun) (*International Center for Bamboo and Rattan, 100 102, Beijing, China; yueyd@icbr.ac.cn): Use of HPTLC for simultaneous determination of three fungicides in tomatoes. J. Planar Chromatogr. 20, 419-421 (2007). HPTLC of tricyclazole, thiram, and folpat on silica gel prewashed with methanol, with hexane - acetone 3:2 in an unsaturated twin-trough chamber. Densitometric evaluation at 235 nm. The limit of detection was 12, 30, and 40 ng/zone, for tricyclazole, thiram, and folpat respectively.

food analysis, HPTLC, densitometry, quantitative analysis 29e

100 061 H.S. RATHORE*, C. VARSHNEY (*Department of Applied Chemistry, Z. H. College of Engineering and Technology, Aligarh Muslim University, Aligarh-202002, India; hsrathore2003@yahoomail.com): Chromatographic behavior of dithiocarbamate fungicides on cellulose plates. J. Planar Chromatogr. 20, 287-292 (2007). TLC of mancozeb, NaDDC, propineb, ziram, and zineb on cellulose or cellulose impregnated with heavy metal salts with mobile phases prepared from water, n-butyl acetate, isopropanol, and lauryl sulfate. Visualization with iodine vapor. Plates were also coated with cereal flour, and with mixtures of cellulose and cereal flour.

agricultural, toxicology, qualitative identification 29c

100 062 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081 Lublin, Poland; tomasz.tuzimski@am.lublin.pl): Separation of multicomponent mixtures of pesticides by graft thin-layer chromatography on connected silica and octadecyl layers. J. Planar Chromatogr. 20, 13-18 (2007). Graft TLC separation of 28 pesticides (aziprotryne, fenvalerate, desmetryn, terbutryn, pyriproxyfen, benzthiazuron, fluoroglycofen-ethyl, bensultap, benalaxyl, thiabendazole, metalaxyl, tetramethrin, imazalil, atrazine, chlorfenvinphos, methoxychlor, carbaryl, alachlor, bromopropylate, captan, diuron, tetradifon, napropamide, metribuzin, metamitron, p,p‘-DDE, dinoseb, monolinuron) on connected layers - silica and octadecyl silica wettable with water, achieved by two dimensional planar chromatography using a non-aqueous mobile phase in the first dimension and an aqueous reversed-phase mobile phase in the second dimension. HPTLC on silica gel with 1) ethyl acetate - n-heptane 1:4 or 3:7 in the first dimension and, after cutting into strips, connection with RP 18 plates and transfer with methanol, with 2) methanol - water 3:2 or 3:1 in the second dimension. Detection under UV light at 254 or 366 nm.
agricultural, HPTLC

qualitative identification 29

30. Synthetic and natural dyes

100 063 Claudia CIMPOIU*, A. HOSU, R. BRICIU, V. MICLAUS (*“Babes-Bolyai“ University, Faculty of Chemistry and Chemical Engineering, 11 Arany Janos, 400028 Cluj-Napoca, Romania; ccimpoiu@chem.ubbcluj.ro): Monitoring the origin of wine by reversed-phase thin-layer chromatography. J. Planar Chromatogr. 20, 407-410 (2007). TLC of the color pigments from different sorts of red wine (Cabernet Sauvignon, Merlot, and Burgundy) on RP-18 with acetonitrile - water - formic acid 20:29:1 in a saturated chamber. Evaluation in visible light and under UV light at 366 nm, and by spraying with a methanolic solution of 0.5 mg/mL DPPH (2,2.diphenyl-1-picrylhydrazyl). Densitometric evaluation. RP-TLC is a tool for monitoring wine, for identification of the origin, and for detection of adulteration.

food analysis, qualitative identification, densitometry 30b

100 064 T. CSERHÁTI (Institute of Materials and Environmental Chemistry, Chemical Research Center, Hungarian Academy of Sciences, P. O. Box 17, 1525 Budapest, Hungary; tevi@chemres.hu): Study of the absorption characteristics of a zeolite support in normal and reversed-phase thin-layer chromatography. J. Planar Chromatogr. 20, 381-384 (2007). Study of the retention behavior of 36 synthetic dyes in adsorption and reversed-phase TLC on zeolite layers with n-hexane, te-

trahydrofuran, and bidistilled water. Significant linear correlations were found between the retention of the dyes chromatographed with the different mobile phases, proving the regular retention behavior of the analytes. No linear relationship was found between the physicochemical properties of the dyes and their retention, suggesting the separation capacity of zeolite differs markedly from that of silica and silica coated with hydrophobic ligands.

30a

100 065 N. EL-SHAER*, J. BADR, M. ABOUL-ELA, Y. GOHAR (*Department of Pharmacognosy, Faculty of Pharmacy, University of Alexandria, Alexandria, Egypt, gihan96@hotmail.com): Determination of lawsone in henna powders by high performance thin layer chromatography. J. Sep. Sci. 30, 3311-3315 (2007). HPTLC of lawsone in the leaves of Lawsonia alba on silica gel with chloroform - methanol 17:3. Quantitative determination by absorbance measurement at 334 nm. The hRf value of lawsone was 40 and selectivity regarding matrix was given. Linearity was between 100 and 1000 ng/zone. The precision was 1.72 % and recovery (by standard addition) was 98.8 %.

quality control, cosmetics, HPTLC, quantitative analysis, densitometry 30b

32. Pharmaceutical and biomedical applications

100 066 J.N. ABRAHAM*, S.L.PRABHU, S.G. VASANTHARAJU, C. DINESH KUMAR, A. SHIRWAIKAR (*Manipal College of Pharmaceutical Science, Karnataka, India): Stability Indicating HPTLC method for the Determination of Granisetron Hydrochloride in Bulk & Pharmaceutical dosage form. 59th Indian Pharmaceutical Congress F-95, 414, (2007). HPTLC of granisetron hydrochloride on silica gel aluminium plates with chloroform - methanol 4:1, with 0.1 mL of ammonia. The hRf value of granisetron hydrochloride was 42. Densitometry in absorbance mode at 301 nm. Linearity was between 400 and 1600 ng/zone. The limit of detection and quantification was 80 ng and 160 ng/zone, respectively. The drug was subjected to acid and alkali hydrolysis, oxidative and thermal degradation. The degration products were well separated from the main compound.

pharmaceutical research, HPTLC, densitometry, quantitative analysis 32a

100 067 P.B. ASWAR*, P.S. GANGANE, R.D. JAWARKAR (*I.B.S.S.B'S. College of Pharmacy, Malkapur, Buidhana, Maharashtri, India): Separation of plant Constituents form Caesalpinia Bonduc (L.) Roxb by HPTLC method. 59th Indian Pharmaceutical Congress C-100, 248, (2007). An HPTLC method has been developed for the estimation of flavonoids tannin and saponin in Caesalpinia bonduc. The dried powdered leaves of the plant were extracted with methanol and used for evaluation. HPTLC on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 20:2:2:5 for flavonoids, chloroform - glacial acetic acid - methanol - water 16:8:3:2 for saponin, and ethyl acetate - toluene - formic acid 6:6:1 for tannin. Detection by spraying with 5 % methanolic sulphuric acid for saponin, 5 % alcoholic aluminium chloride solution for flavonoids, and 5 % ferric chloride solution for tannin. Densitometric evaluation at 254 nm and 366 nm.

pharmaceutical research, traditional medicine, HPTLC densitometry, postchromatographic derivatization, qualitative identification, quantitative analysis 32c

100 068 S. BABOOTA*, M. FAIYAZUDDIN, S. AHMAD, J. ALI, A. AHUJA, A. KUMAR (*Faculty of Pharmacy, Jamia Hamdard, New Delhi, India): A novel & validated HPTLC method for the analysis of Cymbopogan citratus. 59th Indian Pharmaceutical Congress F-127, 420, (2007). HPTLC of citral (active constituent of Cymbopogan citratus) on silica gel with toluene - ethyl acetate 17:3. Detection by spraying with vanillin-sulfuric acid reagent. Densitometric evaluation at 595 nm. The hRf value of citral was 55. Linearity was between 1 and 10 ng/zone. The citral content in Cymbopolon citratus was found to be 67 - 81 %.

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

100 069 S.B. BAGADE, D.B. MESHRAM, M.R. TAJNE* (*Department of Pharmaceutical Sciences, R. T. M. Nagpur University Campus, Nagpur, Maharashtra, India): Simultaneopus estimation of aceclofenac, paracetamol and chlorzoxazone in fixed dose combination tablet by HPTLC. 59th Indian Pharmaceutical congress F-146, 424, (2007). HPTLC of paracetamol, acetofenac and chlorzoxazone on silica gel with toluene - chloroform - methanol - ethyl acetate 6:2:2:1. Densitometric quantification at 225 nm. The hRf values of aceclofenac, paracetamoland chorzoxazole was 22, 42, and 73, respectively. Linearity was between 700-2400, 1000-3200, and 300-1000 ng/zone for aceclofenac, paracetamol and chorzoxazole, respectively. Recovery was between 100.5 and 101.5 % for all compounds.

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

100 070 S. BANERJEE (Department of BIOtECHNOLOGY, KOLKATA, WEST BENGAL, INDIA): Expression pattern study of important flavonoids and sapogenins in plants and in callus culture of the medicinal plant Calendula officinalis. 59th Indian Pharmaceutical congress C-323, 302,(2007). TLC of flavonoids and sapogenins from different plant parts (leaves and petals) of Calendula officinialis on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:20, and chloroform - glacial acetic acid - methanol - water 15:8:3:2.

herbal, HPTLC, densitometry 32e

100 071 P. BANERJI*, A.K. MAJI, D. MUKHARJEE, P.K. MUKHARJEE (*Ulyssses Pharamaceuticals Pvt. Ltd. West Bengal, INDIA): Chromatographic evaluation of a gastroprotective phytoformulation. 59th Indian Pharmaceutical congress C-299, 296, (2007). The gastroprotective drug Gastse was prepared by mixing standardized alcoholic extracts of Capsicum annum, Chelidonium majus, Strychnos nux vomica, and Arsenicum album. HPTLC of Gastse on silica gel with n-hexane - ethyl acatate 52:6. Evaluation at UV 254 nm and 366 nm. Densitometric determination at 200 nm and 400 nm.

herbal, HPTLC, densitometry 32e

100 072 D.T. BAVISKAR*, S.C. JAGDALE, N.O. GIRASE, A.Y. DESHPANDE, D.K. JAIN (*M. A. H. College of Pharmacy, Dhule, India): Determination of valdecoxib from its bulk drug and pharmaceutical preparations by HPTLC. Indian Drugs 44(10), 734 (2007). HPTLC of valdecoxib on silica gel with toluene - ethyl acetate 1:1. Quantitative evaluation by densitometry at 262 nm. Valdecoxib was well separated from rofecoxib. Linearity was between 800 and 1000 ng/zone. Recovery was 98.9 %.

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

100 073 Agnieszka BAZYLKO*, Anna K. KISS, J. KOWALSKI (*Department of Pharmacognosy and Molecular Basis of Phytotherapy, Faculty of Pharmacy, Warsaw Medical University, ul. Banacha 1, 02-097 Warszawa, Poland): High-performance thin-layer chromatography method for quantitative determination of oenothein B and quercetin glucuronide in aqueous extract of Epilobii angustifolii herba. J. Chromatogr. A 1173 (1-2), 146-150 (2007). HPTLC of oenothein B and quercetin glucuronide in aqueous extract of Epilobii angustifolii herba on RP-18 W phase with 1) 25 % acetonitrile in water (with 50 mM H3PO4) over 80 mm for oenothein B, and 2) with acetonitrile over 40 mm for quercetin glucuronide. Quantification of oenothein B and quercetin glucuronide by densitometry at 270 and 350 nm, respectively. Linearity was between 1.14 and 2.28 µg/zone for oenothein B and 77 and 691 ng/zone for quercetin glucuronide. Aqueous extract of Epilobii angustifolii herba contained 152.46 ± 4.92 mg/g oenothein B and 22.07 ± 1.38 mg/g quercetin glucuronide.

pharmaceutical research, quality control, herbal, qualitative identification, HPTLC densitometry comparison of methods, quantitative analysis 32c

100 074 F. BEGUM*, R. SULTANA, S. KHANAM (*AL-AMEEN COLLEGE OF PHARMACY, BANGALORE, KARNATAKA, INDIA): Validation and application of HPTLC methods for estimation of curcumin and 6-gingerol. 59th Indian Pharmaceutical congress C-325, 303, (2007). HPTLC of curcumin and 6-gingerol in Curcuma longa and Zingiber officinalis on silica gel with chlorofom - ethanol - glacial acatic acid 24:1:2 for curcumin, and n-hexane - ethanol 2:3 for 6-gingerol. Densitometry at 430 nm for curcumin and 280 nm for 6-gingerol. The developed method was found suitable for routine analysis of extract and marketed formulations.

herbal, densitometry, HPTLC, quantitative analysis 32e

100 075 P. BHANDARI, N. KUMAR, A. GUPTA, B. SINGH*, V. KAUL (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology, Palampur, India, bikram_npp@rediffmail.com): A rapid RP-HPTLC densitometry method for simultaneous determination of major flavonoids in important medicinal plants. J. Sep. Sci. 30, 2092-2096 (2007). HPTLC of flavonoids in Bauhinia variegata, Bacopa monnieri, Centella asiatica, Ginkgo biloba, Lonicera japonica, Rosa bourboniana, Rosa brunonii, and Rosa damascena on RP-18 with two-fold development with water (5 % formic acid) - methanol 7:3 and water (5 % formic acid) - methanol 1:1 as mobile phases. Quantitative determination by absorbance measurement at 280 nm. The hRf values of apigenin (1), quercetin (2), rutin (3), luteolin (4), and quercitrin (5) were 19, 29, 34, 51, and 63, respectively. Linearity was between 150 and 800 ng/zone for (1) and (3) and between 200 and 1000 ng/zone for (2), (4) and (5). The limits of detection and quantification for (1) - (5) were 30 and 166 ng/zone, 40 and 200 ng/zone, 20 and 150 ng/zone, 40 and 200 ng/zone, and 40 and 200 ng/zone, respectively. Recovery was between 97 and 99.8% for (1) - (5).

herbal, HPTLC, quantitative analysis, densitometry 32e

100 076 R. BHUSHAN*, D. GUPTA (*Department of Chemistry, Indian Institute of Technology, Roorkee-247 667, India): Thin-layer chromatography separation of enantiomers of verapamil using macrocyclic antibiotic as a chiral selector. Biomed. Chromatogr. 19 (6), 474-478 (2005). HPTLC of enantiomers of verapamil on silica gel impregnated with vancomycin, a macrocyclic antibiotic, with acetonitrile - methanol - water 6:1:1. Detection by exposure to iodine vapors.

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

100 077 Merce BONFILL*, Susanna MANGAS, Rosa CUSIDO, Lidia OSUNA, M. TERESA PINOL, J. PALAZON (*Laboratorio de Fisiología vegetal, Facultad de Farmacia, Universidad de Barcelona, Avda. Diagonal 643, E-08028 Barcelona, Spain): Identification of triterpenoid compounds of Centella asiatica by thin-layer chromatography and mass spectrometry. Biomed. Chromatogr. 20 (2), 151-153 (2005). TLC of the four principal triterpenoid components of Centella asiatica on silica gel plates with the combination of ethyl acetate and methanol. Detection by spraying with anisaldehyde solution, followed by heating at 100 °C for 5 min. Evaluation under white light. The developed method is a modification of the method described in the European Pharmacopoeia (5th edn). Confirmation of the separated compounds by MALDI-TOF mass spectrometry.

HPTLC, quantitative analysis 32e

100 078 V.V. BYAHATTI*, K.V. PAI, A.M. KHAN, Marina D'SOUZA (*Devaki Amma memorial college of Pharmacy, Kuvempu University, Chelembra, Kerala, India): Antilithiatic activity of a phenolic compound from Bergenia Ciliata - A preliminary study. 59th Indian Pharmaceutical congress E-243, 283, (2007). HPTLC of Bergenia ciliata leaves and rhizomes successively (Soxhlet) extracted with petroleum ether (40-60°C), chloroform, n-butanol, and ethyl acetate, on silica gel with ethyl acetate - glacial acetic acid - formic acid - water 128:50:50:122. Evaluation under UV 254 nm.

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

100 079 M. CHALOOSI*, M. AMOLI-DIVA, F. GHOLAMIAN, S. MOZAFFARI (*Faculty of Chemistry,

Tarbiat Moalem University, 49 Mofateh Avenue, Tehran, Iran): Separation and determination of nitroguanidine and guanidine nitrate by HPTLC. Chromatographia 66 (3-4), 295-296 (2007). HPLC of nitroguanidine and guanidine nitrate on silica gel layers with dioxane - tetrahydrofuran 1:1. Detection under UV 210 nm for guanidine nitrate and 265 nm for nitroguanidine. Quantification by absorbance densitometry using peak area calibration. The method was used for separation and quantification of the compounds for online and off-line quality control of synthesis.
pharmaceutical research

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

100 080 D.S. CHAUHAN*, P. DHUMAL, R. DANG, K.K. MUEEN AHMED, R. SULTANA (*Al-Ameen College of Pharmacy, Bangalore, Karnataka, India): Comparative study of mature and immature tubers of Imomoea mauretania by using HPLC and HPTLC analysis. 59th Indian Pharmaceutical congress C-305, 297, (2007). Phytoconstituents of mature and immature tubers of Ipomoea mauritiana (methanolic and aqueous extracts) have been studied. HPTLC on silica gel with chloroform - methanol - formic acid 6:3:1. Detection by spraying with vanilin - sulphuric acid reagent. Densitometric evaluation at 365 nm. Mature tubers were found to contain higher concentration of phytoconstiuents than immature tubers.

pharmaceutical research, traditional medicine, herbal, qualitative identification, postchromatographic derivatization, HPTLC, comparison of methods, densitometry, quantitative analysis 32e

100 081 Shruti CHOPRA*, F. AHMAD, S. MOTWANI (*Department of Pharmaceutics, Faculty of Pharmacy, Jamia Hamdard, Hamdard Nagar, New Dehli 110 062, India; shrutichopra21@yahoo.com): Validated analysis of the biomarker trigonelline. CBS 97, 9-11 (2006). HPTLC of trigonelline in fenugreek (Trigonella foenum-graecum) on silica gel in a saturated twin-trough chamber with n-propanol - methanol - water 4:1:4 over 80 mm. Quantitative determination by absorbance measurement at 269 nm. The hRf value of trigonelline was 46 and selectivity regarding matrix was given. Linearity was between 100 and 1200 ng/zone. The inter- and intraday precision was below 1 %. The limit of detection and quantification was 2.3 and 7.6 ng/zone, respectively. Recovery (by standard addition) was 99 - 101 %.

