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Wirkungsbezogene Analytik von bestrahlten Sonnenschutzprodukten mittels HPTLC und in situ Detektion mit *Vibrio fischeri*

1

◀ *Dr. Urs Hauri, Vera Baumgartner, Dr. Christopher Hohl (von links nach rechts)*

Das Kantonale Laboratorium Basel-Stadt ist eine Behörde, die die Einhaltung der gesetzlichen Vorgaben für Lebensmittel und Bedarfsgegenstände wie Spielzeug oder Kosmetika überwacht. Die Abteilung Non-Food unter der Leitung von Dr. Christopher Hohl* ist auf die Analyse von Zusatzstoffen (z. B. Konservierungsstoffe), Färbemitteln und UV-Filtern in Gebrauchsgegenständen spezialisiert. Die Arbeit beinhaltet auch die Entwicklung neuer Analysemethoden zum Auffinden von Analyten mit toxikologischer Relevanz mittels GC, HPLC und HPTLC. Die Einführung von Toxizitätstests auf der HPTLC-Platte macht es seit kurzem möglich, beim Screening auf unbekannte Substanzen, eventuell toxikologisch relevante Peaks erkennen zu können.

Einleitung

Sonnenschutzmittel haben den Zweck, die menschliche Haut vor schädlicher UVA- und UVB-Strahlung zu schützen. In einigen Formulierungen ist jedoch ein Abbau der enthaltenen UV-Filter möglich, sobald sie dem Sonnenlicht ausgesetzt sind – dies wurde bereits 1997 gezeigt [1]. Die toxikologische Relevanz der dabei entstehenden Abbauprodukte wurde bisher noch nicht untersucht. Daher wurde bei der folgenden Methode die Chromatographie mit einer Bioaktivitäts-Detektion gekoppelt, was die Untersuchung der spezifischen Bioaktivität von Fotoabbauprodukten in Sonnenschutzprodukten möglich macht.

Die wirkungsbezogene Analytik mittels HPTLC-Biolumineszenz-Kopplung wurde ausgewählt, um mit Hilfe von *Vibrio fischeri* eine Verknüpfung von chemisch-physikalischen und biologischen Detektionsmethoden herzustellen. Eine Hemmung der Biolumineszenz wird durch eine Störung im Bakterienstoffwechsel verursacht, wobei der Grad der Hemmung mit der Toxizität des Stoffes korreliert. Seit 1970 wird das *Vibrio fischeri*-Bakterium für ökotoxikologische Tests eingesetzt, vor allem in der Wasseranalytik (Küvettentest, DIN 38412 L34), aber auch für Chemikalientests. Ein passendes Testkit zum Einsatz der Bakterien als Detektionsmethode für die HPTLC ist unter dem Namen Bioluminex erhältlich (www.chromadex.com).

Das Prinzip der Methode ist wie folgt: Die Proben wurden auf eine HPTLC-Platte aufgetragen, mittels automatisierter Mehrfachentwicklung (AMD) aufgetrennt, im UV bei 254 nm und 366 nm detektiert und anschliessend in eine *Vibrio fischeri*-Lösung getaucht. Zusätzlich wurden Zonen von vorrangigem Interesse, die mit der *Vibrio fischeri*-Detektion entdeckt wurden, erneut mit HPLC-DAD und LC-MS analysiert, um sie mit den bisherigen HPLC-Daten über UV-Filter-Fotoabbauprodukte vergleichen zu können.

Schicht

HPTLC-Platten LiChrospher Si 60 F_{254} (Merck), 20 x 10 cm, vorgewaschen durch Entwicklung mit Methanol, anschliessend auf dem DC-Plattenheizer 30 min bei 120 °C getrocknet.

Probenvorbereitung

Zur Herstellung von Fotoabbauprodukten wurden UV-Filter-Standards in adäquaten Lösungsmitteln gelöst und auf Objektträgern mit künstlichem Licht (Atlas Suntest CPS+) bestrahlt. Die Extraktion vom Objektträger erfolgte mit Ethanol/Aceton.

Die Sonnenschutzmittel-Proben wurden sowohl auf Objektträger ausgestrichen und mit künstlichem Licht oder mit Sonnenlicht bestrahlt, als auch auf die Haut aufgetragen und mit Sonnenlicht bestrahlt (30 min am frühen Nachmittag im Sommer, 47° nördliche Breite). Die Extraktion erfolgte sowohl von den Objektträgern als auch von der Haut mit Ethanol/Aceton.

Probenauftragung

Bandförmig mit dem Linomat 5, Bandlänge 6 mm, Abstand zum unteren Plattenrand 8 mm, Auftragevolumen 25 µL bei UV-Filter-Standards, 10 µL bei Sonnenschutzmittelproben (etwa 2 µg/Zone UV-Filter-Substanz auf der Platte)

Chromatographie

UV-Filter-Standards: AMD 2-System mit Diisopropylether – n-Hexan in 6 Schritten ohne Vorkonditionierung, Trocknungszeit 2–3 min, Endlaufhöhe 50 mm.

Sonnenschutzmittel-Proben: AMD 2-System mit t-Butylmethylether – n-Hexan in 7 Schritten mit Vorkonditionierung, Trocknungszeit 2–3 min, Endlaufhöhe 50 mm.

Die Platten wurden mindestens 30 min bei 120 °C auf dem DC-Plattenheizer nachgetrocknet.

Densitometrie

Multi-Wellenlängenscan von 200–400 nm mit TLC Scanner 3 und WinCATS Software.

Detektion

UV-Detektion bei 254 nm und 366 nm mit Digi-Store 2; anschliessend Biodetektion, wobei die Platte mittels Tauchgerät 1 s in die *Vibrio fischeri*-Lösung getaucht wurde; die Auswertung erfolgte mit dem BioLuminizer (Belichtungszeit 55 s).

Ergebnisse und Diskussion

Durch die *Vibrio fischeri*-Lösung ergaben sich einige Einschränkungen bei der Wahl des Schichtmaterials, des Fliessmittels und beim Trocknen der Platte. Nur die polaren Kieselgel- und LiChrospher-Schichten eigneten sich für das Tauchverfahren mit wässriger Bakterienlösung. Zur Entwicklung des Trennsystems konnten nur Fliessmittel verwendet werden, die auf *Vibrio fischeri* nicht toxisch wirkten und ohne Rückstände von der Plattenoberfläche abdampften. Die Methode ist zur Analyse von temperaturlabilen oder leicht flüchtigen Substanzen nicht geeignet, da die Platte lange und intensiv getrocknet werden muss, um die organischen Fliessmittel vollständig zu entfernen.

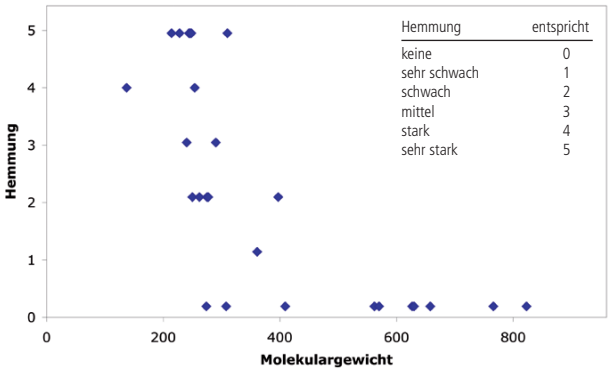
Das eigentliche Potential der Methode wurde jedoch sowohl bei der Untersuchung von reinen Standards als auch bei Kosmetika-Proben deutlich. Die Detektion von UV-Filter-Standardsubstanzen mit *Vibrio fischeri* war sehr gut möglich, bioaktive Substanzen erschienen als dunkle Zonen auf hellem, lumineszierenden Hintergrund. Die Hemmwirkung war stark von den einzelnen UV-Filtern abhängig, es waren alle Abstufungen zwischen sehr starker Lumineszenz-Unterdrückung und keinem Effekt möglich.

Der Vergleich von konventioneller Detektion mit der Biodetektion zeigte, dass die Empfindlichkeit meist unterschiedlich war: Einige Zonen, die im UV ein hohes Signal zeigten, konnten mit der Biodetektion gar nicht detektiert werden, während andere Zonen viel deutlicher hervortraten. In einigen Fällen war die Empfindlichkeit auch gleich.

Zwischen der Bioaktivität der UV-Filter und ihrer Molmasse (MW) bestand ein Zusammenhang. Alle neueren UV-Filter (nicht vor 1998 im Gebrauch) mit

einem MW von über 400 zeigten keinen hemmenden Effekt auf *Vibrio fischeri*.

