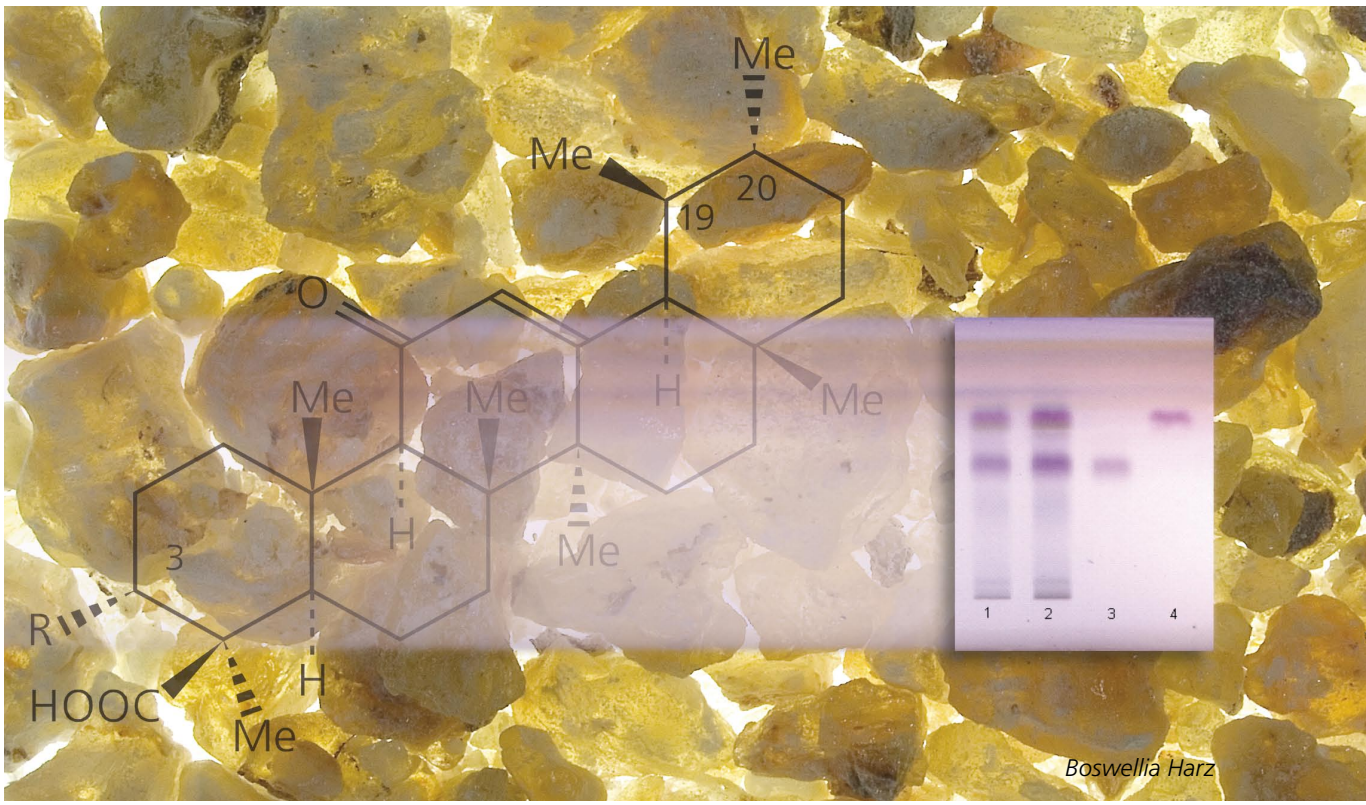
CBS 104 • März 2010

CAMAG LITERATURDIENST PLANAR-CHROMATOGRAPHIE

CBS

CAMAG BIBLIOGRAPHY SERVICE



Boswellia Harz

Analytische Aufgaben – effizient mit HPTLC gelöst

CAMAG 104

CBS
CAMAG BIBLIOGRAPHY SERVICE

Nr. 104, März 2010
CAMAG Literaturdienst
Planar-Chromatographie
Herausgegeben von Gerda Morlock
cbs@camag.com
Eigenverlag CAMAG Schweiz