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

100 082 T. CSERMELY, G. PETROIANU, K. KUCA, J. FÜRÉSZ, F. DARVAS, Z. GULYÁS, R. LAUFER, Huba KALÁSZ* (*Department of Pharmacology and Pharmacotherapy, Semmelweis University, 1089 Budapest, Nagyvárad tér 4, Hungary; huba.kalasz@gmail.com): TLC of quaternary pyridinium aldoximes, antidotes of organophosphorus esterase inhibitors. J. Planar Chromatogr. 20, 39-42 (2007). Displacement TLC of quaternary pyridinium aldoximes (e. g. pralidoxime and obidoxime) on silica gel impregnated with paraffin oil by continous development with 10 % paraffin oil in n-hexane for 18 h with water - acetone - hydrochloric acid 8:1:1. Detection under UV light at 254 nm.

toxicology, clinical chemistry research, quantitative analysis 32a

100 083 K. DALVI*, V. VAIDYA, S. MENON, M. KEKARE, W. SHAH (*Therapeutic Drug Monitoring Laboratory, 194, Scheme No. 6, Road No. 15, Sion Koliwada, Sion (East), Mumbai-400 022, India; tdmlab@vsnl.net; vaidya_vikas@yahoo.com): Thin-layer chromatographic determination of α-amyrin in the bark of Mallotus philippensis Lamk. J. Planar Chromatogr. 20, 279-281 (2007). HPTLC of α-amyrin on silica gel with dichloromethane - toluene 19:1 in a twin-trough chamber saturated for 5 min. Visualization by spraying with anisaldehyde reagent and heating for 10 min at 105 °C. Quantitation by densitometry at 586 nm.

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

100 084 A.A. DATE*, M. S. NAGARSENKER (*Department of Pharmaceutics, Bombay College of Pharmacy, Kalina, Santacruz (E.), Mumbai, 400098, India): HPTLC determination of cefpodoxime proxetil in formulations. Chromatographia 66 (11-12), 605-608 (2007). HPTLC of both isomers of cefpodoxime proxetil on silica gel plate with toluene - acetonitrile 3:2. Quantification by densitometry at 234 nm. The limit of detection and quantification was 150 and 400 ng/zone, respectively.

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

100 085 K. DHALWAL, V. SHINDE*, K. MAHADIK, A. NAMDEO (*Poona College of Pharmacy, Bharati Vidyapeeth Deemed University, Maharashtra, India, vaibhavshinde2@rediffmail.com): Rapid densitometric method for simultaneous analysis of umbelliferone, psoralen, and eugenol in herbal raw materials using HPTLC. J. Sep. Sci. 30, 2053-2058 (2007). HPTLC of umbelliferone (1), psoralen (2), and eugenol (3) in the dried fruit pulp of Aegle marmelos and in the fruit of Trachyspermum ammi and Foeniculam vulgare on silica gel with toluene - methanol 19:1. Quantitative determination by absorbance measurement at 331 nm for (1), 304 nm for (2), and 280 nm for (3). The hRf values were 30, 58, and 70 for (1), (2), and (3), respectively. Linearity was between 1 and 5 ng/zone, 16 and 96 ng/zone, and 200 and 1000 ng/zone for (1), (2), and (3), respectively. The limits of detection and quantification were 0.8 and 1.2 ng/zone for (1), 8 and 16 ng/zone for (2), 60 and 150 ng/zone for (3), respectively. Recoveries were 98.9 %, 100.1 %, and 99.3 %, for (1), (2), and (3) respectively.

herbal, quality control, HPTLC, quantitative analysis 32e

100 086 V.D. DHAVALE*, P.N. RANJANE, S.V. GANDHI, K.G. BOTHARA (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): HPTLC method for simultaneous determination of escitalopram oxalate & clonazepam in combined tablet dosage form. 59th Indian Pharmaceutical Congress F-94, 413, (2007). HPTLC of escitalopram oxalate and clonazepam in combined tablet dosage form on silica gel aluminium plate with toluene - ethyl acetate - triethylamine 7:3.5%:3. Quantitative determination by densitometric scanning at 258 nm. The calibration curve was linear over a range of 250 and 2500 ng/zone for escitalopram oxalate, and 50 and 500 ng/zone for clonazepam.

pharmaceutical research, HPTLC, densitometry, quantitative analysis 32a

100 087 V.V. DIGHE. G.M. PATHAK*, K.M. TULPULE, V.N. GOKAM (*Department of Chemistry, S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai 400 019, India; gayatribonds@yahoo.com; v2gayatri@gmail.com): HPTLC method for quantification of apigenin in the dried root powder of Gmelina arborea Linn. J. Planar Chromatogr. 20, 179-182 (2007). HPTLC of apigenin on silica gel with chloroform - acetone - formic acid 76:16:8 in a twin-trough chamber saturated for 30 min. Quantification by densitometric scanning at 340 nm.

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

100 088 R.P. DIXIT*, C.R. BARHATE, M.S. NAGARSENKER (*Department of Pharmaceutics, Bombay College of Pharmacy, Kalina, Santacruz (E), Mumbai, 400 098, India): Stability-indicating HPTLC method for simultaneous determination of ezetimibe and simvastatin. Chromatographia 67 (1-2), 101-107 (2008). HPTLC of simvastatin and ezetimibe on silica gel with n-hexane - acetone 3:2. Quantification by densitometry in absorbance mode at 234 nm. The hRf value of simvastatin was 39 and of ezetimibe 50. Linearity was between 200 and 1600 ng/spot with correlation coefficients r2 = 0.9917 for simvastatin and r2 = 0.9927 for ezetimibe. Limits of detection and quantitation were 25 and 150 ng per band, respectively. For investigation of stability simvastatin and ezetimibe were subjected to acid, pH 6.8 phosphate buffer, oxidation, dry heat, and wet heat. The degradation products were well resolved from the pure drug, therefore the method could be effectively used for stability-indicating analysis.

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

100 089 P. G. SHETTY*, K. V. MANGAONKAR, R. T. SANE, K. K. JARIPATKE, S. SINGH (*S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai-19, India; prabhagshetty@rediffmail.com and prabhashetty@hathway.com): Pharmacokinetic analysis of ursolic acid in Alstonia scholaris R. Br. by high-performance thin-layer chromatography. J. Planar Chromatogr. 20, 117-120 (2007). HPTLC of ursolic acid (3beta-hydroxyuro-12-enoic acid) on silica gel prewashed with methanol in a twin-trough chamber with toluene - ethyl acetate - triethylamine - methanol 7:2:1:1. After derivatization with Liebermann-Burchard reagent the chromatograms were evaluated densitometrically at 366 nm in the fluorescence mode.

clinical chemistry research, HPTLC, densitometry, quantitative analysis 32e

100 090 S.P. GANDHI*, C.R. SHAH, N.J. SHAH, D.R. PATEL, B.N. SUHAGIA (*Shri B.M. Shah College of Pharm. Edu. and Res. Gujrat, India): Method development, validation and determination of study of sumatriptane succinate in bulk poder and tablet forms by RP-HPLC and HPTLC methods. 59th Indian Pharmaceutical congress F-9, 392, (2007). HPTLC of sumatriptan succinate in bulk and tablet formulations on silica gel with methanol - water - glacial acetic acid 40:80:1. Densitometric evaluation at 230 nm.

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

100 091 S.V. GANDHI*, S.S. SABNIS, S.I. KHAN, R.T. JADHAV (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): High performance thin layer chromatographic determination of rabeprazole sodium & domperidone in combined dosage form. 59th Indian Pharmaceutical Congress F-83, 410, (2007). HPTLC of rabeprazole sodium and domperidone on silica gel with toluene - acetone - methanol 9:9:1. The hRf value of domperidone was 32 and of rabeprazole sodium 53. Densitometry at 285 nm. Lienarity was between 50 and 800 ng/zone for both compounds. The method was found suitable for routine analysis of formulations.

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

100 092 A. GOEL*, G.N. SINGH, F.J. AHMED, R.M. SINGH, R. GOEL (*Central Indian Pharmacopoeia Laboratory, Govt. Of India, Ministery of Health and Family Walfare, Ghaziabad, Uttar Pradesh, India): Development and validation of HPTLC method for determination of 6-gingerol in herbal extracts. 59th Indian Pharmaceutical congress F-220, 442, (2007). HPTLC of 6-gingerol in herbal extracts on silica gel with n-hexane - ethyl acetate - ammonia 14:5:1 in a chamber saturated for 45 min. Densitometric evaluation at 254nm. The hRf value of 6-gingerol was 52. The method was linear in the range of 100 - 1200 ng/zone.

herbal, HPTLC, densitometry, quantitative analysis 32e

100 093 D.E. GRAY*, D. MESSER, A. PORTER, B. HEFNER, D. LOGAN, R.K. HARRIS, A.P. CLARK, J.A. ALGAIER, J.D. OVERSTREET, C.S. SMITH (*Midwest Research Institute, 425 Volker Blvd, Kansas City, MO 64110, USA; dgray@mriresearch.org): Analysis of flavonol aglycones and terpenelactones in Gingko biloba extract: A comparison of high-performance thin-layer chromatography and column high-performance liquid chromatography. J. Assoc. Off. Anal. Chem, 90, 1203-1209 (2007). HPTLC of terpenelactones (total bilobalide, gingkolide A, and gingkolide B) on prewashed and sodium acetate preimpregnated silica gel with toluene - ethyl acetate - acetone - methanol 50:25:25:3 or ethyl acetate - hexane 9:1; also HPTLC of flavonol glycosides (quercetin, kaempferol, isorhamnetin as standards) on prewashed and preimpregnated silica gel with chloroform - acetone - formic acid - acetic acid 50:11:6:6. Plates were developed in solvent equilibrated, vapor saturated twin-trough chambers at 30°C. Densitometry in absorbance mode at 370 nm (for aglycones) and at 290 nm following a 1 s immersion in acetic anhydride and heating at different temperatures for varying lengths of time (for terpenelactones). Good relationship (95%) was determined between HPTLC and HPLC for determination of total flavonol glycosides. The HPTLC flavonol aglycone method also performed well in terms of accuracy and consecutive plate repeatability.

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

100 094 Anna GUMIENICZEK*, A. BERECKA, D. MATOSIUK, H. HOPKALA (*Department of Medicinal Chemistry, Medical University of Lublin, Jaczewskiego Str. 4, 20-090 Lublin, Poland; anna.gumieniczek@am.lublin.pl): Standardized reversed-phase thin-layer chromatographic study of the lipophilicity of five anti-diabetic thiazolidinediones. J. Planar Chromatogr. 20, 261-265 (2007). TLC of 5 anti-diabetic thiazolidinediones on RP-18 with binary mobile phases containing water and the organic modifier acetone, 1,4-dioxane, or methanol. Linear relationships were obtained between the Rm values of the compounds and the concentration of organic modifier in the mobile phase. TLC of ciglitazone, pioglitazone hydrochloride, darglitazone, englitazone, and rosiglitazone maleate against nine compounds of known lipophilicity (e. g. izatine, 2,6-dichloroacetanilide, 2,4-dichloroacetanilide, 4-nitrophenol etc.). Plates were developed in horizontal chambers. Visualization under UV light at 254 nm.

qualitative identification 32a

100 094 P.D. HAMRAPURKAR, S. PAWAR, V. JADHAV* (*Prin. K. M. Kundanani College of Pharmacy, Mumbai, Maharashtra, India): Quantitative determination of phyllanthin in Phyllanthus amarus using HPTLC. 59th Indian Pharmaceutical congress F-210, 440, (2007). HPTLC of phyllanthin from an extract of Phyllanthus amarus on silica gel with n-hexane - toluene - ethyl acetate 2:2:1. Quantification by densitometry at 206 nm. The hRf value was 0.27. Linearity was in the range of 200 - 1200 ng/zone. The method was applied for the determination of phyllanthin conent in Phyllanthus amarus. Extraction by supercritical fluid extraction gave higher yields than conventional extraction methods.

pharmaceutical research, herbal, HPTLC, densitometry 32e

100 095 P.D. HAMRAPURKAR*, P. KARISHMA (*Prin. K. M. Kundnani College of Pharmacy, Plot No. 23, Jote Joy Building, Rambhau Salgaonkar Road, Cuffe Parade, Colaba, Mumbai 400 005, India; kmkcp@vsnl.com): HPTLC determination of stigmasterol and tocopherol acetate in Leptadenia reticulata and in its formulation. J. Planar Chromatogr. 20, 183-187 (2007). HPTLC of stigmasterol on silica gel prewashed with methanol in a saturated twin-trough chamber with chloroform - ethanol - formic acid 98:2:1. Detection by dipping in a 5 % methanolic sulfuric acid solution and heating at 105 °C for 5 min. Quantitation by scanning at 580 nm. Also TLC of dl-apha-tocopherol acetate on silica gel with cyclohexane - diethylether 9:1. Scanning at 200 nm.

traditional medicine, herbal, quantitative analysis 32e

100 096 Erzsébet HÁZNAGY-RADNAI*, S. CZIGLE, I. MÁTHÉ (*Institute of Pharmacognosy, University of Szeged, Eötvös 6, H-6720 Szeged, Hungary; haznagy.radnai@pharm.u-szeged.hu): TLC and GC analysis of the essential oils of Stachys species. J. Planar Chromatogr. 20, 189-196 (2007). TLC of sabinene, limonene, linalool, and beta-caryophyllene on silica gel with benzene - ethyl acetate 9:1. Detection by spraying with a solution of vanillin in concentrated sulfuric acid, followed by heating at 105 °C for 2 min.

traditional medicine,herbal, qualitative identification 32e

100 097 T.W. INGLOT*, K. DABROWSKA, G. MISZTAL (*Department of Medicinal Chemistry, Skubiszewski Medical University, Jaczewskiego 4, 20-090 Lublin, Poland; chemia.lekow@am.lublin.pl): The normal-phase retention behavior of some angiotensin-II receptor antagonists. J. Planar Chromatogr. 20, 293-301 (2007). TLC of candesartan, eprosartan, losartan, telmisartan, and valsartan on silica gel, aluminum oxide, amino-, cyano-, and diol-phase in a horizontal chamber in sandwich technique. Diol-phases were developed with hexane - isopropanol - formic acid 40:60:1, cyano-phases with hexane - dioxane - formic acid 30:70:1. Detection and quantification at 254 nm.

pharmaceutical research, qualitative identification, densitometry, quantitative analysis 32a

100 098 A. JACOB*, S. SABOO, S. PRABHU, S.G. VASANTHARAJU, C. DINESH KUMAR, S. SHAHNAWAZ, G. GAUTHAM SHENOY (*Manipal College of Pharmaceutical Sciences, Karnataka, India): Stability indicating HPTLC method for the determination of duloxetine hydrochloride in bulk & pharmaceutical dosage form. 59th Indian Pharmaceutical Congress F-57, 403, (2007). HPTLC of duloxetine hydrochloride on silica gel with chloroform - methanol 4:1. Densitometric evaluation at 217nm. The hRf value of duloxetine was 45. The limit of detection and quantification was 120 and 240 ng/zone, respectively. Degradation products (acid, alkali, oxidative and thermal) were well separated from the main component. The proposed HPTLC method was routinely applied for identification and quantification of the drug in the formulation.

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

100 099 A.N. JADHAV, C.S. RUMALLA, B. AVULA, I.A. KHAN* (*National Center for Natural Products Research, University of Mississippi, University, MS 38677, USA): HPTLC method for determination of 20-hydroxyecdysone in Sida rhombifolia L. and dietary supplements. Chromatographia 66 (9-10), 797-800 (2007). HPTLC of 20-hydroxyecdysone from Sida rhombifolia L. on silica gel plates with chloroform - methanol 4:1. Quantification by densitometry at 250 nm in absorbance mode. Linearity was between 200 and 1000 ng/zone. This method was successfully applied for quantitative evaluation of dietary supplements. In addition, for six different Sida species unique fingerprints were obtained on the HPTLC plate.

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

100 100 R.B. KAKDE*, V.H. KOTAK, N.N. BAWANE (Dept. of Pharmaceutical Sciences, R.T.M. Nagar, University Campus, Nagpur, Maharashtra, India): Simultaneous estimation of amlodipine besylate and bisoprolol fumarate in pharmaceutical preparation by HPTLC. 59th Indian Pharmaceutical congress F-54, 402, (2007). HPTLC of amlodipine besylate and bisoprolol on silica gel with methanol - ethyl acatete - ammonia 1:12:1. Densitometric evaluation at 229 nm. The method was linear in the range of 500-1000 ng/zone. Recovery was 99.9 -101.5 %.

pharmaceutical research, quality control, HPTLC, densitometry 32a

100 101 A. KARTHIK, G.S. SUBRAMANIAN*, P. MUSMADE, A. RANJITHKUMAR, M. SURULIVELRAJAN, N. UDUPA (*Department of Pharmaceutical Quality Assurance, Manipal College of Pharmaceutical Sciences, Manipal, Karnataka-576104, India; ganrajesh@gmail.com): Stability-indicating HPTLC determination of rivastigmine in the bulk drug and in pharmaceutical dosage forms. J. Planar Chromatogr. 20, 457-461 (2007). HPTLC of rivastigmine ((-)-S-N-ethyl-3-[(1-dimethylamino)ethyl]-N-methylphenylcarbamate) and impurity on silica gel with chloroform - methanol 2:3 in a twin-trough chamber with chamber saturation for 30 min. Densitometric analysis was performed in absorbance mode at 210 nm. Limits of detection were 30 and 100 ng/zone, respectively.

quality control, HPTLC, densitometry, quantitative analysis 32a

100 102 A.D. KAURA, V. RAVICHANDRANA, P.K. JAINA, R.K. AGRAWAL* (*Pharmaceutical Chemistry Research Laboratory, Department of Pharmaceutical Sciences, Dr. Hari Singh Gour University, Sagar, MP 470003, India): High-performance thin layer chromatography method for estimation of conessine in herbal extract and pharmaceutical dosage formulations. J. Pharm. Biomed Anal. 46(2), 391-394 (2008). HPTLC of conessine silica gel aluminum plates with toluene - ethyl acetate - diethyl amine 13:5:2 in a twin trough chamber saturated with mobile phase at 25 °C. Detection by spraying with modified Dragendorff‘s reagent. Quantification by densitometry in absorbance mode at 520 nm. Linearity was between 1 and 10 µg/zone (r2 = 0.9998).