Substanz	Abk.	Molekular-gewicht	Hemmung von *Vibrio fischeri*
4-Aminobenzoesäure	PABA	137	stark
Benzophenon-1	B-1	214	sehr stark
Benzophenon-3	B-3	228	sehr stark
3-Benzylidene-camphor	3-BC	240	mittel
Benzophenon-8	B-8	244	sehr stark
Benzophenon-2	B-2	246	sehr stark
Isoamylmethoxycinnamat	IMC	248	sehr stark
2-Ethylhexylsalicylat	EHS	250	schwach
4-Methylbenzyliden Camphor	MBC	254	stark
Homosalate	HMS	262	schwach
2-Phenyl-5-benzimidazolesulfonsäure	PBSA	274	keine
Menthylanthranilat	MA	275	schwach
2-Ethylhexyl-4-(dimethylamino)-benzoat	EHDP	277	schwach
Ethylhexylmethoxycinnamat	EHMC	290	mittel
2-Hydroxy-4-methoxybenzophenon-5-sulfonsäure	B-4/5	308	keine
tert. Butylmethoxydibenzoylmethan	BMDBM	310	sehr stark
Octocrylen	OC	361	sehr schwach
Diethylamino hydroxybenzoyl hexylbenzoat	DHHB	397	schwach
4-(2-oxo 3-bornylidenemethyl) phenyl trimethylammonium methyl sulfat	CBMS	409	keine
Terephtalydene dicamphor sulfonsäure	TDSA	562	keine
Drometrizole Trisiloxan	DTS	570	keine
Bis ethylhexyloxyphenol methoxyphenyl triazin	BEMT	627	keine
Disodium Phenyl Dibenzimidazole Tetrasulfonat	DPDT	630	keine
Methylene bis-benzotriazolyl tetramethylbutylphenol	MBBT	658	keine
Diethylhexylbutamidotriazon	DEBT	766	keine
Octyltriazon	EHT	823	keine



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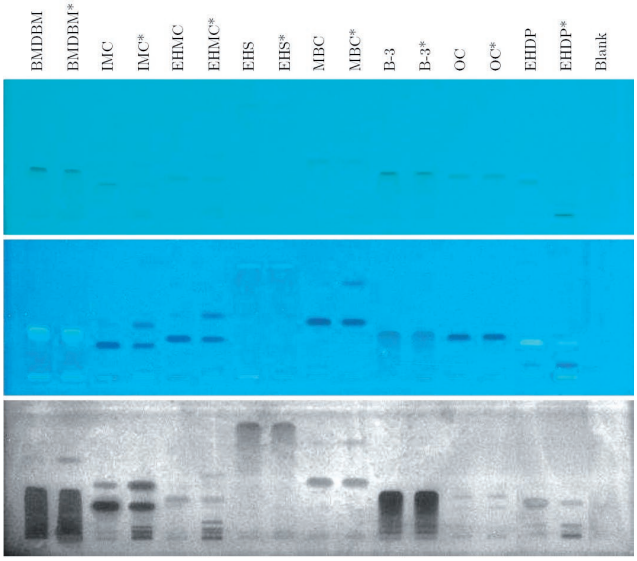
Der Vergleich von bestrahlten UV-Filter-Standards mit nicht bestrahlten Standards war ebenfalls sehr aufschlussreich. Fotoabbau wurde bei Ethylhexylmethoxycinnamat (EHMC), Isoamylmethoxycinnamat (IMC) und Ethylhexyldimethylaminobenzoat (EHDP) beobachtet. Alle Fotoabbauprodukte wirkten stärker hemmend als die UV-Filter selbst. Dabei muss jedoch berücksichtigt werden, dass der Fotoabbau eines UV-Filters stark von der Matrix und der Kombination der UV-Filter abhängig ist.

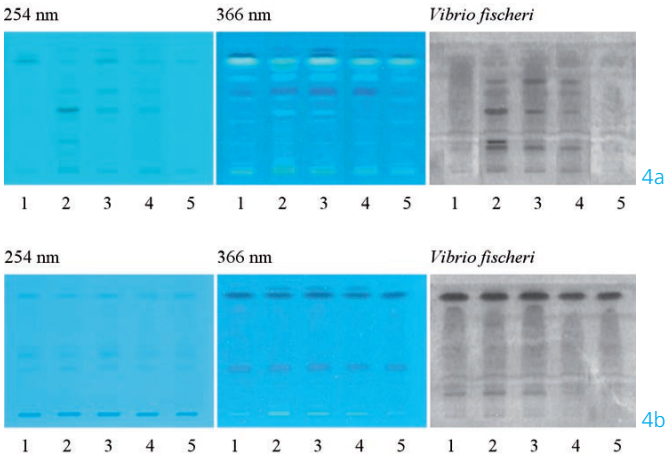


3

▲ *Detektion einiger unbestrahlter und bestrahlter (markiert mit *) UV-Filter-Standards bei 254 nm, 366 nm und mit Vibrio fischeri (von oben nach unten)*

Daher wurden die weiteren Tests mit fünf kommerziell erhältlichen Sonnenschutzmitteln durchgeführt. Die bestrahlten und unbestrahlten Sonnenschutzmittel-Extrakte wurden sowohl mit HPTLC als auch mit HPLC-DAD und LC-MS analysiert. Die meisten Sonnenschutzmittel enthielten Matrixkomponenten (z. B. Konservierungsstoffe), die die Lumineszenz ebenfalls hemmten. Zwei der Sonnenschutzmittel zeigten einen sehr starken Abbau, der zwar im UV sichtbar war, aber mit *Vibrio fischeri* wesentlich deutlicher wurde. Die Zonen, die erst nach der Bestrahlung auftauchten, wurden detaillierter untersucht, da angenommen wurde, dass es sich hierbei um Fotoabbauprodukte handelte. Die folgende Sonnenschutzmittel-Probe, bestehend aus Ethylhexylmethoxycinnamat, t-Butylmethoxydibenzoylmethan und 2-Ethylhexyl-4-(dimethylamino)-benzoat, zeigt einen starken Abbau, während die aus t-Bu-

tylmethoxydibenzoylmethan, 4-Methylbenzylidencamphor, 2-Phenyl-5-benzimidazolsulfonsäure und Octyltriazon bestehende Probe eine stabile Formulierung aufweist.



▲ *UV-Detektion versus Vibrio fischeri-Detektion von zwei Sonnenschutzmittelproben (Objektträger: Bahn 1: unbestrahlt, Bahn 2: künstliches Licht, Bahn 3: Sonnenlicht; Haut: Bahn 4: Sonnenlicht, Bahn 5: unbestrahlt): Probe mit starkem Fotoabbau (oben) und Probe mit stabiler Formulierung (unten)*

Um HPTLC- und HPLC-Methoden zu vergleichen und die Abbauprodukte zu identifizieren, wurden die Sonnenschutzmittel-Extrakte auf einer HPTLC-Platte aufgetrennt, die betreffenden Zonen ausgekratzt und erneut mit HPLC-DAD und LC-MS analysiert. Dieses Verfahren zur Identifikation gelang sehr gut, auch wenn die Trennleistung der HPTLC in diesem Fall insofern der HPLC unterlegen war, als eine Zone mit hoher Bioaktivität auf der HPTLC-Platte mehrere Peaks in der HPLC ergeben konnte. Die Peakhöhen der Substanzen aus der Biolumineszenz-Detektion korrespondierten nicht mit denen der konventionellen Detektoren (HPTLC-UV, HPLC-DAD, LC-MS). In einem Fall wäre eine stark bioaktive Substanz mit den verwendeten physikalisch-chemischen Detektoren unentdeckt geblieben.

Die Detektion mit *Vibrio fischeri* kann für zwei verschiedene Zwecke eingesetzt werden: Erstens ermöglicht die charakteristische Selektivität dieser Detektionsmethode das Auffinden von vorher nicht detektierten Substanzen. Zweitens ist es mit *Vibrio fischeri* als Bioaktivitätsindikator möglich, über die Hemmung die Fotoabbauprodukte herauszugreifen, die einer näheren toxikologischen Untersuchung bedürfen.

Die komplette Arbeit kann unter dem Titel »Bio-activity based Analysis of irradiated Sunscreens using HPTLC and in situ Detection with *Vibrio fischeri*« heruntergeladen werden von: http://www.kantonslabor-bs.ch > Infos (Information) > Themen-papiere (Opinions)

oder via 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.

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Produktkontrolle: Bromierung und Oxidation des Alkaloids Deoxypeganin

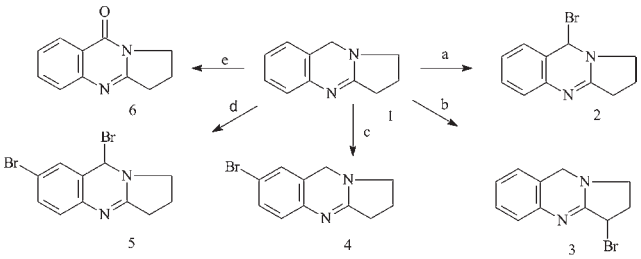
5

▲ *Dr. N. Mukarramov, Prof. Dr. Kh. Shakhidoyatov (links nach rechts)*

Im Institut für Chemie pflanzlicher Substanzen, geleitet von Professor Shakhidoyatov*, laufen in der Abteilung Organische Synthese seit vielen Jahren Arbeiten zur Synthese und chemischen Modifizierung von Naturstoffen einschliesslich der Alkaloide. Die Strukturaufklärung erfolgt mittels IR-Spektroskopie, H^1-/C^{13}-NMR-Spektroskopie, Massenspektrometrie und Röntgenfluoreszenzanalyse. Die Planar-Chromatographie wird zur Prozesskontrolle, Identifizierung und Reinheitsprüfung eingesetzt.

Einführung

Das Alkaloid Deoxypeganin (DOP) wird aus der Pflanze *Peganum harmala* [1] isoliert und als Anticholin-Esterase-Zubereitung bei der medizinischen Behandlung eingesetzt [2]. Für die Synthese oder chemische Modifizierung von Naturstoffen ist es notwendig, den stufenweisen Reaktionsverlauf und die möglichen Reaktionsrichtungen zu überwachen. Es ist wichtig, die Strukturen der Edukte, der Zwischenprodukte und der Endprodukte zu kennen. Bei der DOP (1)-Bromierung können folgende Reaktionen ablaufen:



Richtung (a) bevorzugt die Bromierung in Position 4, wodurch 4-Bromdeoxypeganin (2) entsteht. Richtung (b) führt zu α-Bromdeoxypeganin (3). Zusätzlich erfolgt die Bromierung auch am Phenylring in Position 6 (c) unter Bildung von 6-Bromdeoxypeganin (4). Die zweifache Bromierung (d) bildet 4,6-Dibromdeoxypeganin (5). Ein anderer Reaktionstyp in Gegenwart von N-Bromsuccinimid (NBS) ist die Oxidation (e), die zum Alkaloid Deoxyvasicinon (DOV, 6) und 6-Bromdeoxyvasicinon (7) führt.