IN DIESER AUSGABE

CAMAG

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

CAMAG (Deutschland)
Bismarckstr. 27–29 • D-12169 Berlin
Tel. +49 30 516 55 50 • Fax +49 30 795 70 73
info@camag-berlin.de

www.camag.com

Druck auf FSC/PEFC-zertifiziertem Papier

Vergleich zwischen HPLC und HPTLC zur Trennung und Quantifizierung von Boswelliasäuren in *Boswellia serrata*-Extrakten

1

Von links: Prof. Alexander Shikov, Dr. Olga Pozharitskaya, Dr. Svetlana Ivanova, Prof. Valery Makarov, Dr. Vera Kosman*

Die Abteilung für Standardisierung und Neue Technologien des Sankt Petersburger Instituts für Pharmazie wurde bereits in CBS 102 vorgestellt. In ihrer täglichen Routine ist die HPTLC besonders hilfreich bei der Optimierung der Extraktion, der Bestimmung der antiradikalischen Aktivität und zur Erforschung der Kinetik von aktiven Metaboliten in Plasma. Zur Zeit verwenden sie über 20 validierte quantitative HPTLC-Methoden, wie etwa zur Quantifizierung von Chlorogen-, Kaffee- und Ferulasäuren in Kaffeebohnenextrakten, Curcuminoiden in Plasma und arzneilichen Darreichungsformen oder Gallensäuren und Cholesterol in Gallenflüssigkeit von Ratten.

Einleitung

Boswellia serrata Roxb. (Indischer Weihrauch) ist eine indische Heilpflanze, deren Gummiharz traditionell – neben anderen Anwendungen – als fiebersenkendes, schmerzlinderndes, diuretisches oder antidysenterisches Arzneimittel eingesetzt wird. Die entzündungshemmende und antiarthritische Aktivität des Extrakts wurde bereits in Tierversuchen und klinischen Studien überprüft [1, 2]. Die aktiven Hauptkomponenten des *Boswellia serrata*-Extrakts sind Triterpensäuren [2] wie β-Boswelliasäure (BA), Acetyl-β-boswelliasäure (ABA), 11-Keto-β-boswelliasäure (KBA) und Acetyl-11-keto-β-boswelliasäure (AKBA). Für die Analyse von Boswelliasäuren wurden bereits eine nicht-wässrige Titrationsmethode [3] sowie Methoden für die HPLC [4], GC-MS [5], LC-MS [6] und HPTLC [7] veröffentlicht.

Ziel dieser Arbeit war es, die Leistungsfähigkeit zweier chromatographischer Methoden, HPTLC und HPLC, zur Identifikation und Quantifizierung der vier wichtigsten Boswelliasäuren in *Boswellia serrata*-Extrakten zu vergleichen.

Chromatographische Schicht/HPLC-Säule

- HPTLC-Platten Kieselgel 60 F_{254}, 10 x 10 cm (Merck)
- HPLC-Säule Luna C_{18} (150 x 4,6 mm, 5 µm, Phenomenex, USA) und Vorsäule Security Guard 20 mm mit demselben Säulenmaterial

Standardlösungen

Methanolische Lösungen (0,5 mg/mL) von BA, ABA, KBA und AKBA

Probenvorbereitung

Kommerziell erhältliche standardisierte *Boswellia serrata*-Extrakte wurden von New Ambadi Estates Pvt. Ltd. (Chennai, Indien) zur Verfügung gestellt. Die Probelösungen wurden durch Lösen der *Boswellia serrata*-Extrakte in Methanol hergestellt (10 mg/mL).

Probenauftragung/Injektion

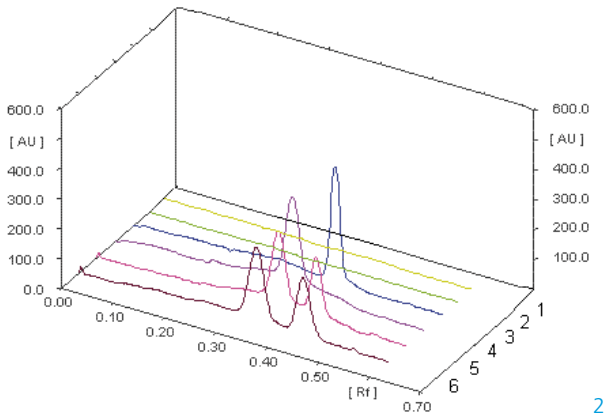
- Bandförmig mit Linomat 5, Bandlänge 6 mm, Abstand von allen Rändern 10 mm, Auftragevolumen 1–20 µL
- Injektionsvolumen 20 µL für die HPLC-Analyse