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

100 103 L. KOMSTA*, R. SKIBINSKI, A. IWANCZYK, G. MISZTAL (*Department of Medicinal Chemistry, Skubiszewski Medical University, Jaczewskiego 4, 20-090 Lublin, Poland; lukasz.komsta@am.lublin.pl): Retention data for some statin-type antihyperlipidemic drugs in normal-phase TLC. J. Planar Chromatogr. 20, 107-115(2007). TLC of atorvastatin, cerivastatin, fluvastatin, lovastatin, and simvastatin on silica gel, diol and cyano layers in horizontal chambers with binary mobile phases contaning hexane and a polar modifier in different proportions. Optimum normal-phase system was diol phase with hexane - tetrahydrofuran 3:2. Visualization under UV light at 254 nm. Quantitation by densitometry.

pharmaceutical research, qualitative identification, densitometry, quantitative analysis 32a

100 104 L. KOMSTA*, R. SKIBINSKI, A. IWANCZYK, H. HOPKALA (*Department of Medicinal Chemistry, Skubiszewski Medical University, Jaczewskiego 4, 20-090 Lublin, Poland; lukasz.komsta@am.lublin.pl): Separation of statin-type antihyperlipidemic drugs by reversed-phase TLC. J. Planar Chromatogr. 20, 235-237 (2007). TLC of atorvastatin, cerivastatin, fluvastatin, lovastatin, and simvastatin on RP-18 in horizontal chamber with sandwich configuration with methanol - buffer eluents of different composition. The best selectivity - separation of all the compounds - was achieved with methanol - phosphate buffer pH 7.60 4:1. Detection and densitometry at 254 nm.

quality control, quantitative analysis, densitometry 32a

100 105 A. KUCINSKAITÉ, L. POBLOCKA-OLECH, Miroslawa KRAUZE-BARANOWSKA*, V. BRIEDIS, A. SAVICKAS, M. SZNITOWSKA (*Department of Pharmacognosy, Medical University of Gdansk, Hallera 107, 80-416 Gdansk, Poland; krauze@amg.gda.pl): Use of SPE-TLC for quality control of Rhodiola rosea extracts. J. Planar Chromatogr. 20, 121-125 (2007). TLC of salidroside, rosavin, rosarin, and rosin on silica gel with ethyl acetate - methanol - water 77:13:10. UV detection was performed at 215 nm for salidroside and at 245 nm for rosavin. For visualization the plates were also sprayed with vanillin - phosphoric acid reagent.

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

100 106 R.S. KUMAR, S. DEBNATH*, G.N.K. GANESH, S. GUPTA, M.K. SAMANTA (*J.S.S. College of Pharmacy, Ooty, Tamil nadu, India): Qualitative and quantitative analysis of artemisinin, quercitin and rutin from Artemisia dracunculus by HPTLC technique. 59th Indian Pharmaceutical congress C- 260, 287, (2007). HPTLC of artemisinin, quercitin and rutin from Artemisia dracunculus (Asteraceae) on silica gel with ethyl acetate - dichloromethane - formic acid - glacial acetic acid - water 100:25:10:10:1 for artemisinin, and n-hexane - acetone - ethyl acetate 16:1:1 for quercitin and rutin. Evaluation under UV 254 nm. hRf values were 15, 26 and 91 for quercitin, artemisinin, and rutin, respectively. The alcoholic extract (dried) of the plant was found to contain 0.84 % artemisinin, 0.14 % quercitin, and 0.019 % rutin.

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

100 107 S. KUMAR*, K. KARTHIKEYAN, S. PATHAK, P.V. RAJ, N. UDUPA (*Manipal College of Pharmaceutical Sciences, Karnataka, India): A new HPTLC determination of rosiglitazone in bulk drug and pharmaceutical dosage forms. 59th Indian Pharmaceutical congress F-152, 426, (2007). HPTLC of rosiglitazone on silica gel aluminum layer with chloroform - ethyl acetate - ammonia 70:30:1. The hRf value of rosiglitazone was 40. Densitometric analysis in absorbance mode at 230 nm. Linearity was between 100 and 1000 ng/zone. Recovery (by standard addition method) was 99.6 %. Limit of detection and quantification was 10 ng and 50 ng/zone, respectively.

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

100 108 J.K. LALLA*, P.D. HAMRAPURKAR, A. SINGH (*Prin. K. M. Kundnani College of Pharmacy,

Plot No. 23, Jote Joy Building, Rambhau Salgaonkar Marg, Cuffe Parade, Colaba, Mumbai 400 005, India; jklalla@vsnl.net; jklalla@mtnl.net.in) : Quantitative HPTLC analysis of the eugenol content of leaf powder and a capsule formulation of Ocimum sanctum. J. Planar Chromatogr. 20, 135-138 (2007). HPTLC of eugenol (4-allyl-2-methoxyphenol) on silica gel prewashed with methanol in a twin-trough chamber saturated for 20 min with toluene - ethyl acetate - formic acid 93:7:1.4). Quantitation by scanning at 289 nm.

herbal, traditional medicine, HPTLC, quantitative analysis 32e

100 109 J.K. LALLA*, P.D. HAMRAPURKAR, S.J. SACKET (*Prin. K. M. Kundnani College of Pharmacy, Plot No. 23, Jote Joy Building, Rambhau Salgaonkar Road, Cuffe Parade, Colaba, Mumbai 400 005, India; jklalla@mtnl.net, jklalla@vsnl.net): Estimation of guggulsterone E and Z in solid dosage forms containing Commiphora mukul, Hook. J. Planar Chromatogr. 20, 197-202 (2007). HPTLC of guggulsterone E and Z on silica gel prewashed with methanol with petroleum ether - ethyl acetate - formic acid 30:10:1 in a twin-trough chamber saturated for 20 min. Detection and densitometric scanning at 254 nm.

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

100 110 G. MAHESHWARI, G.S. SUBRAMANIAN*, A. KARTHIK, A. RANJITHKUMAR, P. MUSMADE, K. GINJUPALLI, N. UDUPA (*Manipal College of Pharmaceutical Sciences, Manipal, Karnataka-576104, India; ganrajesh@gmail.com): High-performance thin-layer chromatographic determination of etoricoxib in the bulk drug and in pharmaceutical dosage form. J. Planar Chromatogr. 20, 335-339 (2007). TLC of etoricoxib (rofecoxib as internal standard) on silica gel in a filter-paper-lined twin-trough chamber previously saturated with mobile phase vapor for 30 min with toluene - 1,4-dioxane - methanol 17:2:1. Densitometric analysis of etoricoxib was performed in absorbance mode at 235 nm. The limits of detection and quantitation were 30 and 100 ng/zone, respectively.

quality control, densitometry, quantitative analysis 32a

100 111 S.K. MANDAL*, D. CHAKRABARTI, S. GHOSH, S.DEB (*Research & Development, East India Pharmaceutical Works Ltd., Kolkata, W.B.): A high performance thin layer chromatography (HPTLC) method for the estimation of gallic acid from Emblica officinalis (AMLAKI). 59th Indian Pharmaceutical Congress C-220, 277, (2007). HPTLC of gallic acid in fruits of Emblica officinalis, on silica gel with touene - ethyl acetate - acetic acid - formic acid 4:9:4:3. Densitometry at 277 nm for quantitative evaluation. The method was linear in the range of 10 and 1000 ng/mL, with an average recovery of 101.4 %. The method was found suitable for routine quality check-up of Emblica officinalis.

traditional medicine, quality control, herbal, HPTLC, densitometry, review, quantitative analysis qualitative identification 32c

100 112 A. MASLANKA, J. KRZEK* (*Collegium Medicum, Jagiellonian University, Department of Inorganic and Analytical Chemistry, 9 Medyczna Street, 30-688 Cracow, Poland; jankrzek@cm-uj.krakow.pl): Use of TLC with densitometric detection for determination of impurities in chlorpromazine hydrochloride, trifluoperazine dihydrochloride, promazine hydrochloride, and doxepin hydrochloride. J. Planar Chromatogr. 20, 463-475 (2007). TLC of chlorpromazine hydrochloride, trifluoperazine dihydrochloride, promazine hydrochloride, and doxepin hydrochloride on silica gel with 1-butanol - aqueous ammonia solution 5:1 or cyclohexane - acetone - diethylamine 8:1:1 after saturation with mobile phase vapor. Detection by inspection under UV light at 254 nm. Scanning densitometry was performed at 254 nm and at the wavelengths of maximum absorbance of the substances.

quality control, densitometry, quantitative analysis 32a

100 113 S. MENNICKENT*, A. SORBAZO, M. VEGA, C. GODOY, M. DIEGO (*Department of Phar-

macy, Faculty of Pharmacy, University of Concepcion, Concepcion, Chile, smennick@udec.cl): Quantitative determination of clozapine in serum by instrumental planar chromatography. J. Sep. Sci. 30, 2167-2172 (2007). HPTLC of clozapine in human serum on silica gel with chloroform - methanol 9:1. Quantitative determination by absorbance measurement at 290 nm. Linearity was between 10 and 100 ng/zone. The intra-assay variation was between 2.10 and 3.33 % (n=5) and inter-assay variation was between 2.67 and 4.44 % (n=9). The limits of detection and quantification were 0.03 and 0.05 ng/µL, respectively. Recovery was between 97.0 and 99.0 %, and selectivity regarding matrix was given.

pharmaceutical research, clinical routine analysis, HPTLC, quantitative analysis, densitometry
32c

100 114 S. MENNICKENT*, M. NAIL, M. VEGA, M. DIEGO (*Department of Pharmacy, Faculty of Pharmacy, University of Concepcion, Concepcion, Chile, smennick@udec.cl): Quantitative determination of L-DOPA in tablets by high performance thin layer chromatography. J. Sep. Sci. 30, 1893-1898 (2007). HPTLC of L-DOPA in tablets on silica gel with acetone - chloroform - n-butanol - acetic acid glacial - water 12:8:8:8:7. Quantitative determination by absorbance measurement at 497 nm. The hRf value of L-DOPA was 37 and selectivity regarding matrix was given. Linearity was between 100 and 500 ng/µL. The intra-assay variation was between 0.26 and 0.65 % and inter-assay variation was between 0.52 and 2.04 %. The limits of detection and quantification were 1 and 3 ng/µL, respectively. No significant difference was found between this method and the official HPLC method.

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

100 115 D.V. MHASKE, S.R. DHANESHWAR*, S.S. KADAM (*DEpt. of Quality Assurance Tech. and Pharm. Chem., Pune, India) : Stability indicating HPTLC method for determination of irbesartan in pharmaceutical dosage form. Indian J. Pharm. Educ. Res. 41(3), 261 (2007). HPTLC of irbesartan on silica gel with toluene - ethyl acetate - acetic acid 70:30:2. Densitometric aevaluation at 305 nm. Linearity was between 500-6000 ng/zone. Limit of detection and quantification was 100 and 400 ng/zone, respectively. Recovery was more than 100 %. The degradation products as result of acid and alkali hydrolysis, oxidation, dry and wet heat treatment and photodegradation were well separated from tirbesartan.

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

100 116 D.V. MHASKE*, S.R. DHANESHWAR (*Department of Quality Assurance Techniques and Pharm. Chem., Bharati Vidyapeeth University, Centre for Advanced Pharmaceutical Research, Erandwane, Pune, 411038, Maharashtra, India): Stability Indicating HPTLC and LC Determination of Dasatinib in Pharmaceutical Dosage Form. Chromatographia 66 (1-2), 95-102 (2007). HPTLC of dasatinib in the presence of its degradation products, on silica gel sheets with toluene - chloroform 7:3. Quantification by densitometry at 280 nm. The hRf value of dasatinib was 23 and selectivity regarding matrix was given. Validation of the method as per the ICH guidelines. Dasatinib was subjected to acid-alkali hydrolysis, oxidation, dry heat, wet heat and photodegradation. The drug was susceptible to acid-alkali hydrolysis and oxidation. The drug was found to be stable in neutral, wet heat, dry heat and photo-degradation conditions.

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

100 117 R. MYTHREYI*, A.C. KUMAR, C. SUDHA, P. THOMAS, V. MADHAVAN (*M.S. Ramaiah College of Pharmacy, Bangalore, Karnataka, India): HPTLC Fingerprinting of Z-guggulsterone in some marketed ayurvedic formulations. 59th Indian Pharmaceutical Congress C-43, 234, (2007). HPTLC of Z-guggulsterone (a steroidal ketone present in oily resin of Commiphora mukul) and five marketed ayurvedic tablet formulations containing guggul, on silica gel with toluene - propane-1-ol - glacial acetic acid 8:1:1. Densitometry at 254 nm. The hRf of E-guggulsterone

was 55. Several marketed formulations were evaluated for Z-guggulsterone, the UV spectra of the standard and that of the formulations were comparable in respect of Z-guggulsterone. The method was found suitable for evaluation of Z-guggulsterone in presence of other phytochemicals present in formulations.

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

100 118 S. NAMUR*, L. CARINO, M. GONZALES-DE LA PARRA (*Fundación Liomont A. C. Mexico City, México, Privada Jesús del Monte 77, Cuajimalpa CP 05000, México): Development and validation of a high-performance thin-layer chromatographic method, with densitometry, for quantitative analysis of tizoxanide (a metabolite of nitazoxanide) in human plasma. J. Planar Chromatogr. 20, 331-334 (2007). HPTLC of tizoxanide (with nitazoxanide as internal standard) on silica gel prewashed with methanol in a twin-trough chamber with toluene - ethyl acetate - acetic acid 62:134:4. UV detection and quantitation at 313 nm for the internal standard and at 410 nm for tizoxanide.

quality control, HPTLC, densitometry, quantitative analysis 32a

100 119 M.G. PAI, R. GUDE*, S. BHENDE, D. VERLEKAR (*Goa College of Pharmacy, Panaji, Goa, India): A new validated HPTLC method for the quantitative estimation of atorvastatin calcium and amlodipine besylate in tablets. 59th Indian Pharmaceutical congress F-154, 426, 2007. HPTLC of atorvastatin calcium and amlodipine besylate on silica gel with ethyl acetate - 1,4-dioxane - methanol - ammonia 10:1:2:1. Quantitative evaluation by densitometry at 254 nm. The hRf value was 35 and 56 for atorvastatin and amlodipine, respectively. Linearity was between 200 and 1000 ng/zone for atorvastatin and 400 and 2000 ng/zone for amlodipine. Recovery was 99.8 - 100.9 % for both compounds. The method was found suitable for routine quality control of formulations containing both drugs in combined formulations.

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

100 120 M.G. PAL, V. KARPE*, R. GUDE (*Goa College of Pharmacy, Panaji, Goa, India): A new validated method for the quantitative estimation of famotidine and domperidone in tablets. 59th Indian Pharmaceutical congress F-173, 431, (2007). HPTLC of famotidine and domperidone on silica gel with ethyl acetate - methanol - toluene - ammonia 40:15:20:2. Densitometry at 285 nm. Linearity was between 100 and 600 ng/µL for both compounds. Limit of detection and quantification was 33 and 100 ng/zone, respectively. Recovery was 99.5 %.

pharmaceutical research, quality control, HPTLC, densitometry 32a

100 121 R. PANDEY, R. VERMA, M. GUPTA* (*Analytical Chemistry Division, Central Institute of Medicinal and Aromatic Plants, Lucknow, India, guptammg@rediffmail.com): High-performance thin-layer chromatography method for quantitative determination of 4alpha-methyl-24beta-ethyl-5alpha-cholesta-14,25-dien-3beta-ol, 24beta-ethylcolesta-5,9(11),22E-trien-3beta-ol, and betulinic acid in Clerodendrum inerme. J. Sep. Sci. 30, 2086-2091 (2007). HPTLC of 4alpha-methyl-24beta-ethyl-5alpha-cholesta-14,25-dien-3beta-ol (1), 24beta-ethylcolesta-5,9,11,22E-trien-3beta-ol (2), and betulinic acid (3) in Clerodendrum inerme on silica gel with toluene - ethyl acetate 47:3. Quantitative determination by absorbance measurement at 620 nm. The hRf value was 48, 34 and 22 for compounds (1) , (2) and (3), respectively. Linearity was between 100 and 2500 ng/zone. The limits of detection and quantification were 5, 6, and 10 µg/mL and 14, 18, and 29 µg/mL, respectively, for (1), (2), and (3).