Zur Reaktionskontrolle wird die Planar-Chromatographie aufgrund ihrer vielfältigen Vorteile genutzt:

- **Analysen unterschiedlicher Reaktionsansätze können zeitgleich durchgeführt werden.**
- **Die Analytik benötigt nur 10 min und erfordert keine spezielle Probenvorbereitung.**
- **Die HPTLC ist eine kostengünstige Technik und benötigt nicht viel Fliessmittel, i. e. nur 5 mL pro Platte.**
- **20 Proben können parallel analysiert werden.**
- **Die HPTLC mit nachfolgender Spektrenidentifikation erlaubt die Trennung komplexer Substanzgemische zur Identitätsprüfung und Gehaltsbestimmung.**

Probenvorbereitung

Ca. 1 mg Substanz, exakt eingewogen, wurde in 1 mL Methanol gelöst.

Schicht

HPTLC-Platten Kieselgel 60 F_{254} (Merck, Germany), alternativ DC-Folien AL Sil G/UV (Whatman, UK) oder HPTLC-Plattten AF-UV (Sorbfil, Russische Föderation), alle 20 × 10 cm

Probenauftragung

Strichförmig mit dem Linomat 5, max. 20 Bahnen, Bandlänge 3 mm, Auftragevolumen je 2 und 4 µL der Probelösung, Bahnabstand 7 mm, linker Randabstand 15 mm, unterer Randabstand 8 mm

Chromatographie

In der Doppeltrogkammer 20 × 10 cm mit Chloroform – Methanol – Aceton – Cyclohexan 5:1:5:5

(Merck-/Whatman-Schichten) bzw. 4:1:4:4 (Sorbfil-Platte). Die Laufstrecke betrug 60 mm vom unteren Plattenrand. Nach der Entwicklung wurde die Platte für 10 min im Kaltluftstrom getrocknet.

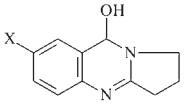
Densitometrie

Absorptionsmessung bei 254 nm mit TLC-Scanner 3 und winCATS-Software

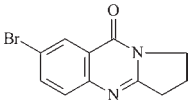
Anmerkung: Es wird empfohlen, die Messung bei der jeweils optimalen Wellenlänge jeder Substanz durchzuführen, optional mit dem Mehrwellenlängenscan, um die beste Detektierbarkeit zu gewährleisten.

Ergebnisse und Diskussion

Die Bromierung von DOP (1) mit NBS im Verhältnis 1:1 in Chloroform führte zu den Verbindungen 2 und 5, die im alkalischen Milieu die entsprechenden Hydroxyderivate (8) und (9) bilden. Die Verbindung 8 ist identisch mit dem natürlichen Alkaloid Peganol (4-Hydroxydeoxypeganin), das bereits aus *Peganum harmala* isoliert wurde [4]. Die Bromierung im Verhältnis 1:4 mit anschliessender Kalilaugen-Behandlung (5 % KOH) führt zur Bildung von 6-Brompeganol (9) und Nebenprodukt 6-Bromdeoxyvasicinon (7).



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



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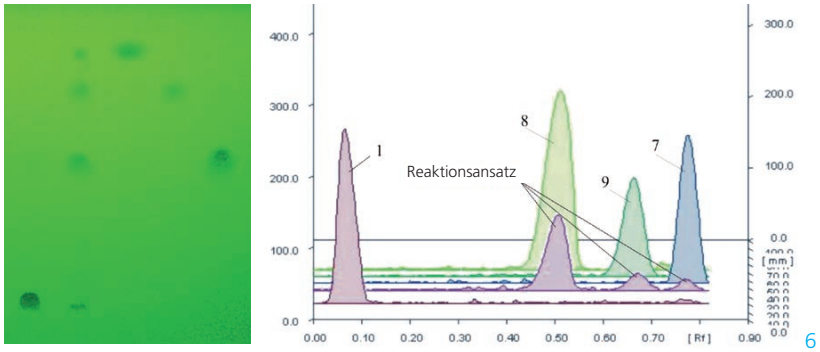
Anders als in Chloroform reagiert das in Wasser gelöste DOP-Hydrochlorid mit NBS (Verhältnis 1:1) zu 6-Bromdeoxypeganin (4). Ein Verhältnis von 1:4 verschiebt die Reaktion zugunsten der Bildung von 6-Bromdeoxyvasicinon (7). Die Oxidation von DOP*HCl mit Kaliumpermanganat ergibt Peganol (8) und daneben unverändertes DOV (6).

Offensichtlich führen die Bromierung von DOP in Chloroform und DOP*HCl in Wasser zu unterschiedlichen Reaktionsprodukten. Da die Isolierung der gebildeten Produkte und deren gravimetrische Bestimmung aufwendig sind, wurde die quantitative HPTLC verwendet.



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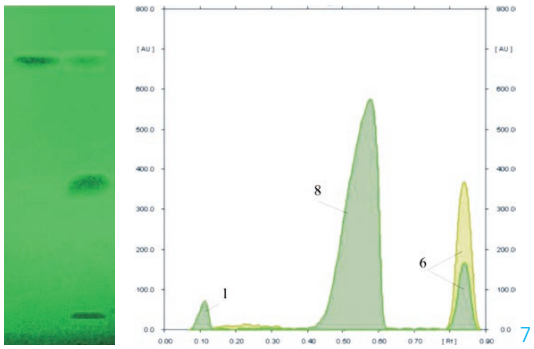
▲ *Chromatogramm des DOP-Produktes in Chloroform; Bahn 1: DOP, Bahn 2: Reaktionsprodukt von 1 mit Brom und Kalilauge, Bahn 3: 6-Bromdeoxyvasicinon (7), Bahn 4: 6-Brompeganol (9), Bahn 5: Peganol (8)*



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▲ *Chromatogramm des DOP*HCl-Oxidationsproduktes mit $KMnO_4$; Bahn 1: Deoxyvasicinon (6), Bahn 2: Reaktionsprodukt, enthält Peganol (8) neben nicht umgesetztem DOV (6)*

Die beste Selektivität für die Trennung von Peganol (8) und 6-Brompeganol (9) wurde auf den Merck-Platten erhalten.

hR_F-Werte im Reaktionsansatz			
Platten	Merck	Whatman	Sorbfil
Substanzen			
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

Weitere Informationen erhalten Sie auf Anfrage von den Autoren.

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CAMAG unter neuer Leitung


8

Herr Rolf Rolli trat am 1. September 2007 als neuer Vorsitzender der Geschäftsleitung (CEO) bei CAMAG ein. Er bringt reiche Erfahrung als Leiter vergleichbarer Unternehmen ein. Unter anderem war er tätig in der Forschung & Entwicklung bei Sandoz in Basel und in USA und Leiter Entwicklung bei Knoll Pharma. Seit 1992 leitete er verschiedene Life Sciences Unternehmen wie Life Technologies (Invitrogen)/Schweiz, SOTAX, Zymark Schweiz & Deutschland. Vor seinem Eintritt bei CAMAG war er Geschäftsführer von Merck Biosciences, einer Schweizer Tochtergesellschaft von Merck Darmstadt. In allen seinen Tätigkeiten hatte Herr Rolli engen Bezug zu Kunden und Distributoren in vielen Ländern. Er organisierte und leitete Workshops im In- und Ausland und nahm mit zahlreichen Vorträgen an internationalen Symposien teil.

Seit seinem Eintritt weht ein frischer Wind in der CAMAG und auch unsere internationalen Distributoren zeigen sich erfreut über die von ihm ausgehende tatkräftige Unterstützung. In den wenigen Monaten seiner Tätigkeit bei CAMAG unternahm er bereits ausgedehnte Auslandsreisen, u.a. nach USA, Indien und Mittleren Osten. Auf seine Initiative begann CAMAG mit der Erschliessung der Kosmetik-Industrie als neues, lukratives Anwendungsgebietes für die Planar-Chromatographie, was sich auch in Beiträgen dieses CBS widerspiegelt.

Erwarten Sie von Herrn Rolli's Tätigkeit als CEO einen Sprung nach vorn, für CAMAG und für die Planar-Chromatographie!

(gez. Dieter Jänchen)

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- Publikationsorgan und Stelle
- Kurze Inhaltsangabe für den DC/HPTLC-relevanten Teil mit Hinweisen auf benutzte Trennsysteme, Detektionsverfahren, Auswertung, Ergebnisse, etc.
- Key Words

So kann der DC/HPTLC-Anwender bei einer neuen Fragestellung sich vorab ein Bild über die Lösbarkeit der Aufgabe machen und durch Transfer- oder Analogieschlüsse diese effizienter angehen.

Eine anschliessende Studie der als relevant erkannten Literatur ersetzt der CBS allerdings nicht.

Seit 1997 ist die umfangreichste Literatursammlung im Bereich Planar-Chromatographie (DC/HPTLC) als Datenbank verfügbar. Die Datenbank enthält alle auf Englisch erschienenen Literaturzitate ab CBS 51 (Mai 1983) und wird laufend erweitert – zurzeit können mehr als 8000 Einträge durchsucht werden.