Chromatographie

- In der Doppeltrogkammer mit Kammersättigung (15 min mit befeuchtetem Filterpapier) mit n-Hexan – Ethylacetat – Eisessig 16:5:1 bis zu einer Laufhöhe von 7 cm (ca. 10 min).
- HPLC-Anlage (Waters Inc., USA) mit zwei Pumpen der Serie 510, einer automatischen Pumpensteuerung und einem einstellbaren UV-Detektor Model 484. Die Trennung erfolgte bei Raumtemperatur und einer Flussrate von 1 mL/min mit einem 20-minütigen linearen Gradienten von 75 % bis 100 % Acetonitril (weitere Phase: 0,03 %ige, wässrige Trifluoressigsäure-Lösung), gefolgt von einer 15-minütigen isokratischen Stufe.

Densitometrie

- Absorptionsmessung bei 254 nm für KBA und AKBA und nach Derivatisierung bei 560 nm für BA und ABA mit TLC-Scanner 3 und winCATS-Software.
- HPLC-UV-Detektion bei 254 nm für KBA und AKBA bzw. bei 210 nm für BA und ABA.



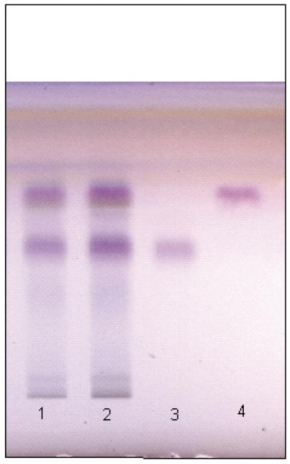
2

HPTLC-Densitogramm bei 254 nm von BA (Bahn 1), ABA (Bahn 2), AKBA (Bahn 3), KBA (Bahn 4), und zwei B. serrata-Extrakten (Bahnen 5 und 6).

Postchromatographische Derivatisierung

Zur Detektion von BA und ABA wurde die Platte manuell in Anisaldehyd-Schwefelsäurereagenz getaucht (1 mL Anisaldehyd und 2 mL konz. Schwefelsäure in 100 mL Eisessig) und danach 5 min bei 110 °C auf dem DC-Plattenheizer erhitzt.

Anmerkung (Editor): Durch automatisiertes Tauchen mit Hilfe der DC-Tauchvorrichtung (Tauchzeit 1 s, Tauchgeschwindigkeit 3,5 cm/s) kann die Präzision verbessert werden.



3

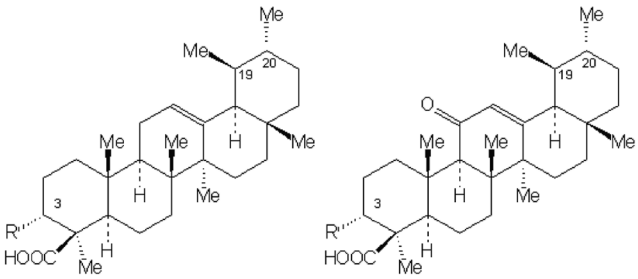
Visualisierung mit Anisaldehyd-Schwefelsäurereagenz: Bahnen 1 und 2 B. serrata-Extrakt mit 5 bzw. 10 µg/Band, Bahn 3 BA und Bahn 4 ABA

Ergebnisse und Diskussion

Für beide Methoden wurde die Quantifizierung der Boswelliasäuren mittels externer Standardkalibration durchgeführt und beide Methoden wurden gemäss ICH-Richtlinien zur Validierung analytischer Methoden validiert. Die Nachweisgrenze für KBA und AKBA lag bei der HPLC mit einem Injektionsvolu-

men von 20 µL bei 6–8 ng und für BA und ABA bei 60 – 80 ng. Bei der HPTLC wurden nur 2 µL aufgetragen. Die hier erreichten Nachweisgrenzen lagen bei 150 ng für KBA und AKBA und bei 100 ng für BA und ABA. Bei Bedarf kann die Nachweisgrenze durch ein höheres Auftragevolumen noch verbessert werden. Hinsichtlich der Präzision ergaben sich bei der HPLC relative Standardabweichungen (*%RSD*) von 6 bis 18 % für die Boswelliasäuren. In der HPTLC waren die Präzisionen für AKBA und KBA ≤ 2 % und für BA und ABA nach Derivatisierung etwa 10 %, was mit automatisiertem Tauchen noch verbessert werden könnte.