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

100 122 P.B. Panjabrao, N.G. Patil, O.N. Amrite, N.G. Pardesi, C.H. andGadgoli* (*Saraswathi Vidya Bhavan,s College of Pharmacy, Dombivli, India): Nyctanthes arbor-tristis as a substitute for saf-

fron colour. Indian Drugs 44(8), 640 (2007). TLC, HPTLC and UV spectrophotometric methods have been developed and evaluated to compare the colour properties of saffron and Nyctanthes arbor-tristis which shows an orange red colour. HPTLC and TLC of methanolic extracts of saffron and calyx of Nyctanthes arbor-tristis on silica gel with ethyl acetate - isopropanol - water 13:5:2. Both extracts showed a major zone with hRf 23 corresponding to crocin, the major colour constituent of saffron. The presence of crocin was confirmed by UV spectra. TLC, HPTLC and UV data confirm the coloring similarity of Nyctanthes arbor-tristis.

pharmaceutical research, traditional medicine, quality control, HPTLC, comparison of methods densitometry, quantitative analysis 32e

100 123 P.M. Patel, K.N. Patel, R.K. Goyel* (*Dept. of Pharmacognosy, L. M. College of Pharmacy, Ahemdabad, India): A HPTLC method for quantitative estimation of swetiamarin in marketed polyherbal antidiabetic formulations. Indian J. Pharm. Sci. 69(3), 446 (2007). HPTLC of swetimarin (a phytoconstituent of Enieostemma Littorale) in chloroform extracts of a polyherbal antidiabetic commercial formulation on silica gel with benzene - methanol 4:1. Detection by spraying with 10 % methanolic sulphuric acid and heating at 130° C for 2 min. Evaluation by densitometry at 536 nm. The linearity range was 50-1500 ng/zone. Recovery was more than 96 %.

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

100 125 A.V. PATEL*, J.V. PATEL, P.U. PATEL, C.N. PATEL (*Shree Sarvajanik College of Pharmacy, Mehsana, Gujarat, India): Simultaneous estimation of cilostazol and aspirin in synthetic mixture using HPTLC method. 59th Indian Pharmaceutical congress F-23, 395, (2007). HPTLC of aspirin and cilostazol in synthetic mixture on silica gel with methanol - ethyl acetate - toluene - ether 2:4:4:1. Linearity was between 75 and 600 ng/mL for aspirin and 100 and 800 ng/mL for cilostazol. Recovery was between 98 and 101 %.

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

100 126 Heena PATEL*, J.V. PATEL, P.U. PATEL, C.N. PATEL (*Shri. Sarvajanik College og Pharmacy, Mehsana, Gujarat, India): HPTLC method for the estimation of cilostazol in bulk and dosage forms. 59th Indian Pharmaceutical congress F-224, 443, (2007). HPTLC of cilostazol on silica gel with ethyl acetate - toluene - methanol - ether 4:4:2:1. The hRf value was 64. Limit of detection and quantification was 7 ng/spot and 20 ng/spot respectively.

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

100 127 P.M. PATEL*, N.M. PATEL, R.K. GOYAL (*Shri. B. M. Shah College of Pharma Education & Research, Modosa, Gujarat, India): Standardization of polyherbal formulations used in diabetes mellitus. 59th Indian Pharmaceutical Congress C-17, 228 (2007). HPTLC of curcumin, charantin, and swetiamarin in polyherbal formulations on silica gel with benzene - methanol 4:1 for charantin, chloroform - methanol - formic acid 74:4:1 for curcumin, and ethyl acetate - methanol - water 77:15:5 for swetiamarin. Densitometry at 536 nm for charantin, 425 nm for curcumin, and 238 nm for swetiamarin. hRf values were 33, 89, and 54 for charantin, curcumin, and swetiamarin respectively.

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

100 128 S.A. PATEL*, P.U. PATEL, M.M. PATEL, U.V. BANGORIYA, S.K. PATEL (*S. Patel College of Pharmaceutical Education and Research Ganpat University, Kherva, India): HPTLC method for linezolidin tablets. Indian J. Pharma. Sci. 69(4), 571 (2007). HPTLC of linezolid on silica gel with methanol - benzene 1:4. Densitometric evaluation at 258 nm. The hRf value was 45. Linearity was between 200 and 1400 ng/zone. Limit of detection and quantification was 17 and 51 ng/zone, respectively.

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

100 129 G. PATHAK*, M. CHINTAMANENI, V. ADDEPALLI (*School of Pharmacy & Technology Management, SVKM'S NMIMS University, Mumbai, India): Standardization of traditional ayurvedic Arjuna for formulations by using modern research tools. 59th Indian Pharmaceutical Congress C-66, 240, (2007). Evaluation of Arjunakhseerpak and Arjunaristha, two ayurvedic formulations used for cardiovascular system, which contain Terminalia arjuna as the main ingredient. Simultaneous HPTLC of arjungenin and arjunolic acid from Terminalia arjuna on silica gel with toluene - ethyl acetate - acetic acid 10:10:1. Detection by spraying with 10 % sulphuric acid followed by heating. Densitometric evaluation at 366 nm. The UV spectra of the sandards arjungenin and arjunolic acid was found to be similar to that of arjungenin and arjunolic acid in the formulation. The method was found suitable for standardization of arjuna formulations.
pharmaceutical research

traditional medicine, quality control, HPTLC, densitometry, quantitative analysis
postchromatographic derivatization 32c

100 130 L.J. PATIL*, B.N. SUHAGIA, P.B. SHAH (*Shri B. M. Shah College of Pharmacy, Modasa, India): RP-HPLC and HPTLC methods for the estimation of nebivolol hydrochloride in tablet dosage form. Indian J. Pharm. Sci. 69(4), 594 (2007). HPTLC of nebivolol HCl in tablet dosage form, on silica gel with ethyl acetate - toluene - methanol - ammonia 10:60:20:1. Quantitative evaluation by densitometry at 280 nm. The hRf value was 33. Linearity was in the range of 100 - 600 ng/mL. Limit of detection and quantification was 30 and 100 ng/zone, respectively. The method was compared with an RP-HPLC method in respect of different analytical parameters. The RP-HPLC and the HPTLC method were found comparable, but the HPTLC method was more sensitive.
pharmaceutical research

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

100 131 U.K. PATIL*, C.S. BHARGAV, S. JAIN, S.K. YADAV (*VNS Institute of Pharmacy Bhopal, Madhya Pradesh, India): Development and densitometric standarization of Convolvulus pluricaulis acontaining herbal medicinal products by quantification of marker compound. 59th Indian Pharmaceutical congress C-327, 303, (2007). HPTLC of 3-β-23-24 -trihydroxy-olean-12-en-28-oic acid in Convolvulus pluricaulus on silica gel with n-hexane - ethyl acetate 7:4. Quantitative determination by densitometry at 226. The limit of detection and quantification was14.2 and 50 ng/zone, respectively. Recovery was 94.5 % - 97.2 %. The method was applied for analysis of herbal syrup and tablets.

herbal, HPTLC, densitometry, quantitative analysis 32e

100 132 S.P. PATTANAYAK, P. SUNITA, A.K. PATTANAYAK*, M. MAZUMDAR, N.K. DHAL (*Birla Institute of Technology, Mesra, Ranchi, Jharkhand, India): Determination of MIC value by gradient plate technique and in antifungal activity by TLC method of isolated compound gedunin, from the plant Azadirecta indica. 59th Indian Pharmaceutical congress C-344, 308, (2007). TLC of different fractions of fruits of Azadirecta indica on silica gel with ethyl acetate - n-hexane 1:1. The MIC value was determined in comparison with gedunin isolated from the plant. The fungi were sub cultured and incubated for seven days, then sprayed on the developed plate, followed by incubation at 37°C . Zones of inhibition were measured. Antifungal activity was observed with Aspergillus niger, Fusarium specis, and Penicillium chrysogenum.

herbal, HPTLC, quantitative analysis 32e

100 133 Nada PERISIC-JANJIC*, G. VASTAG, J. TOMIC, S. PETROVIC (*Department of Chemistry, Faculty of Sciences, Trg D. Obradovica 3, 21000 Novi Sad, Serbia; nadap@uns.ns.ac.yu): Effect of the physicochemical properties of N,N-disubstituted-2-phenylacetamide derivatives on their retention behavior in RP-TLC. J. Planar Chromatogr. 20, 353-359 (2007). TLC of 8 N,N-disubs-

tituted-2-phenylacetamides on RP-18 with acetone, acetonitrile, tetrahydrofuran, dioxane, methanol, ethanol, 1-propanol, and 2-propanol as organic modifiers of aqueous mobile phases. The retention observed with different mobile-phase modifiers was correlated, and good correlation was obtained between lipophilicity measured chromatographically and biological activity predictors. Detection under UV light at 254 nm.

qualitative identification 32a

100 134 Caroline PETITTI (Bayer Sante Familiale, 33 rue de l'industrie, 74240 Gaillard, france; caroline.petitti@bayerhealthcare.com): Determination of aminopropanol in dermatological products. CBS 98, 2-4 (2007). HPTLC of amino-3-propan-1-ol, a degradation product of dexpanthenol, on silica gel with ethanol - water - acetic acid 16:3:1. To the mobile phase the derivatization reagent was added, i.e. 0.5 g ninhydrin were dissolved in 100 mL solvent mixture. Development in the horizontal developing chamber from both plate sides over 40 mm. Detection by heating at 105 °C for 5 min. Quantitative determination by absorbance measurement at 486 nm. The hRf value of amino-propanol was 50. The mean repeatability was 4.9 % at 5 different concentration levels. The relative standard deviation of the intermediate precision (n=9) was 5.7 %. The limit of detection and quantification was 4.5 µg/mL and 15 µg/mL, respectively (related to the application volume of 2 µL). Recovery (n=15) was 102 %.

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

100 135 O. POZHARITSKAYA, S. IVANOVA, A. SHIKOV*, V. MAKAROV, B. GALAMBOSI (*Interregional Center „Adaptogen", St. Petersburg, Russia; alexs79@mail.ru): Separation and evaluation of free-radical scavenging activity of phenol components of green, brown, and black leaves of Bergenia crassifolia by using HPTLC-DPPH method. J. Sep. Sci. 30, 2447-2451 (2007). HPTLC of free gallic (1) and ellagic (2) acids, arbutin (3), hydroquinone (4), and bergenin in the green, brown and black leaves of Bergenia crassifolia on silica gel with toluene - ethyl acetate - formic acid - methanol 15:15:4:1. Quantitative determination by absorbance measurement at 280 nm. Antiradical activity of each component was determined by postchromatographic 1,1-diphenyl-2-picrylhydrazyl radical (DPPH) derivatization and densitometric scanning at 517 nm as negative peak. Linearity was between 40 and 140 ng/zone, 240 and 720 ng/zone, 150 and 980 ng/zone, and 90 and 230 ng/zone for (1), (2), (3), and (4), respectively. The limit of detection was 30, 200, 90, and 40 ng/zone for (1), (2), (3), and (4), respectively. All compounds of the extract excluding bergenin were capable of scavenging DPPH radicals.

herbal, HPTLC, quantitative analysis, densitometry 32e

100 136 C. PRABHU, G.S. SUBRAMANIAN*, A. KARTHIK, S. KINI, M. S. RAJAN, N. UDUPA (*Department of Pharmaceutical Quality Assurance, Manipal College of Pharmaceutical Sciences, Manipal, Karnataka-576104, India; ganrajesh@gmail.com): Determination of telmisartan by HPTLC - a stability indicating assay. J. Planar Chromatogr. 20, 477-481 (2007). HPTLC of telmisartan (4'-{[4-methyl-6-(1-methylbenzimidazol-2-yl)-2-propylbenzimidazol-1-yl]methyl}biphenyl-2-carboxylic acid) on silica gel with chloroform - methanol 43:7 in a twin-trough chamber with chamber saturation for 25 min. Densitometric scanning in absorbance mode at 297 nm.

quality control, HPTLC, densitometry, quantitative analysis 32a

100 137 V. PURACHIMANI, S. JHA* (*Dept. of Pharma Sci., Birla Institute of Technology, Mesra, Ranchi, India): HPTLC standardization of Tinospora cordifolia using tinosporaside. Indian J. Pharm. Sci. 69 (4), 578 (2007). TLC of tinosporaside in defatted methanolic extracts of Tinospora Cordifolia stem bark on silica gel with toluene - acetone -water 5:15:1. Quantitative evaluation by densitometry at 220 nm. The method was found to be linear in the range of 0.5-8 mg. Recovery was 99.3%. The stem bark of the plant contained 0.4 % of tinosporaside.

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

100 138 Alina PYKA*, D. GURAK (*Department of Analytical Chemistry, Faculty of Pharmacy, Medical University of Silesia, Jagiellonska 4, PL-41-200 Sosnowiec, Poland; apyka@slam.katowice.pl): Use of RP-TLC and theoretical computational methods to compare the lipophilicity of phenolic drugs. J. Planar Chromatogr. 20, 373-380(2007). Investigation of the chromatographic behavior of the phenolic drugs niclosamide, hexachlorophene, ibuprofen, pentazocine, ethamivan, bithionol, salicylanilide, caffeic acid, p-coumaric acid, 4-aminosalicylic acid, ferrulic acid, and methyldopa on RP-8 and RP-18 phase prewashed with methanol, with methanol - water mixtures in different volume proportions, with chamber saturation for 20 min. The results calculated using seven different software products indicate, that chromatographic lipophilicity can be used as a measure of the lipophilicity of the compounds investigated. Spectra were acquired with the densitometer in absorbance mode. Densitometric scanning was performed at the respective absorption maxima.

pharmaceutical research, qualitative identification, densitometry 32a

100 139 A.P. RAINA*, A. KUMAR, S.K. PAREEK (*Germplasm Evaluation Division, National Bureau of Plant Genetic Resources, New Delhi, India): HPTLC analysis of hepatoprotective diterpenoid andrographolide from Andrographis paniculata nees (Kalmegh). Indian J. Pharm. Sci. 69(3), 473 (2007). Dried leaves were Soxhlet extracted with methanol and concentrated. HPTLC of andrographolide in Andrographis paniculata on silica gel with chloroform - methanol 7:1 with chamber saturation for 15 min. Densitometric evaluation at 232 nm. The hRf value of andrographolide was 35, of neoandrographolide 15 and of andrographoside 3. The linearity range was 200–1000 ng/zone. Average andrographolide contents were 1.56 % in dried leaves sample.

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

100 140 V.B.A. RAJ*, S.P. DHANABAL, M.J. NANJAN, B. SURESH (*J.S.S. College of Pharmacy, Ooty, Tamil Nadu, India): Estimation of Phytoflavonid Auercetin from Methanolic extract of Tylophora indica by HPTLC techinque. 59th Indian Pharmaceutical Congress C-176, 267, (2007). HPTLC of tylophorine, kaempferol, alpha-amyrin, and quercetin in an alcoholic extract from Tylophora indica on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:26. Densitometry at 366 nm. The method was found suitable for the estimation of quercetin in plant drugs and their formulations.

traditional medicine, quality control, HPTLC, densitometry, quantitative analysis 32c

100 141 R.M. RAJURKAR*, B. DURAISWAMY, S.L. DEORA, S.S. KHADABADI (*Govt. College of Pharmacy, Amravati, Maharashtra, India): Quantitative estimation of asarone content in polyherbal formulation by HPTLC method. 59th Indian Pharmaceutical congress C-275, 291, (2007). HPTLC of asarone in a polyherbal formulation consisting of Acorus Calamus on silica gel with toluene - chloroform - ethyl acetate 18:1:1. Densitometry at 254 nm. The amount of asarone present in extract of sluizaro, a syrup formulation, was determined. The method was found to be suitable for routine quality control.

herbal, HPTLC, quantitative analysis, densitometry 32c

100 142 R. RAMÍREZ-DURÓN, L. CENICEROS-ALMAGUER, R. SALAZAR-ARANDA, M. DE LA LUZ SALAZAR-CAVAZOS, Noemi WAKSMAN DE TORRES* (*Universidad Autónoma de Nuevo León, Departamento de Química Analítica, Facultad de Medicina, PO Box 2316, Sucursal Tecnológico, 64841, Monterrey Nuevo León, Mexico; nwaksman@fm.uanl.mx): Evaluation of Thin-Layer Chromatography methods for quality control of commercial products containing Aesculus hippocastanum, Turnera diffusa, Matricaria recutita, Passiflora incarnata, and Tilia occidentalis. J. Assoc. Off. Anal. Chem, 90, 920-924 (2007). TLC of commercial products containing Aesculus hippocastanum, Turnera diffusa, Matricaria recutita, Passiflora incarnata, and Tilia occidentalis against standardized extracts on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:27 with chamber saturation. Detection by spraying with 1 % methanolic diphenylborinic acid 2-amino ethyl ester, followed by 5 % ethanolic polyethylenglycol 400 (PEG) and visualization under UV light at 365 nm or with anisaldehyde - sulfuric acid reagent followed by

heating at 100 - 110°C and visualization under visible and UV light at 365 nm. The standards contained aescin, apigenin-7-glucoside, other glycoside flavonoids, flavonoid aglycones, quercetin, myricetin, kaempferol, and rutin.

quality control, herbal, qualitative identification 32e

100 143 S. RASTOGI*, M.M. PANDEY, A.K.S. RAWAT (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Lucknow, 226001, India): Determination of Heraclenin and Heraclenol in Heracleum candicans D.C. by TLC. Chromatographia 66 (7-8), 631-634 (2007). TLC of heraclenin and heraclenol in the roots of Heracleum candicans D.C. on silica gel with toluene - ethyl acetate 7:3. Quantitative determination by densitometry at 366 nmn. Linearity was between 4 - 10 µg/zone for heraclenin and 1 - 5 µg/zone for heraclenol. pharmaceutical research

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

100 144 A.K. RAUT*, J.L. TEJWANI, D.B. MESHRAM. S.B. BAGADE, M.R. TEJNE (*Dept. of Pharmaceutical Sciences, R.T.M. Nagar, University Campus, Nagpur, Maharashtra, India): Simultaneous estimation of nebivolol and hydrochlorothiazide in combined dose tablet by HPTLC. 59th Indian Pharmaceutical congress F-37, 399, (2007). HPTLC of nebivolol and hydrochlorthiazide on silica gel with methanol - ethyl acetate - toluene - glacial acetic acid 15:25:4:1. Densitometry at 285 nm. The method was linear in the range of 150 - 350 ng/zone for nebivolol and 370 - 870 ng/zone for hydrochlorothiazide. Recovery was 99.5 %.