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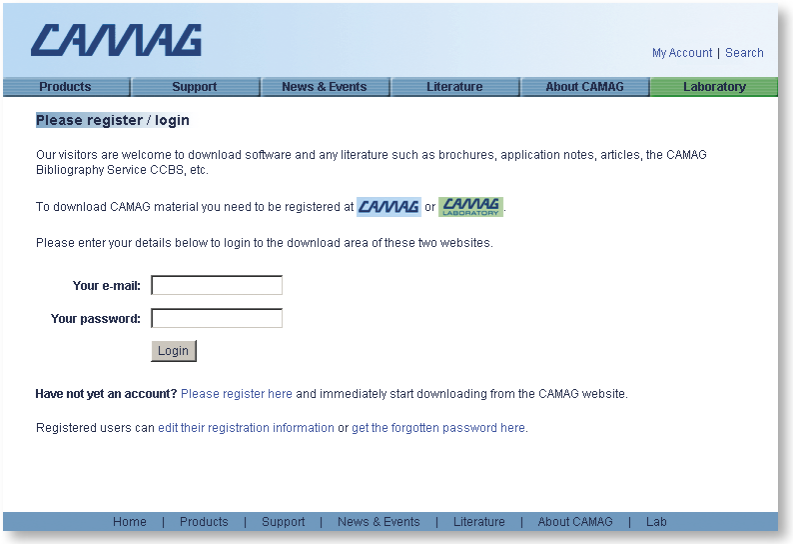

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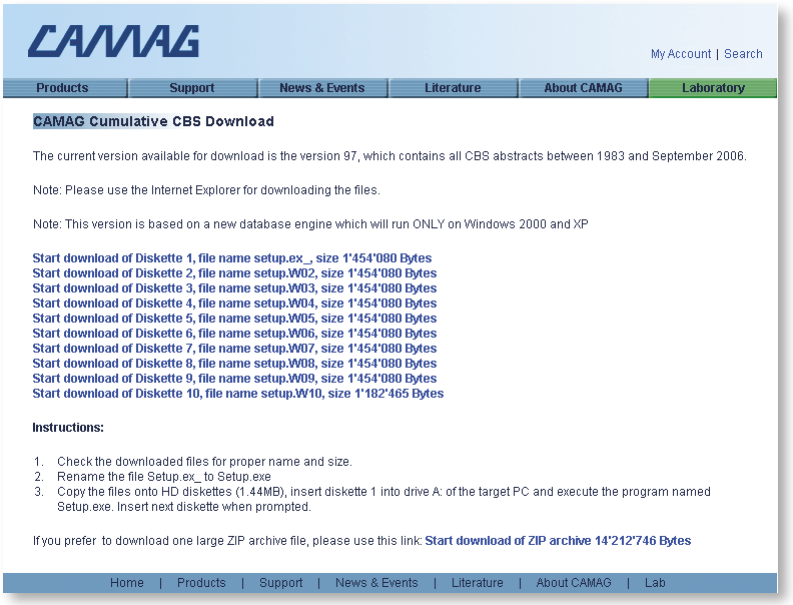

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Benutzung der CCBS-Datenbank

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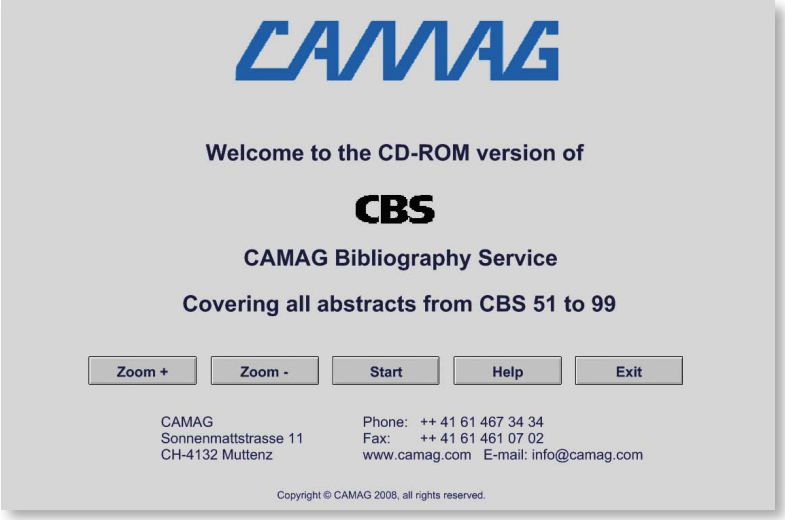
Zum Beispiel lieferte die Suche nach »Ciprofloxacin« 14 Einträge, wovon der 11. Eintrag gerade angezeigt wird. Alle Einträge, in denen das Suchwort gefunden wurde, erscheinen auf dem Bildschirm und können durchgeblättert werden. Die Ansicht zeigt unten links die Gesamtzahl der gefunden Einträge (14), ebenso wie die Nummer des Eintrags, der gerade betrachtet wird (11). Mit den Pfeiltasten kann durch die Suchresultate vor- und zurückgeblättert werden. Ungeeignete Einträge können mit DISCARD ABSTRACT aus den Suchergebnissen entfernt werden. Mit SEARCH WITHIN RESULTS kann die Suche innerhalb der gefundenen Abstracts fortgesetzt und eingegrenzt werden. Die ausgewählten Einträge können mit PRINT SELECTION gedruckt werden. Für eine neue Suche wählen Sie NEW SEARCH. Unter HELP finden Sie weitere Informationen zur Suchfunktion. Mit HOME kommen Sie zurück auf die Startseite und mit EXIT verlassen Sie die Datenbank.



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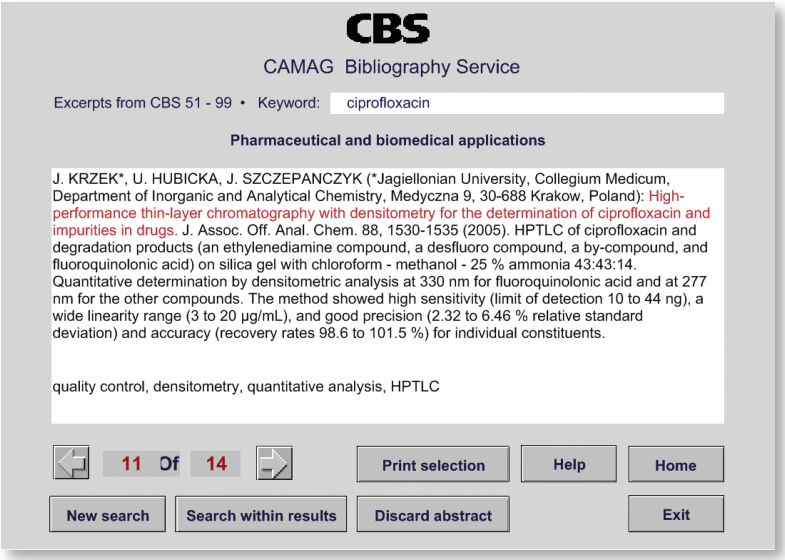

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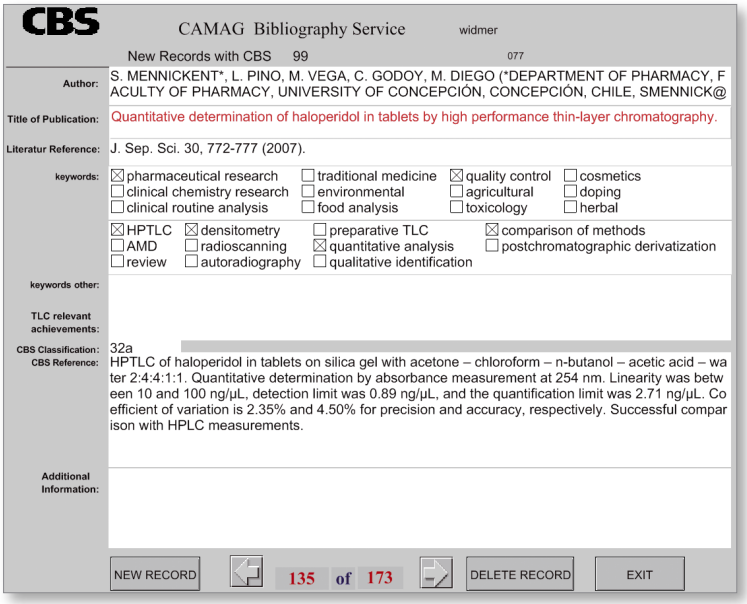
Die Entstehung der Literaturberichte (»gelbe Seiten« des CBS) einst und heute

Der CBS wurde 1965 von Dr. Dieter Jänchen auf Anregung einiger Geschäftsfreunde ins Leben gerufen, die dann auch als die ersten CBS-Referenten dem Herausgeber Berichte über die von ihnen gelesenen dünnschichtchromatographisch relevanten Publikationen lieferten. Um die eingehenden Berichte hinsichtlich ihres Informationsgehalts möglichst gleichwertig zu halten, wurde ein Berichtsformular erstellt, in dessen Rubriken die Referenten – teils handschriftlich (!), teils mit Schreibmaschine – ihre Mitteilungen eintrugen. Aus diesen Berichten verfasste der Herausgeber dann jedes einzelne CBS-Referat. Das war die gängige Praxis bis Ende der 80er Jahre.

Der Kreis der Referenten hatte sich im Laufe der Jahre verändert, und auch das Berichtsformular wurde mehr und mehr verfeinert, so dass der Herausgeber durch geringfügige Umstellungen, Streichungen etc. aus dem vom Referenten vorgeschlagenen Text das CBS-Referat erstellen konnte. In den 90er Jahren hatte sich die Qualität der Berichte so verbessert, dass das Verfassen der Referate an eine fachkundige CAMAG-Mitarbeiterin delegiert werden konnte und der Herausgeber nur noch Korrekturen vornehmen musste.


CBS CAMAG Bibliography Service widmer

New Records with CBS 99 077

Author: S. MENNICKENT*, L. PINO, M. VEGA, C. GODOY, M. DIEGO (*DEPARTMENT OF PHARMACY, FACULTY OF PHARMACY, UNIVERSITY OF CONCEPCIÓN, CONCEPCIÓN, CHILE, SMENNICK@

Title of Publication: Quantitative determination of haloperidol in tablets by high performance thin-layer chromatography.

Literatur Reference: J. Sep. Sci. 30, 772-777 (2007).

keywords: ☒ pharmaceutical research ☐ traditional medicine ☒ quality control ☐ cosmetics ☐ clinical chemistry research ☐ environmental ☐ agricultural ☐ doping ☐ clinical routine analysis ☐ food analysis ☐ toxicology ☐ herbal

☒ HPTLC ☒ densitometry ☐ preparative TLC ☒ comparison of methods ☐ AMD ☐ radioscanning ☒ quantitative analysis ☐ postchromatographic derivatization ☐ review ☐ autoradiography ☐ qualitative identification

keywords other:

TLC relevant achievements:

CBS Classification: 32a

CBS Reference: HPTLC of haloperidol in tablets on silica gel with acetone – chloroform – n-butanol – acetic acid – water 2:4:4:1:1. Quantitative determination by absorbance measurement at 254 nm. Linearity was between 10 and 100 ng/µL, detection limit was 0.89 ng/µL, and the quantification limit was 2.71 ng/µL. Coefficient of variation is 2.35% and 4.50% for precision and accuracy, respectively. Successful comparison with HPLC measurements.