Zwei getrocknete *Boswellia serrata*-Extrakte aus verschiedenen Bezugsquellen wurden dreimal mit beiden Methoden analysiert.



Boswelliasäuren:

BA mit R = α-OH KBA mit R = α-OH

ABA mit R = α-CH_3COO AKBA mit R = α-CH_3COO

Ergebnisse der HPLC- und HPTLC-Quantifizierung von Boswelliasäuren in *Boswellia*-Extrakten

Substanz	Probe 1 (%, w/w)[a]		Probe 2 (%, w/w)[a]	
	HPLC	HPTLC	HPLC	HPTLC
BA	18.9 ± 1.2	19.0 ± 1.7	22.4 ± 0.7	23.4 ± 1.9
ABA	11.9 ± 1.2	12.4 ± 1.4	11.6 ± 0.3	12.3 ± 1.2
KBA	3.8 ± 0.6	3.4 ± 0.1	8.0 ± 0.1	7.7 ± 0.1
AKBA	3.9 ± 0.7	3.6 ± 0.1	5.1 ± 0.2	5.2 ± 0.1
Gesamt	38.5	37.9	47.1	48.6

[a] ± Standardabweichung (*n* = 3), bezogen auf das Trockengewicht

Die beiden Methoden wurden mit dem Student t-Test für gepaarte Stichproben verglichen. Die berechneten t-Werte lagen für die verschiedenen Boswelliasäuren zwischen 2,16 und 2,45 und waren kleiner als der t-Tabellenwert von 2,57. Somit lieferten beide Methoden bei der Bestimmung von Boswelliasäuren aus Extrakten identische Resultate. Es gab keinen statistisch signifikanten Unterschied (P = 99 %) zwischen den Mittelwerten aller Extrakte. Unter Berücksichtigung der kurzen Analysendauer, des geringen Lösungsmittelverbrauchs und dem hohen Probendurchsatz ist die HPTLC die Methode der Wahl in der Routineanalytik.

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

[1] T. Syrovets et al. J. Immunol. 174 (2005) 498

[2] B.A. Shah et al. Nat. Prod. Rep. 26 (2009) 72

[3] R.K. Gupta et al. Indian Drugs 21 (1984) 523

[4] B. Buchele and T. Simmet J. Chromatography B 795 (2003) 355

[5] A. Kaunzinger et al. J. Pharm. Biomed. Anal. 28 (2002) 729

[6] K. Reising et al. Anal. Chem. 77 (2005) 6640

[7] O.N. Pozharitskaya et al. J. Sep. Sci. 29 (2006) 2245

* Prof. Dr. Alexander Shikov, Saint Petersburg Institute of Pharmacy, 47/5, Piskarevsky prospect, 195067, St. Petersburg, Russland, spb.pharmacy@gmail.com

Validierte HPTLC-Methode zur Bestimmung von Hautlipiden

Karin Rothenbühler

In Zusammenarbeit mit der Universität Basel unter Betreuung von Professor Matthias Hamburger hat Frau Karin Rothenbühler ihre Masterarbeit [1] in Pharmazeutischer Biologie im CAMAG Labor durchgeführt.

Einleitung

Aus der Literatur kann man schließen, dass die DC/HPTLC als Methode der Wahl für die Analytik von Hautlipiden anzusehen ist. Dennoch wurde keine validierte quantitative HPTLC-Methode gefunden. Frau Rothenbühler hat deshalb eine HPTLC-Methode für apolare Hautlipide entwickelt und validiert, die auf dem kürzlich veröffentlichten Artikel über die Bestimmung von Phospholipiden mittels HPTLC basiert [2]. Die Methode ist für die wichtigsten Lipide des menschlichen Stratum corneum geeignet: Squalen, Triolein, Palmitinsäure, 1,2-Dipalmitoyl-sn-glycerol, Stearylpalmitat, Cholesterylpalmitat und Cholesterol.