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

100 145 T.S. REDDY, P.S. DEVI* (*Analytical Chemistry Division, Indian Institute of Chemical Technology, Tarnaka, Hyderabad-500007, India; sitadevi@iictnet.org): Validation of a High-Performance Thin-Layer Chromatographic method, with densitometric detection, for quantitative analysis of nebivolol hydrochloride in tablet formulations. J. Planar Chromatogr. 20, 149-152 (2007). HPTLC of nebivolol hydrochloride on silica gel prewashed with methanol with toluene - ethyl acetate - methanol - formic acid 8:6:4:1 in a saturated twin-trough chamber. Densitometric quantification at 285 and 298 nm.

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

100 146 M. REDDY*, G. MUBEEN, Sanjay PAI, P.N. GORLE, L. GANESH, B.K. GOPAL (*R.R. College of Pharmacy, Chikkababavara, Bangalore, Karnataka, India): Development & Validation of HPTLC method for Estimation of Metformin HCl. 59th Indian Pharmaceutical Congress F-55, 402, (2007). HPTLC of metformin hydrochloride on silica gel with methanol - chloroform - ammonium acetate 6:3:1. Densitometry at 236 nm. The method has a range from 100 - 300 ng/zone. The limit of detection and quantification was 25 ng and 100 ng/zone, respectively. The method was applied for analysis of metformin hydrochloride in multi-component formulations containing glipizide, gliclazide and glibenclamide as well as in serum.

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

100 147 E. REICH*, Anne SCHIBLI, Valeria WIDMER, Ruth JORNS, Evelyn WOLFRAM, Alison DEBATT (*CAMAG Laboratory, Sonnenmattstr. 11, 4132 Muttenz, Switzerland; eike.reich@camag.com): HPTLC methods for the identification of green tea and green tea extract. CBS 97, 12-15 (2006). HPTLC of flavonoids from green tea (Camellia sinensis) on silica gel in a saturated 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 (10g 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 into a solution of Fast Blue Salt B. Evaluation under white light. For investigation of the alkaloid profile ethyl acetate - methanol - water 20:2.7:2 and evaluation 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 in ninhydrin reagent, followed by heating at 110 °C for 3 min. Evaluation under white light.

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

100 148 Annalisa ROMANI*, P. PINELLI, C. GALARDI, G. SANI, A. CIMATO, D. HEIMLER (*Dipartimento di Scienze Farmaceutiche, Degli Studi Di Firenze, Florence, Italy, annalisa.romani@unifi.it) : Polyphenols in greenhouse and open-air-grown lettuce. Food Chem. 79, 337-342 (2002). HPTLC of polyphenol compounds (caffeic acid derivatives, quercetin and kaempferol glycosides) in the leaves of Lactuca sativa on RP-18 with water - methanol - acetic acid 25:25:3. Detection by dipping into a solution of 1 % ethanolamine diphenylborate in methanol for 24 h. Quantitative determination by absorbance measurement at 365 and 440 nm. The total flavonoid amount is expressed as isoquercitrin using a three point regression curve in the range of 1 and 5000 ng/zone.

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

100 149 K.K. ROUT, O.P. ROUT, S.K. MISHRA* (*Pharmacognosy and Phytochemistry Division, UDPS. Utkal University, Vani Vihar, Bhubaneswar-751004, Orissa, India; skmishraudps@gmail.com): Estimation of piperine in commercial Ayurvedic formulations. J. Planar Chromatogr. 20, 447-450 (2007). HPTLC of piperine on silica gel, prewashed with methanol, with hexane - acetone 13:7 in a twin-trough chamber with chamber saturation for 5 min. Quantification by densitometry at 340 nm in absorbance mode.

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

100 150 M.L. RUSU, C. MARUTOIU*, I. SANDU, D. TITA, I. GOGOASA, C.-H. BARBU, A. POPESCU (*Lucian Blaga University, Faculty of Agricultural Sciences, Food Industry and Environmental Protection, 7-9 Ion Ratiu Street, 550012 Sibiu, Romania; cmarutoiu@email.ro): HPTLC and GC-MS for separation and identification of eugenol in plants. J. Planar Chromatogr. 20, 139-140 (2007). HPTLC of eugenol (4-allyl-2-methoxyphenol) on silica gel with heptane - ethyl acetate 3:2 in normal, unsaturated chambers. Visualization under UV light at 254 nm.

food analysis, herbal, HPTLC, qualitative identification 32e

100 151 T. S. REDDY, P. S. DEVI* (*Analytical Chemistry Division, Indian Institute of Chemical Technology, Tarnaka, Uppal Road, Hyderabad 500007, A. P., India; sitadevi@iictnet.org): Validation of a High-Performance Thin-Layer Chromatographic method with densitometric detection for quantitative analysis of two anticonvulsants in tablets. J. Planar Chromatogr. 20, 451-456 (2007). HPTLC of levetiracetam and oxcarbazepine on silica gel, prewashed with methanol, with toluene - acetone - methanol 3:1:1 in a twin-trough chamber saturated for 20 min at 25 °C. Densitometric evaluation in absorbance mode at 200 and 261 nm.

quality control, HPTLC, densitometry, quantitative analysis 32a

100 152 M.N. SARAF*, P.G. BIRAJDAR, P. LOYA, S.A. MUKHERJEE (*The Bombay College of Pharmacy, Kalina, Santacruz (E), Mumbai 400 098, India; saraf@bcp.edu.in): Rapid and sensitive HPTLC method for determination of epalrestat in human plasma. J. Planar Chromatogr. 20, 203-207 (2007).. HPTLC of epalrestat (5-[(1Z,2E)-2-methyl-3-phenylpropenylidene]-4-oxo-2-thioxo-3-thiazolidineacetic acid) with nitrofurantoin as internal standard on silica gel with ethyl acetate - toluene - acetic acid 30:20:1. Densitometric scanning in absorbance mode at 290 nm.

clinical chemistry research, HPTLC, densitometry, quantitative analysis 32a

100 153 S.H. SHAH*, C.R. SHAH, N.J. SHAH, N.M. PATEL, B.N. SUHAGIA (*Shri B.M. Shah College of Pharm. Edu. and Res. Gujrat, India): Validation and development of an HPTLC method for the simultaneous estimation of olmesartan medoxomil and hydrochlorothiazide in tablet dosage form. 59th Indian Pharmaceutical congress F-10, 392, (2007). HPTLC of olmesartan medoxomil and hydrochlorothiazide on silica gel with acetonitrile - chloroform - glacial acetic acid 14:4:1 with chamber saturation for 30 min. Densitometric evaluation at 254 nm. Linearity was between 480 and 900 ng/zone for olmesartan and 150 and 600 ng/zone for hydrochlorothiazide. Recovery was 99.7 - 100.4 % . The method can be used for routine quality control of formulations.

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

100 154 C.R. Shah*, D.R. Patel, S.Y. Gabhe, P.K. Tatke (*B. M. Shah College of Pharmaceutical Education and Res. Modasa, India): Isolation, identification and characterization of aloin in Kumariasava and Aloe vera by different analytical techniques. iNDIAN dRUGs 44(8), 632 (2007). TLC, HPTLC and NMR spectroscopic methods are reported for identification and characterization of aloin isolated from Kumariasava and Aloe vera. TLC and HPTLC of chloroform extracts on silica gel with chloroform - ethyl acetate 3:1. Detection under UV 366 nm. The hRf value of aloin was 84. pharmaceutical research

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

100 155 C.R. SHAH*, N.J. SHAH, B.N. SUHAGIA, N.M. PATEL (*Shri B. M. Shah College of Pharmaceutical Education and Research, Department of Pharmaceutical Chemistry, College Campus, Modasa-383315, Gujarat, India; crshah681@yahoo.com) : Simultaneous assay of olanzapine and fluoxetine in tablets by column High-Performance Liquid Chromatography and High-Performance Thin-Layer Chromatography. J. Assoc. Off. Anal. Chem, 90, 1573-1578 (2007). TLC of olanzapine [2-methyl-4-(4-methyl-1-piperazinyl)-10H-thieno[2,3-b] [1,5]benzodiazepine] and fluoxetine [(+/-)-N-methyl-3-phenyl-3-[(alpha,alpha,alpha-trifluoro-p-tolyl)oxy]propylamine] on silica gel using methanol - toluene 2:1 in a twin-trough chamber with chamber saturation. Quantitation by densitometry in absorption mode at 233 nm.

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

100 156 C.R. SHAH*, N.J. SHAH, D.R. PATEL, B.N. SUHAGIA, N.M. PATEL (*Shri B.M. Shah College of Pharm. Edu. and Res. Gujrat, India): Comparative study of RP-HPLC and HPTLC for the determination of atomoxetine hydrochloride in bulk powder and tablet dosage form. 59th Indian Pharmaceutical congress F-8, 397, (2007). HPTLC of atomoxetine on silica gel with acetonitrile - methanol - toluene 2:4:1. Quantification by densitometry at 215 nm. The method was linear in the range of 100 and 310 ng/mol. Mean recovery was 101.3 %.

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

100 157 V. SHARMA, A.P. GUPTA, Pamita BHANDARI, R.C. GUPTA, B. SINGH* (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology, Palampur, 176 061, H.P, India): A Validated and Densitometric HPTLC Method for the Quantification of Withaferin-A and Withanolide-A in Different Plant Parts of Two Morphotypes of Withania somnifera. Chromatographia 66 (9-10), 801-804 (2007). HPTLC of withaferin A and withanolide A in Withania somnifera methanolic extract from different plant parts (leaf, root, stem and fruit) and of two morphotypes, on silica gel with toluene - ethyl acetate - formic acid 5:5:1. Quantification by densitometry in absorption mode at 530 nm. Linearity was between 200 and 3200 ng for both withaferin A and withanolide A. The average recovery of withaferin A and withanolide A was 96.0 and 96.7 %. pharmaceutical research

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

100 158 U. SHARMA, N. SHARMA, A. GUPTA, V. KUMAR, A. SINHA* (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology, Palampur, India, aksinha08@rediffmail.com): RP-HPTLC densitometric determination and validation of vanillin and related phenolic compounds in accelerated solvent extract of Vanilla planifolia. J. Sep. Sci. 30, 3174-3180 (2007). HPTLC of vanillin and nine related phenolic compunds in Vanilla planifolia pods on RP-18 with methanol - water - isopropanol - acetic acid 30:65:2:3. Quantitative determination by absorbance measurement at 280 nm. The hRf value of vanillin was 41. Linearity was between 0.990 and 0.999 ng/zone. Repeatability was better than 3.5 %. The limits of detection and quantification were between 5 and 70 ng/zone and between 10 and 4000 ng/zone, respectively. Recovery was between 95.4 and 102.5 %.

herbal, HPTLC, quantitative analysis, densitometry, Validated RP-HPTLC method for simultaneous determination of vanillin and related 32e

100 159 R.N. SHARMA*, M.S. BAGUL, S.C. CHATURVED, K.K. VASU, M. RAJANI (*B. V. Patel Pharmaceutical Education and Research Development (PERD) Centre, Thaltej, Ahmedabad, India): Validated TLC densitometric method for the quantification of paroxetine hydrochloride in solid dosage form. Indian J. Pharm. Sci 69(3), 436 (2007). HPTLC of paroxetine hydrochloride on silica gel with ethyl acetate - acetic acid - water 15:3:2 with chamber saturation for 15 min. Densitometric evaluation at 296 nm. The method was linear in the range of 160 - 960 ng/zone. Limit of detection was 60 ng/zone. Recovery was 100.8 %.

pharmaceutical research, HPTLC, densitometry, quantitative analysis 32a

100 160 S. SHENOY, M.G. PAI, Dattesh BERLEKAR (*Goa College of Pharmacy, Panaji, Goa, India): Development & validation of a sensitive method for the quantitative analysis of atenolol-losartan potassium in antihypertensive combination by using HPTLC. 59th Indian Pharmaceutical Congress F-24, 419, (2007). HPTLC of atenolol and losartan potassium on silica gel with ethyl acetate - methanol - 1,4 dioxane - ammonia 10:2:1:2 with chamber saturation for 10 minutes. Densitometry at 225 nm. Linearity was between 200 and 1000 ng/zone for both compounds. Recovery was between 97.9 and 99.5%.

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

100 161 P. SHETTY*, K. MANGAONKAR, R. T. SANE (*F-13 Analytical Chemistry Laboratory, S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai 400 019, India; prabhagshetty@rediffmail.com) : HPTLC determination of ursolic acid in Alstonia scholaris R. Br. J. Planar Chromatogr. 20, 65-68 (2007). HPTLC of ursolic acid on silica gel with toluene - ethyl acetate - triethylamine - methanol 7:2:1:1 in a saturated twin-trough chamber. Quantitation by densitometry at 366 nm.

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

100 162 S. SHRIKUMAR, T.K. RAVI, R. CHITRA* (*College of Pharmacy, SRIPMS, Coimbatore, Tamil Nadu, India): A HPTLC fingerprint analysis of various commercial raw materials of Nothapodytes foetida using camptothecin as standard. 59th Indian Pharmaceutical congress F-217, 442, (2007). HPTLC of chloroform, ethanol and water extracts of Nothapodytes foetida on silica gel with toluene - ethyl acetate - glacial acetic acid 25:5:1. Densitometry at 366 nm. The hRf value of camptothecin was 22. Linearity was between 0.5 and 5 ng/zone. All commercial samples of the plant had a similar fingerprint profile.

herbal, densitometry, HPTLC, review 32e

100 163 S. SHRIKUMAR, T.K. RAVI, S.K. DEB* (*College of Pharmacy, SRIPMS, Coimbatore, Tamil Nadu, India): An HPTLC fingerprint analysis of Euphorbia hitra from various geographical locations using quercetin as standard. 59th Indian Pharmaceutical congress F-218, 442, (2007). HPTLC of alcoholic extracts of Euphorbia hitra on silica gel with toluene - ethyl acetate - glacial acetic acid 12:8:1. Densitometry at 377 nm. The hRf value of quercetin was 28. The calibration

range was 10-60 ng/zone. All the flavanoid constituents such as quercetin were well separated. The plant material collected from different locations showed almost similar fingerprint profile.

herbal, HPTLC, densitometry 32e

100 164 N. SHRIVASTAVA*, A. KOTHARI, T. PATEL, M. NIVSARKAR (*B V. Patel Pharmaceutical Education and Research Development Centre, Ahmedabad, India): Phytochemical evaluation and radical scavenging activity of three members from the family of asteraceae. Indian Drugs 44(10), 751 (2007). TLC and HPTLC of methanolic extracts of Eclipta alba, Launaea nudicaulis and Tridax procumbens, on silica gel with toluene - ethyl acetate - methanol 14:5:1. Evaluation under 254 and 366 nm. Detection by spraying with anisaldehyde sulfuric acid followed by evaluation at 525 nm.

pharmaceutical research, clinical chemistry research, herbal, HPTLC, densitometry, postchromatographic derivatization, qualitative identification 32e

100 165 A.P. SINGH, D.P. SINGH, S. SRIVASTAVA, R. GOVINDARAJAN, A.K.S. RAWAT* (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Lucknow-226001, India; pharmacognosy1@rediffmail.com): A validated quantitative HPTLC method for analysis of biomarkers in Ficus carica L. J. Planar Chromatogr. 20, 437-441 (2007). HPTLC of the 4 biomarkers bergapten, psoralen, rutin, and chlorogenic acid, on silica gel with ethyl acetate - formic acid - acetic acid - water 20:2:2:5 for chlorogenic acid and rutin and with petroleum ether - diethyl ether - acetic acid 5:5:1 for bergapten and psoralen in a saturated twin-trough chamber. Quantitative determination at 317 nm.

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

100 166 R.M. SINGH, S.C. MATHUR*, P. SINGH, O. PRAKASH, D.K. SHARMA, P.K. SAINI, G.N. SINGH (*Central Indian Pharmacopoeia Laboratory, Govt. Of India, Ministery of Health and Family Walfare, Ghaziabad, Uttar Pradesh, India): HPTLC method for the determination of cinnamaldehyde in Cinnamomum zeylenicum bark powder. 59th Indian Pharmaceutical congress F-225, 443, (2007). HPTLC cinnamaldehyde in the bark powder of Cinnamomum zeylenicum on silica gel with toluene - ethyl acetate - formic acid 190:10:1. Densitometric evaluation at 295 nm for quantification. The method was linear within the range of 31 and 157 ng/zone. The identity of the compound was confirmed by over overlaying the UV spectra of sample and standard. Cinnamomum bark was found to contain 0.25 % of cinnamaldehyde. Limit of detection and quantification was 3000 and 9900 ng/mL, respectively.

herbal, HPTLC, quantitative analysis 32c

100 167 R. SKIBINSKI*, L. KOMSTA, G. MISZTAL (*Department of Medicinal Chemistry, Medical University of Lublin, 4 Jaczewskiego Str., 20-090 Lublin, Poland; robert.skibinski@am.lublin.pl): The reversed-phase retention behavior of some atypical antipsychotic drugs. J. Planar Chromatogr. 20, 75-80 (2007). TLC of amisulpride, clozapine, olanzapine, quetiapine, risperidone, and ziprasidone on RP-18, RP-8, amino phase, cyano phase and DIOL phase in horizontal chambers with mixtures of phosphate buffer and six modifiers (acetone, acetonitrile, dioxane, ethanol, methanol, and tetrahydrofuran). Best separation on RP-8 with dioxane - phosphate buffer pH 3.51 2:3. Detection under UV light at 254 nm and evaluation by videodensitometry.

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

100 168 P.V. SRINIVAS, S. ANUBALA, V.U.M. SARMA, B.S. SASTRY, J.M. RAO* (*Natural Products Laboratory, Organic Division-I, Indian Institute of Chemical Technology, Hyderabad-500 007, India; janaswamy@iict.res.in): A new, convenient method for quantitative analysis of hedychenone, an anti-inflammatory compound in the rhizomes of Hedychium spicatum (Buch-Hem). J. Planar Chromatogr. 20, 73-74 (2007). HPTLC of hedychenone (a trimethyldecalin terpene) on silica gel with n-hexane - ethyl acetate 4:1 in a saturated twin-trough chamber. After development the plates were dried at 105 °C for 20 min. Densitometric evaluation in absorbance mode at 254 nm.