Additional Information:

NEW RECORD 135 of 173 DELETE RECORD EXIT

▲ *Maske mit Beispiel für eine Referat-Übermittlung*

2003 übernahm Frau Dr. Gerda Morlock die Herausgeberschaft des CBS und führte die elektronische Berichterstattung durch die Referenten ein. Im jetzigen System (seit Januar 2005) werden die Referate von den Referenten direkt in eine einfache Datenbank eingetragen, die auf der bisher verwendeten Vorlage (Word-Datei) basiert, wodurch Aufbau und Inhaltsauswahl der Referate vorgegeben werden. Die Dateien werden von den Referenten per Email an CAMAG übermittelt, wo sie von Frau Valeria Widmer gesammelt und mit meist kleineren Korrekturen versehen werden, um die Qualität und Einheitlichkeit der Referate zu gewährleisten. Dabei wird auch kontrolliert, ob alle nötigen Angaben zur Methode enthalten sind und gegebenenfalls nachgefragt. Die für die gedruckten gelben Seiten der nächsten CBS-Ausgabe zusammengestellten Referate werden anschliessend von Frau Dr. Morlock überprüft. Gleichzeitig mit der Übermittlung an die Druckerei werden sie elektronisch zur bestehenden CCBS-Datenbank hinzugefügt. Somit wird diese zweimal jährlich aktualisiert und kann über die CAMAG-Webseite abgerufen werden.

Die Resonanz aus dem Kundenkreis beweist uns die Beliebtheit dieser weltweit einmaligen DC/HPTLC-Datenbank, mit der schnell und bequem am Arbeitsplatz über Eingabe eines Suchstichworts recherchiert werden kann.

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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

Gerda Morlock
cbs@camag.com

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

Gerda Morlock
cbs@camag.com

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

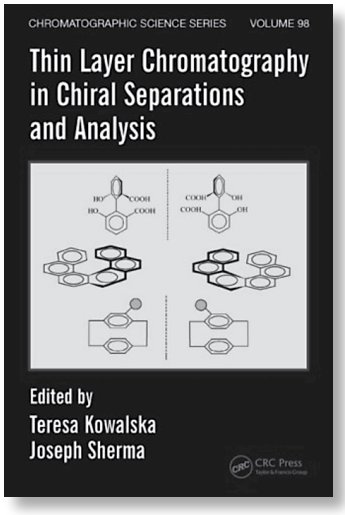
100 185 R. ZHANG* (Zhang Rong), Y. YUE (Yue Yongde), R. HUA (Hua Rimao), W. YAN (Yan Wen) (*Recourses and Environment College of Anhui Agricultural University, Agri-food Security Key Lab of Anhui Province, No. 130, Changjiang West Road, Hefei, China; z_rong163@163.com): Factors affecting the separation 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.

CRC Press/Taylor & Francis Group: Boca Raton, London, New York 2007

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420 pages $169.95

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
www.hptlc.com

info@hptlc.com

Augenmerk auf die Trocknung der Schicht!

9

▲ *Dr. Matthias Loppacher, Leiter Forschung & Entwicklung*

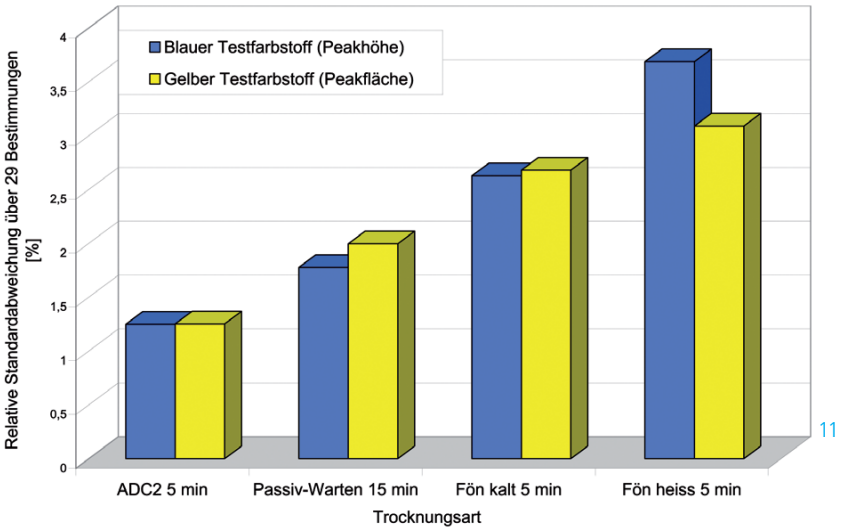
Die automatische Entwicklungskammer ADC 2 wurde auf optimale Reproduzierbarkeit des gesamten Entwicklungsprozesses hin entwickelt. Dies beinhaltet auch die Kontrolle externer Faktoren, wie die relative Feuchte und Trocknung der Schicht. Das neu in die ADC2 integrierte, geschlossene Kreislaufsystem beschleunigt den Luftstrom, richtet ihn auf die Oberfläche der DC/HPTLC-Schicht und ermöglicht damit eine kurze und gleichmäßige Konditionierung der Schicht.

10

▲ *Die ADC 2 ermöglicht die automatische Entwicklung und eine reproduzierbare Chromatographie, die durch das geschlossene Kreislaufsystem unterstützt wird.*

Veröffentlichungen haben gezeigt, dass auch der Trocknungsschritt signifikant zur verbesserten Reproduzierbarkeit des DC/HPTLC-Gesamtsystems beiträgt. Wenn das oben erwähnte, geschlossene Kreislaufsystem offen betrieben wird, kann es nach der Chromatographie hervorragend zur Schnelltrocknung der Schicht ohne jegliche Hitzeeinwirkung eingesetzt werden. Die Diffusionsvorgänge werden dabei direkt nach der Chromatographie mehr und mehr gestoppt, so dass Peakverbreiterungen minimiert sind.

Systematische Studien während der Entwicklung des neuen Trocknungssystems haben gezeigt, dass die Präzision bei verschiedenen Trocknungsarten unterschiedlich ist. Die Trocknung in der ADC2 zeigte im Vergleich zur manuellen Trocknung mit dem Fön eine bis zum Faktor 3 verbesserte Methoden-Präzision. Die ungleiche Trocknung mit dem Fön oder die erhöhte Diffusion durch das lange Warten beim passiven Trocknen verursachen beide schlechtere Reproduzierbarkeiten.



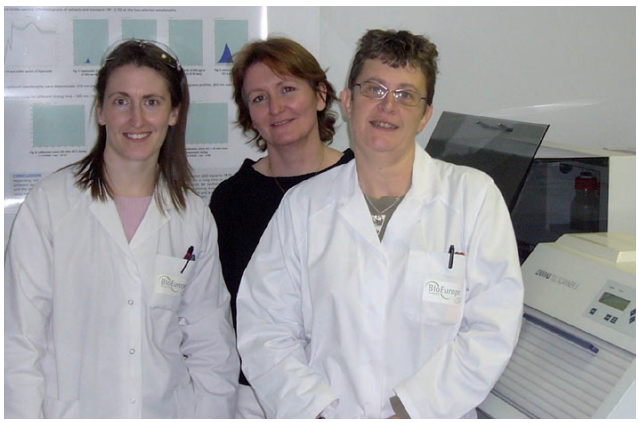
11

▲ *Vergleich der verschiedenen Trocknungsarten, wobei der Trocknungsschritt mittels ADC2 die beste Reproduzierbarkeit zeigte.*

Eine weitere Leistungsstärke der ADC2 ist die reproduzierbare Chromatographie. Je nach Aktivität der Schicht wird Wasser aus dem darüber ziehenden Luftstrom von definierter Luftfeuchte sorbiert oder in diesen desorbiert, bis das Gleichgewicht erreicht ist. Durch das Kreislaufsystem wird der Luftstrom dabei immer wieder durch die Konditioniereinheit geleitet und erneut im Feuchtigkeitsgehalt aufbereitet. Dieses geschlossene Kreislaufsystem ermöglichte eine kompakte, effektive Bauweise, schützt vor Kontamination und kann leicht sauber gehalten werden.

In die ADC2 wurde bewusst die Doppeltrogkammer implementiert, so dass konventionelle Doppeltrog-Methoden direkt übertragbar sind. Manuelle Schritte, die die Reproduzierbarkeit erschweren können, sind nun automatisiert, und äußere Einflussfaktoren haben an Bedeutung verloren.

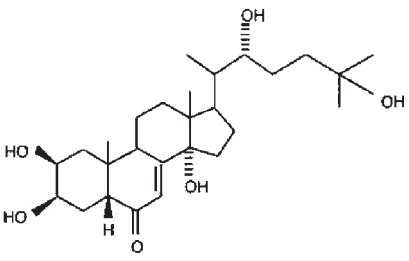
Quantifizierung von ß-Ecdyson in brasilianischem Ginsengsaft *(Pfaffia glomerata)*



▲ *BioEurope Forschungszentrum in Anet (80 km von Paris entfernt): Agnès Gaspar, Sophie Leclere*, Véronique Marignier (links nach rechts)* 12

Das 1985 gegründete KMU BioEurope fusionierte 1992 mit dem französischen Unternehmen Solabia. Die Produktion und der Vertrieb von Zwischenprodukten für pharmazeutische, kosmetische und diagnostische Bereiche und Fermentationszwecke sind Schwerpunkte des Unternehmens, das bei der Pepton-Produktion für die Fermentierung europaweit führend ist. Basierend auf seinem ausgewiesenen Fachwissen auf dem Gebiet der Enzym-katalysierten Syntheseprozesse, entwickelt BioEurope auch unterschiedliche Pflanzenextrakte für kosmetische Zwecke.