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

100 169 S. SRIVASTAVA*, A.K.S. RAWAT (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Lucknow, India; sharad_ks2003@yahoo.com; pharmacognosy1@rediffmail.com): Simultaneous determination of bergenin and gallic acid in different Bergenia species. J. Planar Chromatogr. 20, 275-277 (2007). HPTLC of bergenin and gallic acid on silica gel with ethyl acetate - formaldehyde - acetic acid - water 80:1:2:1 in a saturated twin-trough chamber. Quantitation was performed by scanning at 260 nm in absorption mode.

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

100 170 C. SUDHA*, A.C. KUMAR, R. MYTHREYI, P. THOMAS, V. MADHAVAN (*M.S. Ramaiah College of Pharmacy, Bangalore, Karnataka, India): HPTLC fingerprinting of E-guggulsterone in some marketed ayurvedic formulation. 59th Indian Pharmaceutical Congress C-42, 234, (2007). HPTLC of E-guggulsterone (a steroidal ketone present in oily resin of Commiphora mukul) and five marketed ayurvedic tablet formulations, on silica gel with toluene - propane-1-ol - glacial acetic acid 8:1:1. Densitometry at 254 nm. The hRf of E-guggulsterone was 61. Several marketed formulations were evaluated for E-guggulsterone, the UV spectra of the standard and that of the formulations were comparable in respect of E-guggulsterone. The TLC pattern showed several other zones corresponding to unknown phytochemicals. The method was suitable for quantitative evaluation of formulation in respect of E-guggulsterone in presence of other phyto chemicals.
pharmaceutical research

traditional medicine, quality control, HPTLC, densitometry, quantitative analysis postchromatographic derivatization 32e

100 171 A. SUGANTHI, N. BHARATHI*, K. TLAIYARAJA, T.K. RAVI (*College of Pharmacy, Shri Ramakrishna Institute of Paramedical Sciences, Coimbatore, Tamil Nadu, India): Development and validation of HPTLC method for the estimation of duloxetine hydrochloride from tablet formulation. 59th Indian Pharmaceutical congress F-135, P-2122, (2007). HPTLC of duloxetine hydrochloride on silica gel with toluene - ethyl acetate - glacial acetic acid 14:6:3. Quantification at 232 nm. The hRf value of duloxetine hydrochloride was 40. Linearity was between 100 and 500 ng/zone. Limit of detection and quantification was 20 ng/zone and 50 ng/zone, respectively.

pharmaceutical research, HPTLC, quantitative analysis, densitometry 32a

100 172 B.N. SUHAGIA, I.S. RATHOD, S.A SHAH, S. SUNIL* (*L. M. College of Pharmacy, Ahmedabad, Gujarat, India): Chromatographic determination of oleanolic acid in the seeds of Achyranthes aspera. 59th Indian Pharmaceutical congress F-11, 392, (2007). HPTLC oleanloic acid from seeds of Achyranthes aspera on silica gel with n-hexane - ethyl acatete - acetic acid 30:201. Detection by spraying with anisaldehyde - sulphuric acid reagent. Densitometry at 530 nm for quantification of oleanolic acid. Linearity was between 200 and 1200 ng/zone. The plant tree was found to contain 0.34 % oleanloic acid. The method can be used for routine quality control.

pharmaceutical research, traditional medicine, HPTLC, quantitative analysis, postchromatographic derivatization, densitometry 32e

100 173 R. SULTANA*, S. KHANAM, K. DEVI (*AL-AMEEN COLLEGE OF PHARMACY, BANGALORE, KARNATAKA, INDIA): HPTLC fingerprinting and immunomodulatory activity of Solanum xanthoarpum and Solanum trilobatum. 59th Indian Pharmaceutical congress C-324, 302, (2007). HPTLC of aqueous and alcoholic extracts of different plant parts (fruits, leaves, stem and root) of Solanum xanthocarpum and Solanum trilobatum, on silica get with toluene - ethyl acetate - diethyl amine 7:2:1. Detection at 254 nm and 366 nm, and by spraying with antimony tretrachloride and vanilin - sulphuric acid.

pharmaceutical research, traditional medicine, herbal, postchromatographic derivatization quantitative analysis, HPTLC 32e

100 174 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; pasathe2001@hotmail.com): Determination of rutin in Amaranthus spinosus Linn. Whole plant powder by HPTLC. Chromatographia 65 (11-12), 767-769 (2007). HPTLC of rutin in the whole plant powder of Amaranthus spinosus Linn. on silica gel with ethyl acetate - formic acid - methanol - distilled water 100:9:11:17. Quantification by densitometry at 363 nm. Linearity was between 10 and 60 µg/mL for rutin. The concentration of rutin in the whole plant powder was found to be 0.15 %.

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

100 175 J.V. SUSHEEL*, S. MALATHI, T.K. RAVI (*Department of Pharmaceutical Analysis, College of Pharmacy, SRIPS, Coimbatore, Tamil Nadu, India): Analysis of ropinirole in tablet dosage form. Indian J. Pharm. Sci. 69(4), 589 (2007). HPTLC of ropinirole on silica gel with methanol - acetone 4:1. Aripiprazole was used as internal standard. Under chromatographic conditions both ropinirole and aripiprazole were well separated. Evaluation under UV 254 nm. UV spectrometry was carried out at 250 nm.

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

100 176 V.V. VAIDYA*, S.N. MENON, G.R. SINGH, M.B. KEKARE, M.P. CHOUKEKAR (*Therapeutic Drug Monitoring Laboratory, 194, Scheme No. 6, Road No. 15, Sion Koliwada, Sion (East), Mumbai-400 022, India; vaidya_vikas@yahoo.com; ganeshsingh10@yahoo.com): Simultaneous HPTLC determination of clotrimazole and tinidazole in a pharmaceutical formulation. J. Planar Chromatogr. 20, 145-147 (2007). HPTLC of clotrimazole and tinidazole on silica gel in a twin-trough chamber with toluene - ethyl acetate - methanol - glacial acetic acid 60:30:10:3 with chamber saturation for 10 min. Quantitation by scanning at 254 nm.

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

100 177 A. VELMURUGAN,* S.J. VARGHESE, Joseline JOSE, T.K. RAVI (*College of Pharmacy, Shri Ramkrishna Institute of Paramedical Sciences, Coimbatore, Tamil Nadu, India): Development & Validation of HPTLC method for simultaneous determination of nebivolol & hydrochlorothiazide in tablet dosage form. 59th Indian Pharmaceutical Congress F-65, 405, (2007). HPTLC of nebivolol and hydrochlorothiazide in on silica gel with n-butyl-acetate - formic acid - chloroform 7:1:2. Densitometry at 273 nm. The hRf value of nebivolol was 21 and of hydrochlorothiazide 46. Linearity was between 200 and 600 ng/zone for nebivolol and between 500 and 1000 ng/zone for hydrochlorothiazide. The limit of quantification was 60 ng and 40 ng/zone for nebivolol andhydrochlorothiazide, respectively. Recovery was 102 - 105 % for both compounds. pharmaceutical research

quality control, HPTLC, quantitative analysis, densitometry 32a

100 178 K. WIECKOWSKI, A. CZAJA, A. WOZNIAK, A. MUSIAL, Barbara MALAWSKA* (*Department of Physicochemical Drug Analysis, Jagiellonian University Medical College, Faculty of Pharmacy, Medyczna 9, 30-688 Kraków, Poland; mfmalaws@cyf-kr.edu.pl): A study of the lipophilicity of amide derivatives of alpha-(1,2,3,4-tetrahydroisoquinolin-2-yl)-gamma-hydroxybutyric acid by use of RP-TLC and calculation. J. Planar Chromatogr. 20, 101-106 (2007). TLC of 10 derivatives of N-benzylamides and N-phenylethylamides of alpha-(1,2,3,4-tetrahydroisoquinolin-2-yl)-gamma-hydroxybutyric acid on RP-18 with mixtures of methanol and water after chamber saturation. Visualization under UV light at 254 nm. Retention data obtained by use of this method were linearly dependent on methanol concentration and enabled estimation of relative lipophilicity corresponding to pure water as mobile phase.

pharmaceutical research, qualitative identification 32a

33. Inorganic substances

100 179 M. CURTUI, Maria-Loredana SORAN* (*National Institute of Research and Development for Isotopic and Molecular Technology, 72-103 Donath Street, 400293 Cluj-Napoca, Romania; loredana_soran@yahoo.com): Use of di(n-butyl) and di(iso-butyl)dithiophosphoric acids as complexing agents in the TLC separation of some d and f transition metal ions . J. Planar Chromatogr. 20, 153-158 (2007). TLC of U(VI), Th(IV), lanthanides(III), Co(II), Ni(II), and Cu(II) on silica gel with di(n-butyl) and di(iso-butyl)dithiophosphoric acid as complexing agents in different organic solvents (polar and nonpolar). Visualization with 0.05 % arsenazo III for U(VI), Th(IV), and lanthanides(III), and with 0.1 % rubeanic acid in ethanol for Co(II), Ni(II), and Cu(II). Densitometric evaluation at 505 nm for Cu(II) and Co(II) and at 600 nm for Th(IV), U(VI), and Ni(II).

qualitative identification 33a

100 180 P.A.M. NAJAR*, R.N. CHOUHAN, J.U. JEURKAR, S.D. DOLAS, K.V.R. RAO (*Jawaharlal Nehru Aluminium Research Development and Design Centre, Amaravati Road, Nagpur, India): Thin-Layer Chromatography of Aluminium: Quantitative densitometric determination of Fe2+, Ni2+, Cu2+, and Si4+. J. Chromatogr. Sci. 45 (5), 263-268 (2007). TLC of microgram levels of iron, silicon, copper, nickel, titanium, magnesium, manganese, and zinc present in a high concentration aluminium matrix, on silica gel with aqueous sodium chloride solution. Quantification by densitometry. Comparison of the densitometric quantitative determination results of iron, silicon, nickel, and copper with the respective optical emission spectral analytical data.

comparison of methods, HPTLC, qualitative identification, quantitative analysis 33

100 181 Vukosava ZIVKOVIC-RADOVANOVIC*, Gordana VUCKOVIC (*Faculty of Chemistry, University of Belgrade, P.O. Box 158, 11001 Belgrade, Serbia): Use of different salt solutions in salting-out TLC of Co(III) complexes on silica gel. Chromatographia 67 (3-4), 259-267 (2008). Investigation of saturated aqueous solutions of 28 different salts used as potential mobile phases for salting-out TLC, on silica gel with a series of four mixed bis-aminocarboxylato cobalt(III) complexes. Confirmation of three alkali metal chlorides, and four alkaline earth metal chlorides, four linear dependences previously established on different adsorbents with (NH4)2SO4 solutions by linear regression analysis of chromatographic data obtained for fifteen mixed aminocarboxylato Co(III) complexes (four series) with solutions of ammonium chloride. With Li+, Mg2+, and Ca2+ chlorides the best separation was achieved.

quantitative analysis, HPTLC 33

35. Other technical products and complex mixtures

100 182 D. JUN, P. STODULKA, V. KOLECKAR, K. KUCA* (*Center of Advanced Studies and Department of Toxicology, Faculty of Military Health Sciences, University of Defense, Hradec Kralove, Czech Republic; kucakam@pmfhk.cz): TLC identification of benzalkonium bromide homologs. J. Planar Chromatogr. 20, 283-285 (2007). TLC of benzalkonium bromide homologs (with C2, C4, C6 to C16, C18, C20) on silica gel in twin-trough chambers with isopropanol - water - acetic acid 1:1:4 or methanol - chloroform - acetic acid 50:10:1. Detection with iodine vapor or by derivatization with Dragendorff‘s reagent.

environmental, qualitative identification 35a

100 183 A. MIRZAIE, A. JAMSHIDI, S.W. HUSAIN* (*Department of Chemistry, Faculty of Science, Science and Research Campus, Islamic Azad University. P. O. Box 14155-4933, Poonak-Hesarak, Tehran-14778-93855, Iran; syedwhusain@yahoo.com): Quantitative ion-exchange TLC of p-hydroxybenzoic acid in the presence of preservatives. J. Planar Chromatogr. 20, 303-306 (2007). TLC of p-hydroxybenzoic acid, methyl p-hydroxybenzoate, ethyl p-hydroxybenzoate, propyl p-hydroxybenzoate, benzoic acid, sodium benzoate, sorbic acid, potassium sorbate, salicylic acid, butylated hydroxyanisol, and butylated hydroxytoluene on stannic silicate with n-hexane - ethyl methyl ketone - acetic acid 80:20:3. Quantitation by scanning densitometry at 270 nm. The limit

of detection and quantitation for p-hydroxybenzoic acid was 0.05 and 0.51 µg/zone, respectively. food analysis, quantitative analysis

qualitative identification, densitometry 35b

100 184 A. MOHAMMAD*, N. HAQ (*Analytical Research Laboratory, Department of Applied Chemistry, Aligarh Muslim University, Aligarh-202002, U. P., India; mohammadali4u@rediffmail.com): Selective separation of dodecyltrimethylammonium bromide from other cationic and nonionic surfactants. J. Planar Chromatogr. 20, 347-351 (2007). TLC of DTAB on soil, silica gel, alumina, and kieselguhr with fifteen mobile phases, such as aqueous solutions of ammonium sulfate and urea, with chamber saturation for 10 min. Detection by spraying with modified Dragendorff reagent. Among the systems studied the best system for selective separation of DTAB from multicomponent mixtures of other surfactants was kieselguhr - 0.1 M ammonium sulfate. Semi-quantitative determination by spot-area measurement.

cosmetics, environmental, qualitative identification 35a

37. Environmental analysis

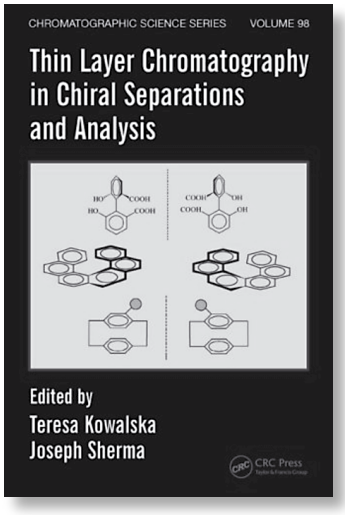
100 185 R. ZHANG* (Zhang Rong), Y. YUE (Yue Yongde), R. HUA (Hua Rimao), W. YAN (Yan Wen) (*Recourses and Environment College of Anhui Agricultural University, Agri-food Security Key Lab of Anhui Province, No. 130, Changjiang West Road, Hefei, China; z_rong163@163.com): Factors affecting the separation of phthalate esters, and their analysis, by HPTLC. J. Planar Chromatogr. 20, 321-326 (2007). Investigation of factors affecting the separation, including the use of different stationary and mobile phases, different methods of development, humidity, and chamber saturation. TLC and HPTLC of dimethyl, diethyl, di-n-butyl, and bis-(ethylhexyl) phthalate on silica gel, prewashed with chloroform - methanol 1:1 or the mobile phase, in horizontal chambers, Vario chambers, and twin-trough chambers with 12 different mobile phases. Best separations were achieved with hexane - acetone 4:1 or hexane - toluene - ethyl acetate 9:8:3. Densitometric evaluation at 220 nm.

environmental, quantitative analysis, HPTLC 37c

38. Chiral separation

100 186 P. PATEL*, R. MASHRU (Dept. Pharmaceutics, Ramanbhai Patel College of Pharmacy, Changa, Gujarat, India): Development of a direct TLC method for separation of isomers of carvedilol using beta-cyclodextrin as a chiral selector in stationary phase. 59th Indian Pharmaceutical congress F-211, 440, (2007). TLC of isomers of carvedilol on chiral silica gel (prepared with beta-cyclodextrin) with methanol - water 5:1. Evaluation at 366 nm. The R(+) and the S(-) isomer of carvedilol had hRf values of 89 and 81, respectively. The linearity range was 50 - 500 µg/zone. Limit of detection and quantification was 10, 12 and 40, 42 µg/zone respectively for R(+) and S(-) isomers. The preparation of R(+) and S(-) isomers of carvedilol was found to be in the ratio 3:2 in bulk and formulations.

pharmaceutical research, densitometry 38



Thin Layer Chromatography in Chiral Separations and Analysis.

Chromatographic Science Series, Volume 98.

Edited by Teresa Kowalska and Joseph Sherma.

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Reading about chiral separations and analysis, most scientists do have in mind HRGC or HPLC on chiral phases, but will be rather surprised that thin layer chromatography (TLC) also can be a tool for chiral separations, which is the merit of Teresa Kowalska and Joseph Sherma as editors of this volume 98 of Chromatographic Science Series. It is the first book providing theoretical basics, principals, capabilities and applications of TLC for the direct and indirect enantioseparations.

The book is structured in 15 chapters first presenting techniques of enantioseparations on TLC followed by different applications mainly in the field of pharmaceutical products and drugs.

After a general introduction (chapter 1) a thorough tutorial on the origin and biological importance of chirality including mechanistic concepts of enantiomer recognition is given. Commercial and non-commercial precoated layers for enantiomer separations are the topic of chapter 4 and 5, respectively, while the capabilities of chiral additives to the mobile phase are treated in chapter 6. In the middle of the book, surprisingly, an overview of chiral separations mechanisms is given, which would be expected as one of the first chapters. Most interesting bottlenecks of densitometric detection of chiral analytes are discussed in chapter 9. It was shown that enantiomers separated on L-arginine impregnated silica not only are separated in vertical direction of development, but also clearly show a left and right drift for the (R)- and (S)-compounds, respectively. Both oszillatory transenantiomerizations and changes in UV spectra are discussed as additional originalities to be respected in chiral analyses.

A review of chirality of pharmaceutical products is introductorily given in chapter 10 followed by examples of chiral TLC separations as β-adrenergic antagonists (chapter 11), amino acids (chapter 12), nonsteroidal anti-inflammatory drugs (chapter 13), and components in selected chiral drugs (chapter 14). The final chapter 15 covers chiral separations using Marfey's reagent.