BioEurope setzt die HPTLC seit 2003 ein und wendet inzwischen über 15 validierte, quantitative Methoden an, wie die Quantifizierung von Coffein und Theobromin in Kakaoextrakten, Asiatin- und Madecassin-Säure in Centella asiatica-Extrakten und Hyperosid in Walnussblättern. Die HPTLC ist – durch die gezielte Quantifizierung von aktiven Inhaltsstoffen in den Pflanzen – hilfreich für die Optimierung der Extraktionsprozesse und die Charakterisierung des Rohmaterials und der Extrakte.



◀ *Strukturformel von ß-Ecdyson*

Einleitung

Pfaffia glomerata wird als brasilianische Ginseng aufgrund ihrer Ernährungsfunktion und prophylaktischen Eigenschaften bezeichnet, die denen der sogenannten traditionellen Ginseng sehr ähnlich sind. Dennoch unterscheidet sich die brasilianische Ginseng von *Panax ginseng* sehr. Sie wächst an südlichen, feuchten Berghängen mitten im brasilianischen Wald, gehört zur Familie der Amaranthaceae und besitzt eine große, knollige Wurzel, aus der durch Kaltpressverfahren ein Saft gewonnen wird. Die Pflanze verdankt ihre Qualität folgenden Inhaltsstoffen:

- Antioxidanzien, wie Selen und Polyphenole
- Saponine (Nortriterpenoide und Pfaffoside)
- Aminosäuren: Arginin, Lysin, Histidin und Glycin
- Steroide, wie zum Beispiel ß-Ecdyson [1]

ß-Ecdyson ist ein Steroidhormon, das für das Häuten von Insekten und Schalentieren und auch von bestimmten Pflanzen verantwortlich ist. Es hat besondere biologische Eigenschaften bezüglich des Hautzellenstoffwechsels und besitzt daher Potential für kosmetische Anwendungen [2]. Die folgende, auf [3] basierende Methode beschreibt die Quantifizierung des ß-Ecdysons in kaltgepressten *Pfaffia glomerata*-Rhizomextrakten.

Probenvorbereitung

1 g Extrakt werden mit 10 mL Methanol 5 min in einem Ultraschallbad gelöst.

Standardlösung

ß-Ecdyson wird in Methanol gelöst (0.04 g/L).

Schicht

HPTLC-Platten Kieselgel 60 F_{254} (Merck), 20 x 10 cm

Probenauftragung

Strichförmig mit dem DC-Probenautomat 4, 16 Bahnen, Bandlänge 6 mm, Bahnabstand 11.3 mm, linker Randabstand 15 mm, unterer Randabstand 8 mm, Auftragevolumen 5 und 3 µL für Proben und 1 bis 5 µL für Standardlösungen (0.04 bis 0.2 µg/Band).

Chromatographie

In der Doppeltrogkammer 20 x 10 cm nach 15 min Kammersättigung mit der unteren Phase von Chloroform – Methanol – Wasser 7:5:2. Die Laufstrecke beträgt 50 mm (vom unteren Plattenrand). Danach wird die Platte bei 80 °C 5 min auf dem DC-Plattenheizer getrocknet.

Postchromatographische Derivatisierung

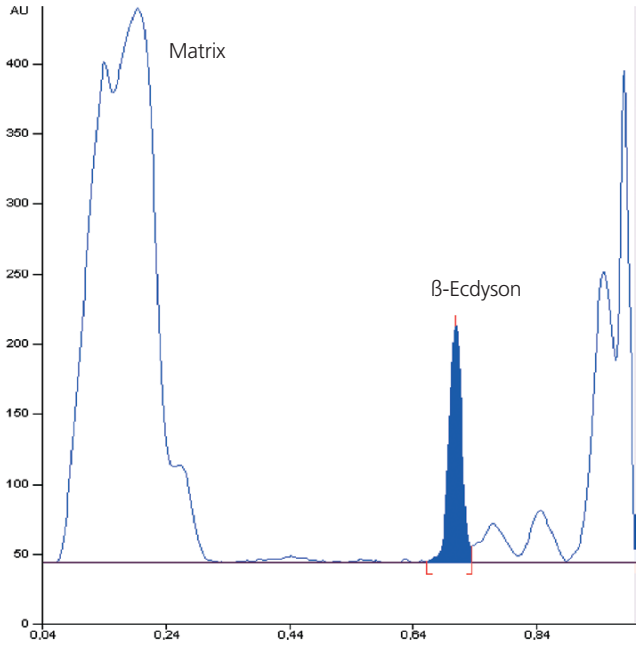
Mit der Chromatogramm-Tauchvorrichtung III wird die Platte für 1 s in das Anisaldehyd-Reagenz (0.5 mL Anisaldehyd wird mit 10 mL Eisessig, 85 mL Methanol und 5 mL konzentrierter Schwefelsäure versetzt) getaucht und anschließend auf dem DC-Plattenheizer bei 120 °C 20 min erhitzt.

Densitometrie

Absorptionsmessung bei 432 nm mit TLC-Scanner 3 und winCATS-Software, polynome Kalibration über die Peakfläche.

Ergebnisse und Diskussion

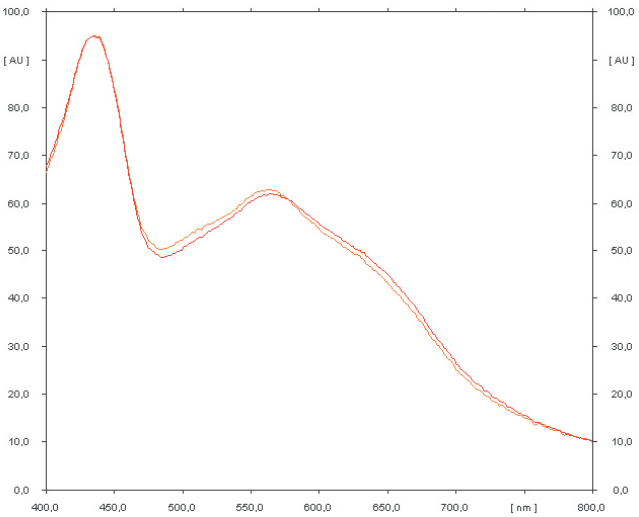
In Anbetracht der komplexen Matrix war die Selektivität der Bestimmung von ß-Ecdyson sehr zufriedenstellend. Ein repräsentatives Densitogramm der Probe zeigt eine deutliche Trennung des ß-Ecdysons von anderen Probenbestandteilen. Bei der vorherigen HPLC-Methode wurde die Reproduzierbarkeit durch die Matrix beeinträchtigt, die über das Chromatogramm verteilt vorlag und nicht so deutlich im Startbereich retardiert wurde wie bei der orthogonalen, vom Trennprinzip umgekehrten HPTLC-Methode.



13

▲ *Trennung von ß-Ecdyson (hR_F 71) und Matrixbestandteilen im kaltgepressten Pfaffia glomerata Rhizomextrakt*

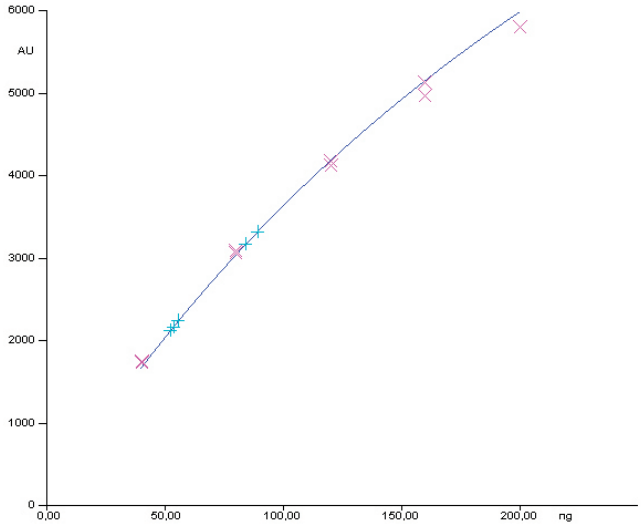
Für die densitometrische Auswertung wurde die optimale Wellenlänge von 432 nm durch die Aufnahme von in situ UV/Vis-Spektren bestätigt. Die Identität von ß-Ecdyson wurde durch den Spektrenvergleich der Probezone mit der Standardzone belegt.



14

▲ *Spektrenvergleich von Probenzone und ß-Ecdyson-Standardzone*

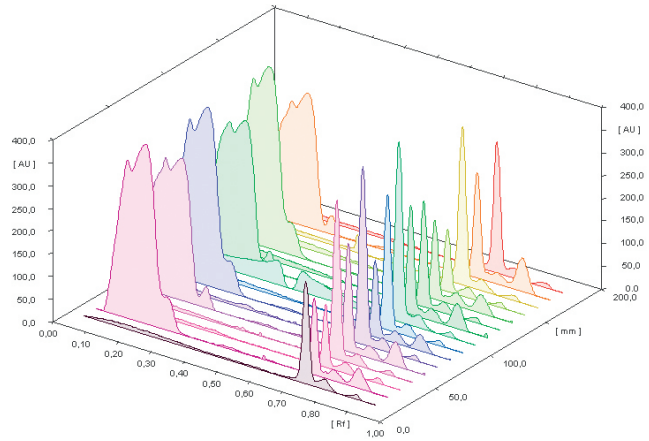
Die Kalibration von ß-Ecdyson wurde im Bereich von 0.04 bis 0.2 µg/Band (sdv 1.6 %) durchgeführt. Die untersuchten Extrakte enthielten 0.4 to 0.6 g/kg ß-Ecdyson, d.h. es wurde ein Gehalt von 0.8 bis 1.2 % ß-Ecdyson im ursprünglichen Trockenextrakt ermittelt.



15

▲ *Kalibrationskurve von ß-Ecdyson (x) mit Analysenproben (+)*

Die parallele Chromatographie mehrerer Bahnen auf einer Platte – simultan und unter identischen chromatographischen Bedingungen – ermöglicht eine schnelle Bestimmung des aktiven Inhaltstoffes ß-Ecdyson in den Trockenextrakten kaltgepresster *Pfaffia glomerata* Rhizome.



16

▲ *Parallele Chromatographie von 16 Bahnen unter identischen chromatographischen Bedingungen zur schnellen Quantifizierung von ß-Ecdyson in Pfaffia glomerata Rhizomextrakten*

Weitere Informationen sind vom Autor auf Anfrage erhältlich.

[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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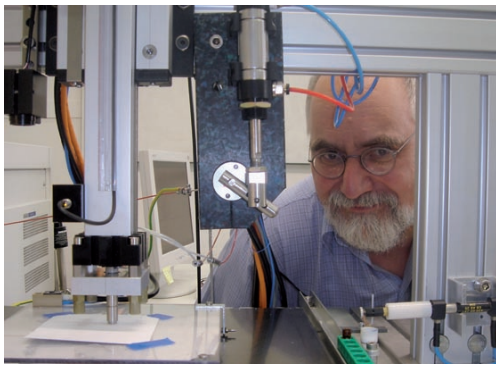
Automatisierte HPTLC/ESI-MS-Kopplung



17

▲ *Dr. Heinrich Luftmann*

Dr. Luftmann, Leiter der Abteilung Massenspektrometrie, Organisch-Chemisches Institut der Westfälischen Wilhelms-Universität in Münster, hatte bereits in CBS 94 die schnelle und kontaminationsfreie Extraktion von DC-Zonen zur Aufklärung von Synthesegemischen mittels Massenspektrometrie aufgezeigt. Inzwischen wurde die Extraktorkopf-Positionierung automatisiert. Zusammen mit Professor Aranda und Dr. Morlock*, Universität Hohenheim in Stuttgart, wurde die automatische Aufnahme von relevanten Zonen einer ganzen HPTLC-Platte aufgezeigt und die Leistungsgüte des automatisierten Interfaces ermittelt [1].

Einleitung

Die Zuverlässigkeit des automatisierten Interfaces wurde untersucht, indem es nicht nur zur Identifizierung der Zonen, sondern auch zur Quantifizierung mittels Elektrospray-Ionisation Massenspektrometrie (ESI-MS) eingesetzt wurde. Die Ergebnisse mittels HPTLC/MS wurden validierten HPTLC/UV-Methoden gegenübergestellt und anhand der Coffein-Quantifizierung in einer Energy Drink-Probe [2] und in Kopfschmerztabletten [3] bewertet. Im Vergleich zu anderen Kopplungs-Ansätzen wurde dabei ohne einen internen Standard zur Korrektur gearbeitet. Somit spiegeln die erzielten Ergebnisse die Leistungsgüte des Interface zur HPTLC/MS-Kopplung unverblümt wider.

Der Vorteil dieses Interfaces liegt darin, dass es in jedes beliebige LC/MS-System ohne Modifizierung integriert werden kann. Gegenüber Desorptionsverfahren kann es die ganze Zone samt ihrem Tiefenprofil extrahieren und erlaubt somit zur HPLC/MS vergleichbare Nachweisgrenzen im pg/Band-Bereich [4]. Generell ist bei der HPTLC/MS die zeitliche Beanspruchung des massenspektrometrischen Detektors im Vergleich zur HPLC/MS geringer, da nach erfolgter densitometrischer Auswertung ganz gezielt, nur von interessierenden Zonen MS-Spektren aufgenommen werden (Vorteil durch das offline Verfahren).

Probenvorbereitung

A) Die Energy Drink-Probe wurde 20 min im Ultraschallbad entgast.
B) Fünf Kopfschmerztabletten wurden im Mörser zerkleinert. Das durchschnittliche Gewicht einer Tablette (0.6 g) wurde in einem 50 mL Messkolben in 40 mL Methanol – Wasser 7:3 20 min unter Schütteln (500/min) und 10 min im Ultraschallbad gelöst und anschliessend bis zur Marke aufgefüllt. Ein Aliquot wurde filtriert (0.45 µm) und 1:20 mit Methanol verdünnt.

Standardlösung

Coffein wurde in Methanol gelöst (0.1 mg/mL).

Schicht

HPTLC-Alufolie Kieselgel 60 F_{254} (Merck), 10 × 10 cm

Probenauftragung

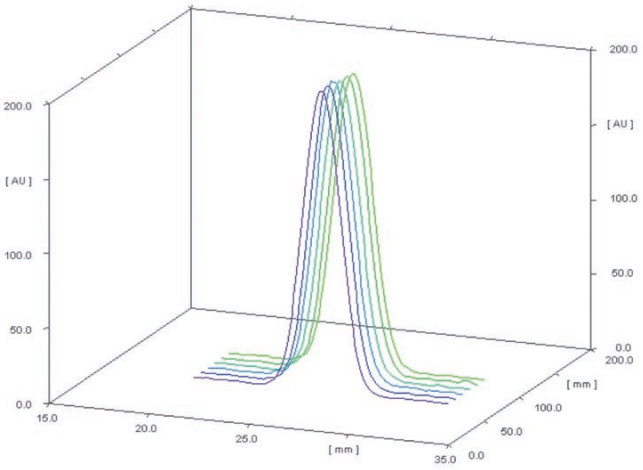
Bandförmig mit dem DC-Probenautomat 4, Bandlänge 3 mm, unterer Randabstand 10 mm, Bahnabstand 7.2 mm, Auftragevolumina 0.5–5 µL (50–500 ng/Band) für die Standardlösung und 1 µL für die Probelösungen, entsprechend 100 (Tablette) bzw. 320 ng/Band Coffein (Energy Drink).

Chromatographie

In der Flachbodenkammer 10 × 10 cm mit Ethylacetat – Methanol – Ammoniak (25 %) 90:15:1 (Tablette) bzw. Chloroform – Ethanol – Essigsäure (37 %) – Aceton – Wasser 54:27:10:2:2 (Energy Drink), Laufstrecke jeweils 80 mm (vom unteren Plattenrand).

Densitometrie

Absorptionsmessung bei UV 274 nm mit TLC-Scanner 3 und winCATS Software; polynome Kalibration über die Peakfläche.



18

▲ *Absorptionsmessung (Analytfenster): Präzision von 6 HPTLC-Zonen (100 ng/Band Coffein)*

Dokumentation

Mit DigiStore 2-Dokumentationssystem; Aufnahme im Auflicht bei UV 254 nm

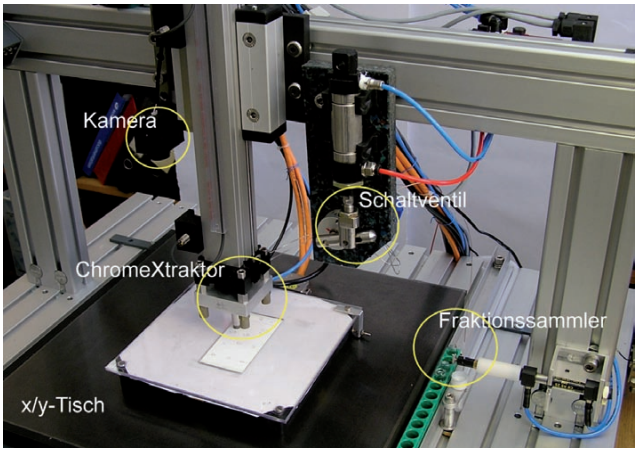

19

▲ *Dokumentation einer Kalibration (Plattenausschnitt)*

Automatisierte online-Extraktion und Aufnahme der Massenspektren

Die Eingangskapillare des Interfaces wurde an einer Pumpe (L6200, Merck-Hitachi, Tokyo, Japan) angeschlossen und die Extraktion erfolgte bei einer Flussrate von 0,1 mL/min mit Methanol – Ammoniumformiat (10 mmol/L) 19:1, pH 4. Die Ausgangskapillare des Interfaces wurde direkt mit dem ESI-MS (Quattro LCZ, Waters-Micromass, Manchester, UK mit Mass Lynx 3.2 Software) verbunden: Kapillarspannung 3.5 kV, Konusspannung 42 V, Extraktorspannung 3 V, R_F-Linsenspannung 0.28 V, Quellentemperatur 110 °C, Desolvatisierungstemperatur 180 °C.

Die HPTLC-Folie wurde auf dem x/y-Tisch (20 x 20 cm) fixiert und in die Bildaufnahme-Position gefahren. Nach Übertragung der Plattenposition wurde durch Anklicken der Zonen auf dem Bildschirm, von denen ein Massenspektrum aufgenommen werden sollte, die Extraktionsreihenfolge festgelegt. Anschliessend wurden die Zonen im 1.8 min-Takt mittels des ovalen Extraktorstempels (4 x 2 mm) extrahiert und nacheinander ins ESI-MS überführt. Zwischen den Extraktionen erfolgte für 4 s eine Stempelkopf-Reinigung.