(HP)TLC as modern instrumental technique of planar chromatography is a powerful tool of liquid chromatography. However, TLC for chromatographic enantioseparations is far less common. TLC is rather flexible concerning pre- and postchromatographic derivatizations, accessible to multi-mode detections and quantifications, matrix-tolerant, and rapid and cost-effective. Why not TLC even in the case of enantioseparations? Of course, it cannot compete with the most important enantio-GC of flavour compounds, but is quite an alternative for polar compounds to be derivatized prior GC as, e.g., amino acids or drugs, or especially for compounds lacking in usable chromophores detectable by HPLC/UV. Finally, combinatorial chemistry needs rapid methods to simultaneously analyze a couple of samples.

With Thin Layer Chromatography in Chiral Separations and Analysis the editors succeeded very well in producing a comprehensive, yet accessible, source of information, having won respected specialists in this field. It provides an excellent overview of the subject both in theoretical and practical aspects. A plenty of relevant references in each chapter supports a more detailed literature study for special applications.

Professor Dr. W. Schwack

University of Hohenheim
Stuttgart, Germany

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

City of Helsinki, Picture Bank/Photo Niko Soveri

Preliminary Speaker Program

News, fundamentals and theoretical aspects

1. Quantitative micro planar chromatography – MPLC
   Rudolf Kaiser, Institute for Chromatography, Bad Duerkheim, Germany
2. Pressurized planar electrochromatography – challenges and perspectives
   Tadeusz Dzido, Medical University, Lublin, Poland
3. A comparative study of molecular lipophilicity indices of some formyl- and acetylpyridine-3-thiosemicarbazone derivatives estimated by RP- HPTLC and calculated Log P values
   Costel Sârbu, Babes-Bolyai University, Cluj-Napoca, Romania
4. The orthogonality of the selectivity space in reversed-phase thin-layer chromatography
   Colin Poole, Wayne State University, Detroit, MI, USA
5. Ultrathin layer chromatography on plates with engineered nanostructure
   Louis Bezuidenhout, University of Alberta, Edmonton, Canada

Specific applications

1. Determination of thiouracils in thin-layer chromatography with iodine-azide detection procedure
   Robert Zakrzewski, University of Łódz, Łódz, Poland
2. Complementarity of TLC and HPLC in investigation of triterpenoids in plant extracts
   Irena Vovk, National Institute of Chemistry, Ljubljana, Slovenia
3. HPTLC for the analysis of API-cleaning samples
   Ties Raijmakers, Organon (Schering-Plough Corporation), Oss, The Netherlands
4. Fermentation monitoring based on HPTLC-OPLC technique: the effect of a complex biological matrix on quantification performances
   Tatiana Bernardi, University of Ferrara, Ferrara, Italy
5. A simple, accurate and rapid HPTLC method for the determination of theophylline in post mortem blood and its validation
   B. Mohan, Forensic Science Laboratory, Bangalore, India
6. Drug screening in autopsy liver samples by overpressured layer chromatography
   Anna Pelander, University of Helsinki, Helsinki, Finland
7. Stability-indicating HPTLC determination of clozapine in tablet dosage form
   Zahid Zaheer, Y.B. Chavan College of Pharmacy, Maharashtra, India
8. Development of an HPTLC method for amino acid identification in peptide
   Roseline Sbaffo-Poasevara, IPSEN, Les Ulis, France

Strong features of HPTLC

1. Changes in emission induced by non-covalent analyte-fluorophore interactions in silica gel as a general detection procedure for thin-layer chromatography
   Vicente Cebolla, CSIC, Zaragoza, Spain
2. New test-kits to detect herbicide effects and resistance in weed plants based on HPTLC-screening
   Helle Weber Ravn, Aarhus University, Silkeborg, Denmark
3. Changes in glycoalkaloid composition during potato processing: simple and reliable quality control via HPTLC
   Jens Mäder, Berlin University of Technology, Berlin, Germany
4. HPTLC analysis of radioactive metabolites of 6-[18F]FDOPA after modulation by enzyme inhibitors
   Sarita Forsback, University of Turku, Turku, Finland
5. Is there a fine future for HPTLC in the modern API plants? Actual experiences
   Louise Vicard, Sanofi-Aventis, Neuville-sur-Saône, France

Specific detection, bioactivity tests, and coupling techniques

1. A new method for the quantification of Quats by TLC
   Bernd Spangenberg, University of Applied Sciences Offenburg, Offenburg, Germany
2. A new multi-enzyme inhibititon test for the detection of insecticidal organophosphates and carbamates by HPTLC
   Wolfgang Schwack, University of Hohenheim, Stuttgart, Germany
3. HPTLC/AMD with Vibrio fischeri as an example for bioactivity-based detection – A new dimension in analytics
   Wolfram Seitz, Zweckverband der Landeswasserversorgung, Langenau, Germany
4. Advanced mass spectrometric approaches for the analysis of analytes on planar separation media
   Vilmos Kertesz, Oak Ridge National Laboratory, Oak Ridge, TN, USA
5. Combined TLC-MALDI analysis of lipids
   Jürgen Schiller, University of Leipzig, Leipzig, Germany
6. Automated coupling of planar chromatography with mass spectrometry
   Gertrud Morlock, University of Hohenheim, Stuttgart, Germany

The poster presentations and the Manufacturers' session are announced at **www.hptlc.com**.

Deadline for registration and last minute poster: May 15th
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Controlling best the drying step

9

▲ *Dr. Matthias Loppacher, Head of R & D*

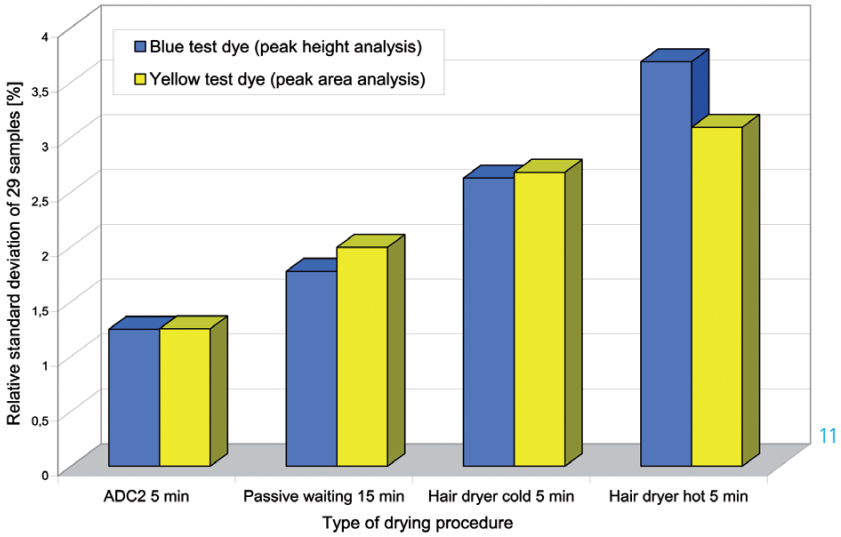
The new automatic developing chamber (ADC 2) has been designed focussing on the reproducibility of the entire development process. This involves also the control of external factors such as the relative humidity and the drying. The new closed loop circulating system accelerates and directs a high velocity air stream across the surface of the HPTLC layer thus enabling short and uniform conditioning of the HPTLC plate.

10

▲ *The ADC 2 for automatic TLC development and reproducible chromatography features the new closed loop system.*

Several papers have demonstrated that the drying procedure contributes significantly to the overall experimental variability of the TLC system. If the closed loop system described above is operated as an open loop system, it demonstrates excellent performance for rapid and heatless drying of the plate after chromatography, which immediately stops any diffusion. This is particularly valuable since any diffusion, which occurs as long as the chromatographic layer is still wet, will unnecessarily broaden the peaks in the chromatogram.

During development of the new drying system, systematic studies were carried out to evaluate the variabilities associated with different drying methods. It was demonstrated that homogeneous, rapid, and heatless drying by the ADC 2 reduced the overall standard deviation by up to a factor of 3, compared to manual use of a hair dryer. Inhomogeneous drying (hair dryer) or diffusion due to an overly long drying time (passive waiting) both cause higher overall uncertainties.



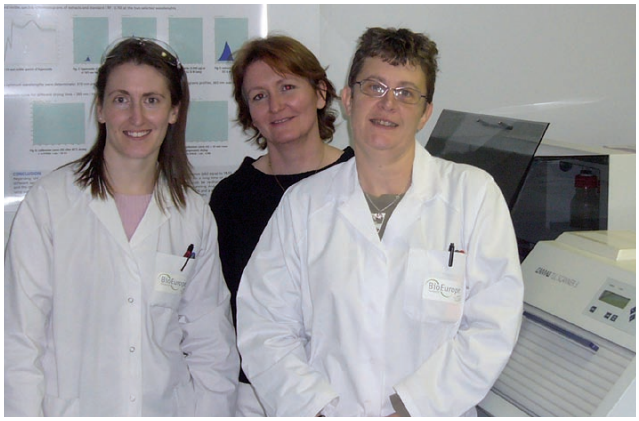
11

▲ *Comparison of different drying procedures showing the best control of the drying step by the ADC2 compared to other common practises*

A further important feature of the ADC2 is the reproducible chromatogram development. Depending on its current state of activity, the TLC plate will adsorb or desorb water until equilibrium with the air stream is reached. Finally, the air stream is recycled and fed back into the conditioning unit. This closed loop allows the air stream to be easily kept clean, as well as enabling a compact, effective system to be constructed.

A final important consideration is that all the methods which have been developed for conventional twin trough chambers are compatible with the system. Manual operations, which may be critical and difficult to repeat on a daily basis, can now be automated, and influences from environmental factors can be eliminated.

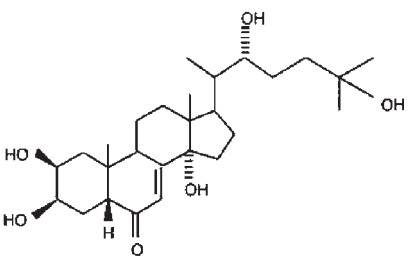
Quantification of ß-ecdysone in a brasilian ginseng juice *(Pfaffia glomerata)*



▲ *BioEurope research center in Anet (80 kms from Paris): Mrs. Agnès Gaspar, Mrs. Sophie Leclere*, Mrs. Véronique Marignier (from left to right)* 12

BioEurope, founded in 1985, merged with the French company Solabia in 1992. It is active in the production and marketing of intermediary products in the pharmaceutical, cosmetic, fermentation and diagnostic fields, and is European leader in peptone production for fermentation. Based on its expertise in enzyme-catalyzed synthesis processes, it also develops different kinds of plant extracts for cosmetic applications.

BioEurope has employed HPTLC since 2003 and currently uses more than 15 validated quantitative methods, such as quantification of caffeine and theobromine in cacao extracts, asiatic and madecassic acids in Centella asiatica extracts, and hyperoside in walnut leaves. HPTLC is helpful for the optimization of the extraction process and for characterization of raw materials or extracts through the specific quantification of active ingredients found in the plants.



◀ *Structure formula of ß-ecdysone*

Introduction

Pfaffia glomerata is called Brazilian ginseng for its nutritional and prophylactic properties, which closely resemble those of the so-called traditional ginseng. But Brazilian ginseng is very different from *Panax ginseng*. It grows on the south-facing slopes of the humid mountains in the center of the Brazilian forest. It belongs to the Amaranthaceae family and possesses a large, tuberous root from which it is possible to extract a sap via the process of cold pressure. The plant owes its quality to the following ingredients:

- Antioxidants such as selenium and polyphenols
- Saponins (nortriterpenoids and pfaffosides)
- Amino acids: arginine, lysine, histidine, glycine
- Steroids (ß-ecdysone [1] for example)

ß-Ecdysone is a hormonal steroid which is responsible for the shedding phenomenon by insects and shell fish and is also found in certain plants. It shows interesting biological properties in relation to skin cell metabolism and has potential for cosmetic application [2]. The following method, based on [3], reports its quantification in a cold-pressed rhizome extract of *Pfaffia glomerata*.

Sample preparation

1 g of extract is dissolved in 10 mL of methanol in an ultrasonic bath for 5 min.

Standard solution

ß-Ecdysone is dissolved in methanol (0.04 g/L).

Chromatogram layer

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

Sample application

Bandwise with Automatic TLC Sampler 4, 16 tracks, band length 6 mm, track distance 11.3 mm, distance from the left side 15 mm, distance from lower edge 8 mm, application volume 5 and 3 µL for samples and 1 to 5 µL for the standard solution (0.04 to 0.2 µg/band).

Chromatography

In the twin-trough chamber 20 x 10 cm after 15 min chamber saturation with the lower phase of chloroform – methanol – water 7:5:2. The migration distance is 50 mm from the lower plate edge. Then the plate is dried at 80 °C for 5 min on the TLC Plate Heater.

Post-chromatographic derivatization

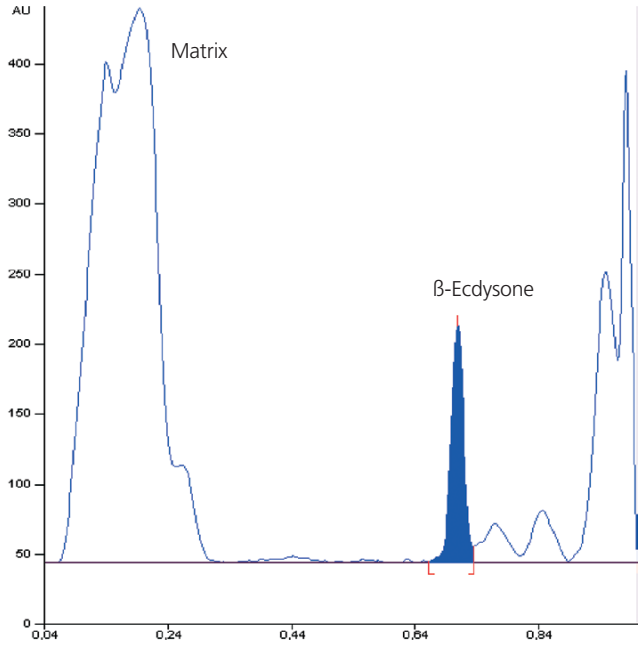
With the Chromatogram Immersion Device III the plate is dipped for 1 s into the anisaldehyde reagent (0.5 mL anisaldehyde is diluted with 10 mL glacial acetic acid, followed by 85 mL methanol and 5 mL concentrated sulphuric acid), followed by heating on the TLC Plate Heater at 120 °C for 20 min.

Densitometry

Absorption measurement at 432 nm with TLC Scanner 3 and winCATS software, polynomial calibration via peak area.

Results and discussion

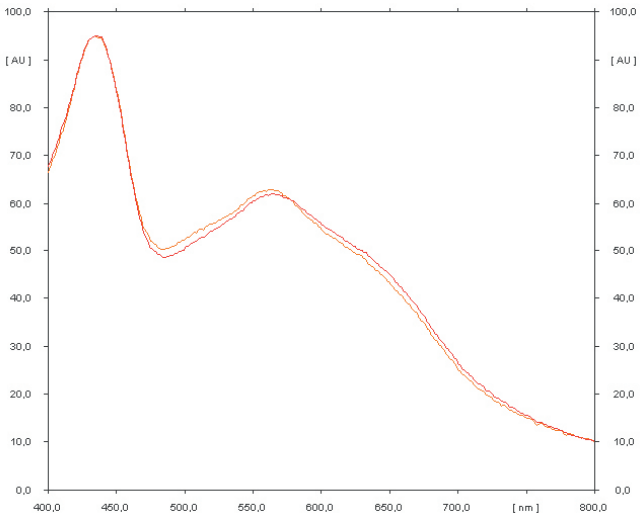
Good selectivity of the active ingredient ß-ecdysone and the matrix was obtained. A representative densitogram of a sample clearly shows the separation of ß-ecdysone from other sample constituents. By the previous HPLC method the matrix influenced the reproducibility negatively because it was spread all over the chromatogram and not as clearly fixed at the starting area as by the orthogonal HPTLC method.



13

▲ *Separation of ß-ecdysone (hR_F 71) from matrix constituents in a cold-pressed Pfaffia glomerata rhizome extract*

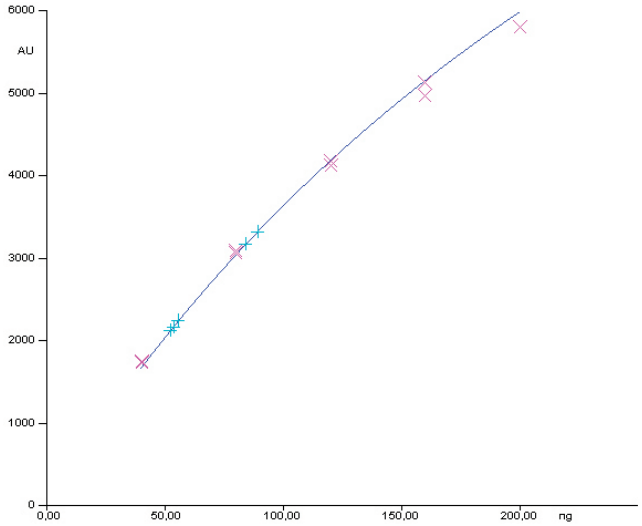
For densitometric evaluation the optimum wavelength of 432 nm was confirmed by in situ UV/Vis-spectra. Identity of ß-ecdysone was proven by spectra comparison of sample and standard.



14

▲ *Spectra comparison of ß-ecdysone sample and standard zone*

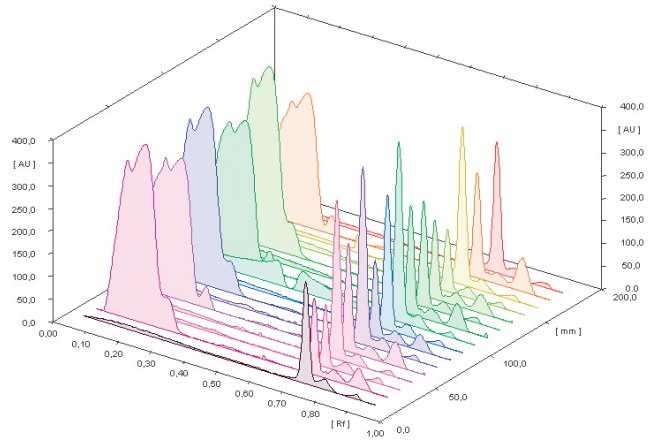
Calibration of ß-ecdysone was performed in the range of 0.04 to 0.2 µg/band (sdv 1.6 %). The evaluated extracts contained 0.4 to 0.6 g/kg ß-ecdysone, meaning 0.8 to 1.2 % ß-ecdysone was found in the original dried extract.