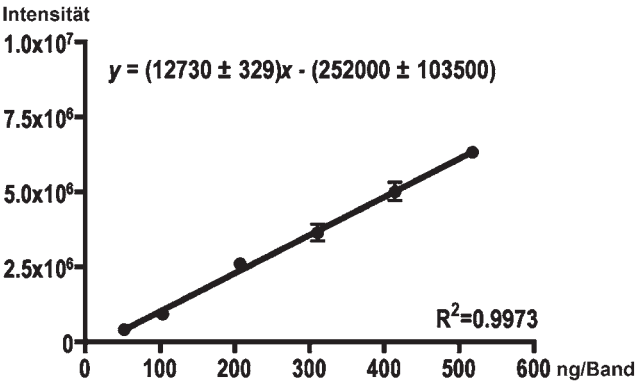


20

▲ *HPTLC/ESI-MS-Kopplung: Automatisierte Positionierung und Extraktion von HPTLC-Zonen*

Ergebnisse und Diskussion

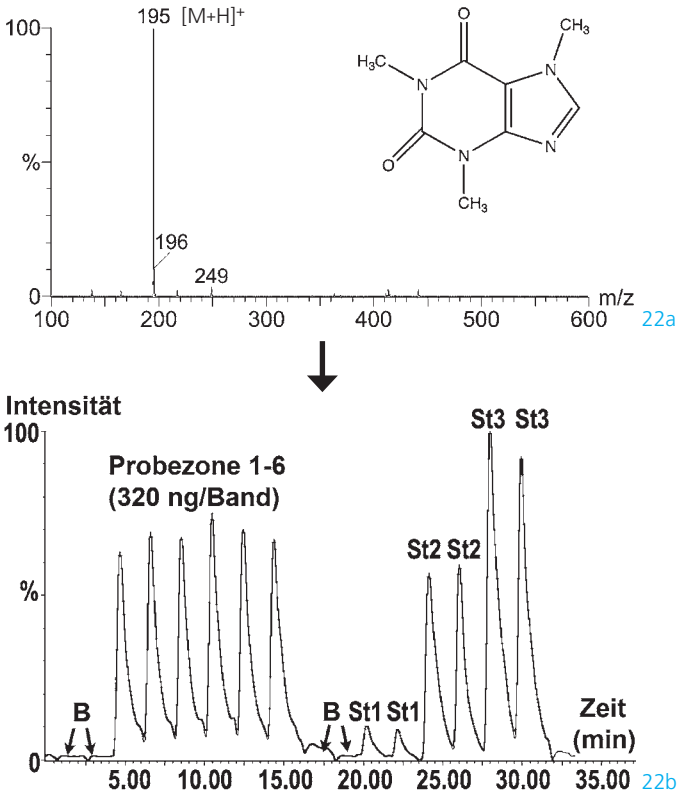
Der FullScan (*m/z* 100–600) zeigte deutlich das protonierte Molekül bei *m/z* 195 $[M+H]^+$. Ohne jeglichen internen Standard wurde das Coffein-Massensignal im SIM (selected ion monitoring)-Modus bei *m/z* 195 $[M+H]^+$ aufgenommen. Die Validierung der HPTLC/ESI-MS-Methode zeigte hoch zuverlässige Daten, i.e. eine lineare Regression mit einem Bestimmtheitsmass r^2 von 0,9973, eine Wiederholbarkeit (%*RSD*, *n* = 6) von 5,6 % und eine Wiederholbarkeit des Mittelwertes über 3 Platten (%*RSD*, *n* = 3) von 1,5 %.



21

▲ *Kalibrierfunktion mittels HPTLC/ESI-MS (SIM-Modus)*

In Kopfschmerztabletten und Energy Drinks wurde Coffein im SIM-Modus bei *m/z* 195 $[M+H]^+$ quantifiziert.



▲ *FullScan (oben) und SIM-Elutionsprofile (unten) extrahierter Coffeinzonen (B: Blindwerte, St: Standardzone)*

Die Genauigkeit der HPTLC/MS-Bestimmung wurde durch den Vergleich mit validierten HPTLC/UV-Methoden untersucht. Gemäss dem t- und F-Test ($P < 0.01$, $\upsilon_1 = 5$, $\upsilon_2 = 4$) lieferten die unterschiedlichen Detektionsmethoden vergleichbare Ergebnisse.

Automatisiertes Interface: HPTLC/ESI-MS versus HPTLC/UV

Coffein in	Arzneimittel Mittelwert ± SD (mg/Tablette)	Energy Drink Mittelwert ± 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)
Etikettenangabe	100	32

Das automatisierte Interface ermöglichte eine personalfreie Aufnahme der Massenspektren und war zudem mit einer Bearbeitungsrate von 1,8 min pro Band um 20 % schneller als das manuelle Interface. Vergleicht man die erreichte Präzision (%*RSD* = 5,6%) und das Bestimmtheitsmass der linearen Regression ($r^2 = 0.9973$) mit der bis dato einzigen HPTLC/MS-Publikation ohne internen Standard (%*RSDs* 7,6%, 14,4 % und 16,8 %, r^2 zwischen 0,95 und 0,98, [5]), so ermöglicht dieses automatisierte Interface im internationalen Vergleich eine äusserst zuverlässige quantitative HPTLC/MS-Kopplung. Darüber hinaus kann das Interface auch zur Wiedergewinnung von Zonen (Fraktionssammler) für weiterführende Untersuchungen mit NMR oder FTIR etc. eingesetzt werden.

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

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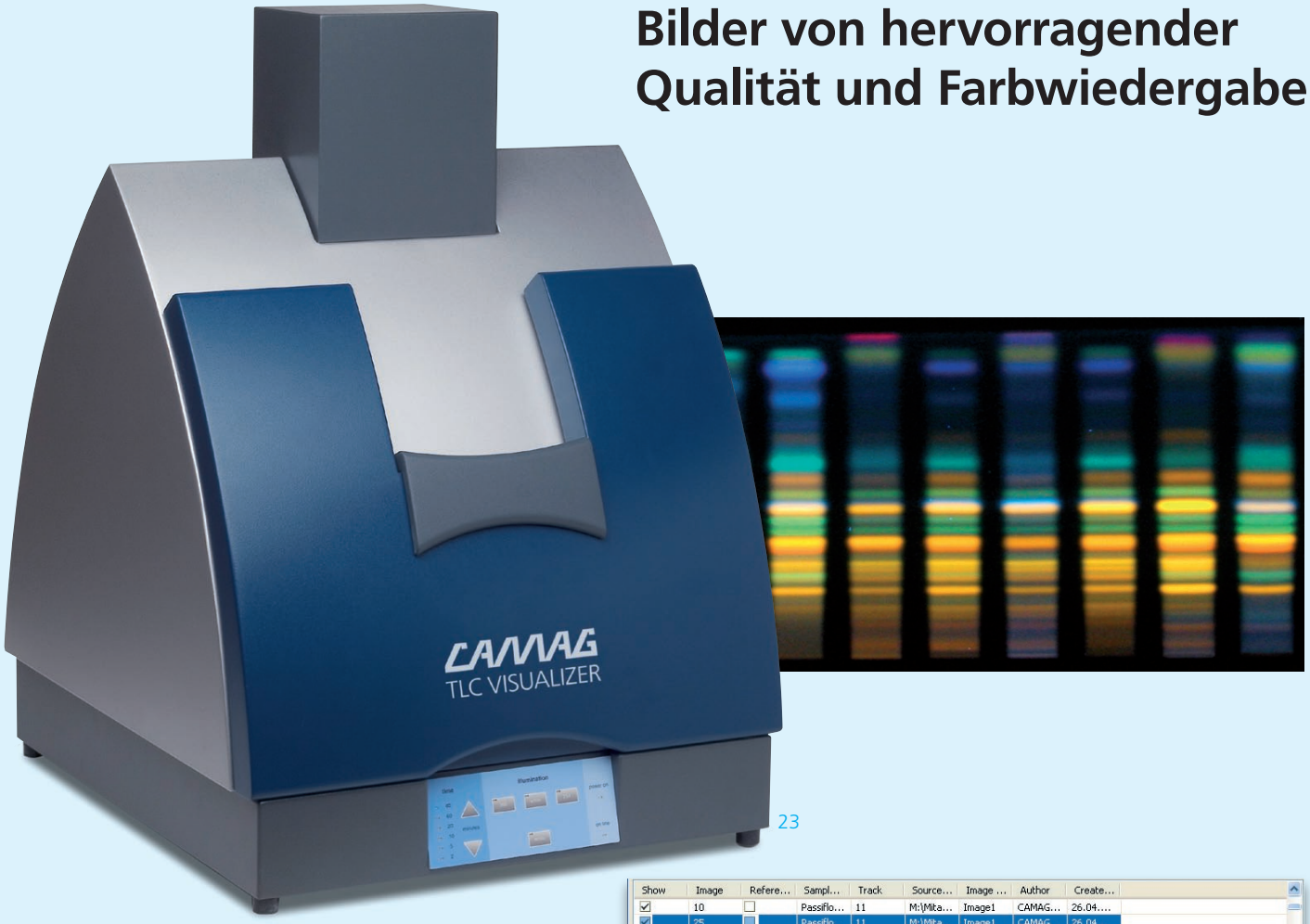
[1] Luftmann, H., Aranda, M., Morlock, G., Rapid Commun. Mass Spectrom. 21 (2007) 3772
[2] Aranda, M., Morlock, G., J. Chromatogr. A 1131 (2006) 253
[3] Aranda, M., Morlock, G., J. Chromatogr. Sci. 45 (2007) 251
[4] Jautz, U., Morlock, G., J. Chromatogr. A 1128 (2006) 244
[5] Van Berkel G., Tomkins B., Kertesz V., Anal Chem 79 (2007) 2778

TLC VISUALIZER

Das leistungsfähige Auswertungs- und Dokumentationssystem



23

Bilder von hervorragender Qualität und Farbwiedergabe

- Lineare und hochauflösende CCD Kamera
- Neue Beleuchtungseinheit für alle Beleuchtungsarten
- Hervorragende Softwareeigenschaften
- Bildoptimierung für beste Resultate in qualitativer und quantitativer Chromatogramm-Auswertung

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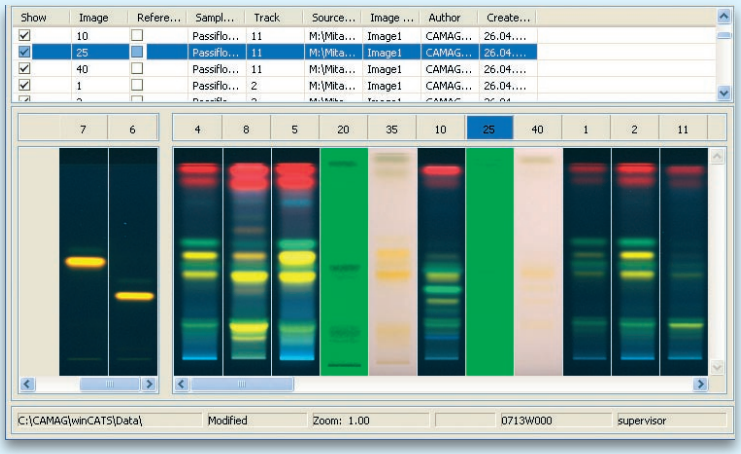

24

CAMAG

Weltweit führend in der Planar-Chromatographie

CBS 100 D 996.1310-3