15

▲ *Calibration of ß-ecdysone (x) with samples (+)*

The parallel chromatography of several tracks on a plate – simultaneously and under identical chromatographic conditions – enabled the rapid determination of the active ingredient ß-ecdysone in the dry extract of the cold-pressed *Pfaffia glomerata* rhizome.



16

▲ *Parallel chromatography of 16 tracks under identical chromatographic conditions for rapid quantification of ß-ecdysone*

Further information is available on request from the authors.

[1] Shiobara Y. et al. Phytochemistry 32 (1993) 1527

[2] Detmart M. et al. Eur. J. Dermatol. 4 (1994) 558

[3] Wagner, H., Bladt, S.: Plant Drug Analysis, Springer, Heidelberg, 1996

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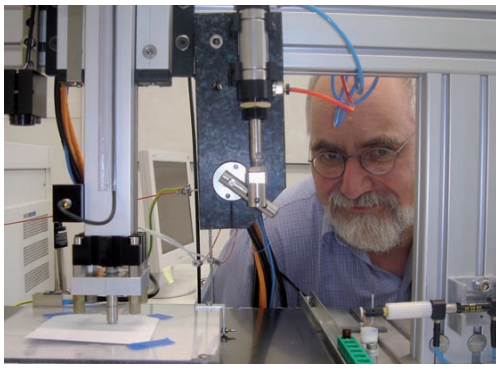
Automated HPTLC/ESI-MS coupling



17

▲ *Dr. Heinrich Luftmann*

In CBS 94 Dr. Luftmann, head of the department of mass spectrometry at the Institute of Organic Chemistry, Westphalian Wilhelm's-University in Muenster, reported about the rapid and contamination-free extraction of TLC zones for structure elucidation of synthesis mixtures by mass spectrometry. By now the positioning of the plunger was automated. Together with Professor Aranda and Dr. Morlock*, University of Hohenheim in Stuttgart, the hands-free recording of relevant zones of a whole HPTLC plate was demonstrated and the performance of the automated interface was established [1].

Scope

The reliability of the automated HPTLC/MS interface was investigated by its employment not only for identification, but also for quantification by electrospray ionization mass spectrometry (ESI-MS). The results obtained by HPTLC/MS were compared to validated HPTLC/UV methods and evaluated via caffeine quantification in energy drink samples [2] and headache tablets [3]. Compared to other coupling approaches an internal standard for correction was not employed. Hence the results directly reflect the interface's performance for HPTLC/MS coupling.

A strong advantage of the interface is that any given LC/MS system can be used without any modification. Compared to desorption techniques the whole zone including its depth profile can be extracted and enables a comparable detectability (pg/zone) to HPLC/MS [4]. Generally the time needed for mass spectrometric detection in HPTLC/MS is comparably low if related to HPLC/MS because after densitometric evaluation mass spectra can be recorded only from zones of interest (advantage by the off-line method).

Sample preparation

A) The energy drink sample was degassed for 20 min in the ultrasonic bath.
B) Five headache tablets were ground with the mortar. The mean weight of a tablet (0.6 g) was dissolved in a 50 mL-measuring flask in 40 mL methanol - water 7:3 by shaking (500/min) for 20 min and placing in the ultrasonic bath for 10 min, followed by filling up to the mark. An aliquot was filtrated (0.45 µm) and diluted 1:20 with methanol.

Standard solution

Caffeine is dissolved in methanol (0.1 mg/mL).

Layer

HPTLC aluminum foil silica gel 60 F_{254} (Merck), 10 x 10 cm

Sample application

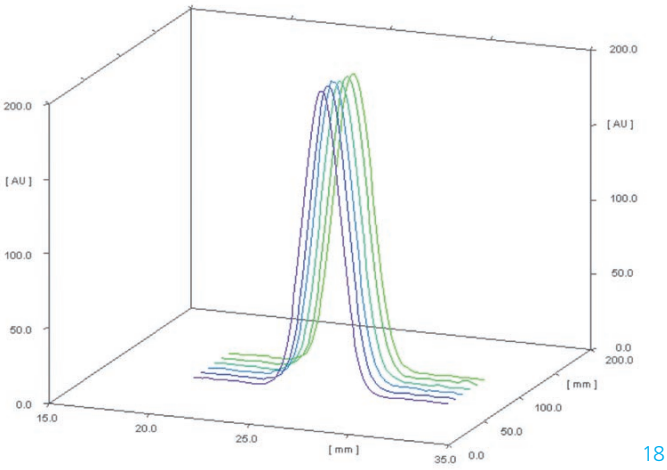
Bandwise with the TLC sampler ATS4, band length 3 mm, distance from lower edge 10 mm, tracks distance 7.2 mm, application volumes 0.5–5 µL (50–500 ng/band) for the standard solution and 1 µL for sample solutions (100 ng/band caffeine (tablet) and 320 ng/band (energy drink).

Chromatography

In the flat bottom chamber 10 x 10 cm with ethyl acetate – methanol – ammonia (25 %) 90:15:1 (tablet) or chloroform – ethanol – acidic acid (37 %) – acetone – water 54:27:10:2:2 (energy drink), migration distance 80 mm each from lower plate edge

Densitometry

Absorption measurement at UV 274 nm by TLC Scanner 3 and winCATS software; polynomial calibration via peak area



18

▲ *Absorption measurement scan (analyte section) showing the precision of 6 HPTLC zones (100 ng/band caffeine)*

Documentation

With DigiStore 2 documentation system; recording in reflection mode at UV 254 nm

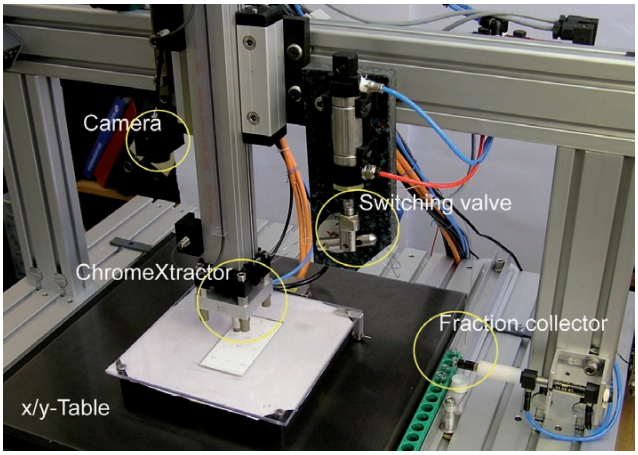

19

▲ *Documentation (plate section) of the calibration*

Automated online extraction and recording of mass spectra

The interface inlet capillary was connected to the pump (L6200, Merck-Hitachi, Tokyo, Japan) and the extraction was performed at a flow rate of 0.1 mL/min with methanol – ammonium formate (10 mmol/L) 19:1, pH 4. The interface outlet capillary was directly connected with the ESI-MS (Quattro LCZ, Waters-Micromass, Manchester, UK using Mass Lynx 3.2 software): capillary voltage 3.5 kV, cone voltage 42 V, extractor voltage 3 V, R_F lense voltage 0.28 V, source block temperature 110 °C, desolvation temperature 180 °C.

Once the HPTLC foil was placed on the x/y-table (20 x 20 cm) and automatically moved to the image recording position, a plate image was acquired by the web camera and transferred to the software, calculating the plate position (x,y-axis). Clicking at the zones on the screen of which a mass spectrum should be recorded, the elution pattern was defined. Then the zones were extracted in a 1.8 min-time via the oval extraction plunger (4 x 2 mm) and one by one transferred into the ESI-MS. Between the extraction steps the plunger head was cleaned for 4 s.

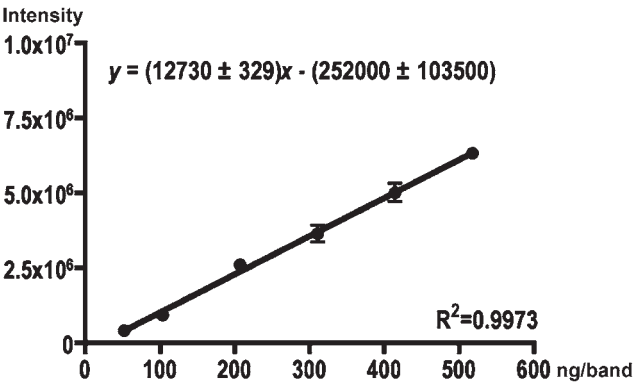


20

▲ *HPTLC/ESI-MS coupling: automated positioning and extraction of HPTLC zones*

Results and discussion

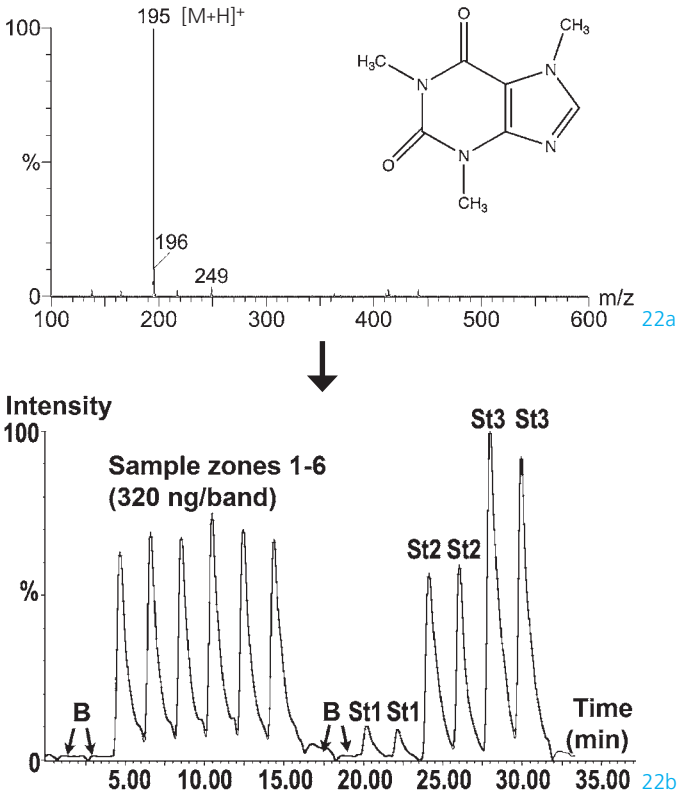
The full scan (*m/z* 100–600) clearly showed the protonated molecule at *m/z* 195 $[M+H]^+$. Without any internal standard the caffeine mass signal was recorded in the SIM (selected ion monitoring) mode at *m/z* 195 $[M+H]^+$. The validation of the HPTLC/ESI-MS method showed highly significant data, i.e. a linear regression with a determination coefficient r^2 of 0.9973, a repeatability (%*RSD*, *n* = 6) of 5.6 % and a reproducibility of the plate mean for 3 plates (%*RSD*, *n* = 3) of 1.5 %.



21

▲ *Calibration function via HPTLC/ESI-MS (SIM mode)*

Caffeine was quantified in headache tablets and energy drinks in the SIM mode at *m/z* 195 $[M+H]^+$.



▲ *Full scan (up) and SIM elution profile (bottom) of extracted caffeine zones*

The accuracy of the determination was investigated by the comparison with validated HPTLC/UV methods. According to the t- and F-test ($P < 0.01$, $\upsilon_1 = 5$, $\upsilon_2 = 4$) comparable results were obtained by the different detections methods.

Automated interface: HPTLC/ESI-MS versus HPTLC/UV

Caffeine in	Tablet Mean ± SD (mg/tablet)	Energy Drink Mean ± SD (mg/100 mL)
HPTLC/ESI-MS (%*RSD*, *n* = 6)	102,09 ± 5,76 (5,6)	32,91 ± 1,60 (4,9)
HPTLC/UV (%*RSD*, *n* = 5)	101,98 ± 2,30 (2,3)	33,71 ± 0,96 (2,8)
Label	100	32

The automated interface working in a 1.8 min-time was 20 % faster than the manual interface. If compared to the only other HPTLC/MS paper without any internal standard (%*RSD*s 7.6%, 14.4 % and 16.8 %, r^2 between 0.95 and 0.98 [5]), the precision (%*RSD* = 5.6 %) and the determination coefficient of the linear regression (r^2 = 0.9973) clearly showed that the interface can be considered as highly reliable for quantitative HPTLC/MS coupling. Moreover this universal interface can also be employed for the recovering of zones of interest (fraction collector) to perform further investigations with NMR or FTIR etc.

Further information is available on request from the authors.

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

[2] Aranda, M., Morlock, G., J. Chromatogr. A 1131 (2006) 253

[3] Aranda, M., Morlock, G., J. Chromatogr. Sci. 45 (2007) 251

[4] Jautz, U., Morlock, G., J. Chromatogr. A 1128 (2006) 244

[5] Van Berkel G., Tomkins B., Kertesz V., Anal Chem 79 (2007) 2778

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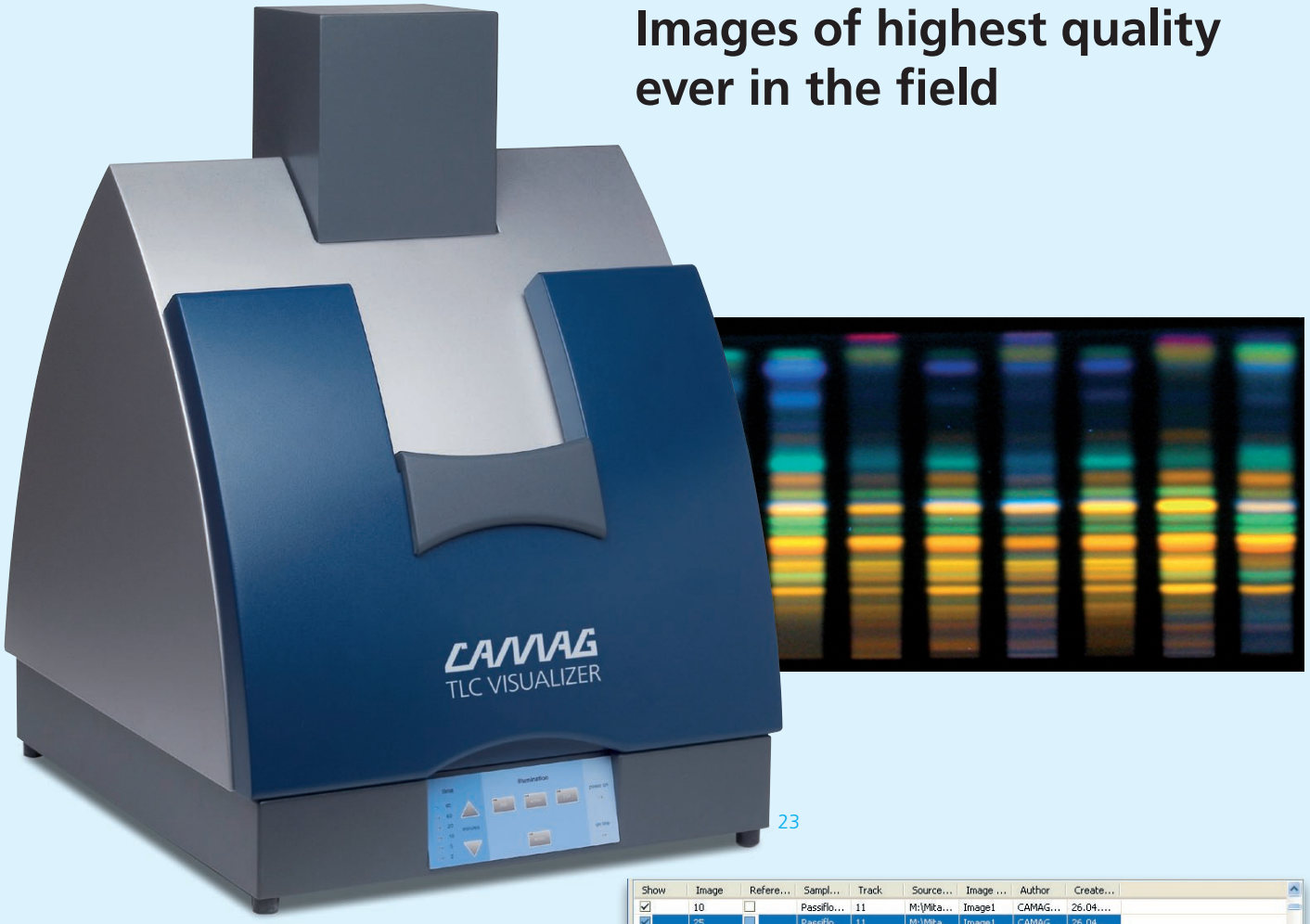

23

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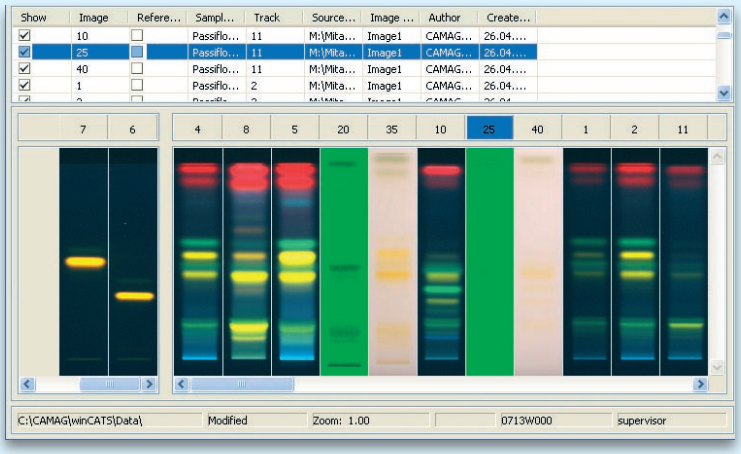

24

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