In Kombination mit einer geeigneten Technik zur Entnahme von Hautproben ist die vorgestellte Methode schnell, robust, zuverlässig und gut geeignet für den Einsatz in Labors der kosmetischen Industrie. Die einfache Derivatisierung als Voraussetzung zur Detektion der Lipide und die Trennung nach funktionellen Gruppen sind Stärken der HPTLC gegenüber der RP-HPLC.

Probenvorbereitung

Hautproben wurden Testpersonen von der Innenseite des Unterarms durch direkte Extraktion mit Ethanol, Abkratzen einer definierten Fläche mittels Rasierklinge oder mithilfe des medizinischen Wundklebers Epiglu entnommen.

Standardlösungen

Je 2 mg Squalen, Triolein, Palmitinsäure, 1,2-Dipalmitoyl-sn-glycerol, Stearylpalmitat, Cholesterylpalmitat und Cholesterol wurden in 10 mL Chloroform – Methanol 1:1 gelöst. Für die Quantifizierung wurde 1 mL dieser Lösung mit 4 mL Chloroform – Methanol 1:1 verdünnt.

Schicht

HPTLC-Platten LiChrospher Kieselgel 60 F_{254} (Merck), 20 × 10 cm, vorgewaschen durch Chromatographie mit Methanol und 20 min bei 120 °C im Ofen getrocknet.

Probenauftragung

Bandförmig mit DC-Probenautomat 4, Bandlänge 8 mm, Bahnabstand mind. 10 mm, unterer Randabstand 8 mm, seitlicher Randabstand mind. 15 mm, Auftragevolumina 2–30 µL für Proben und 2.5–10 µL für Standardlösungen.

Chromatographie

In der automatischen Entwicklungskammer ADC2 nach Kammersättigung für 20 min mit Toluen über eine Laufstrecke von 80 mm (vom unteren Plattenrand), nach 5 min Trocknen zweite Entwicklung mit *n*-Hexan – *t*-Butylmethylether – Essigsäure 80:20:1, Laufstrecke 45 mm. Die Trennung wird von der Plattenaktivität beeinflusst, deshalb wurden die Platten für beide Entwicklungsschritte während 10 min mit einer gesättigten $MgCl_2$-Lösung bei 33 % relativer Feuchte konditioniert.

Postchromatographische Derivatisierung

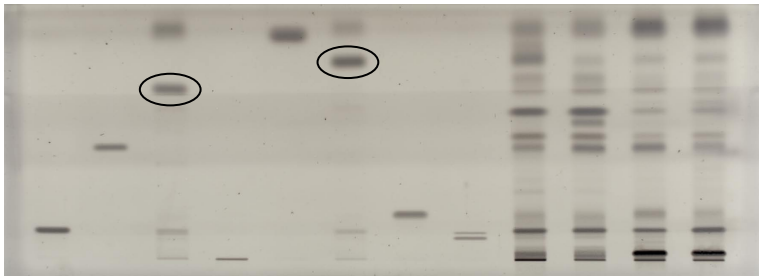
20 g Kupfer(II)-sulfat wurden in 200 mL eiskaltem Methanol gelöst, danach wurden unter Kühlen 8 mL konzentrierte Schwefelsäure und 8 mL Phosphorsäure 85 % zugegeben. Die Platte wird mit der Chromatogramm-Tauchvorrichtung III während 6 s in die Lösung getaucht, 30 s getrocknet und auf dem DC-Plattenheizer für 30 min bei 140 °C erhitzt.

Densitometrie

Absorptionsmessung bei 350 nm mit dem TLC-Scanner 3 mit winCATS Software

Ergebnisse und Diskussion

Mangels einer standardisierten Methode zur Entnahme von menschlichen Hautproben wurden verschiedene Techniken untersucht. So wurden Hautproben von Testpersonen mithilfe verschiedener kommerzieller Klebstreifen, medizinischer Wundkleber, durch direktes Abkratzen mittels Rasierklinge, mittels Kaltwachs, Aluminiumfolie, Frischhaltefolie oder durch direktes Aufbringen organischer Lösungsmittel entnommen.

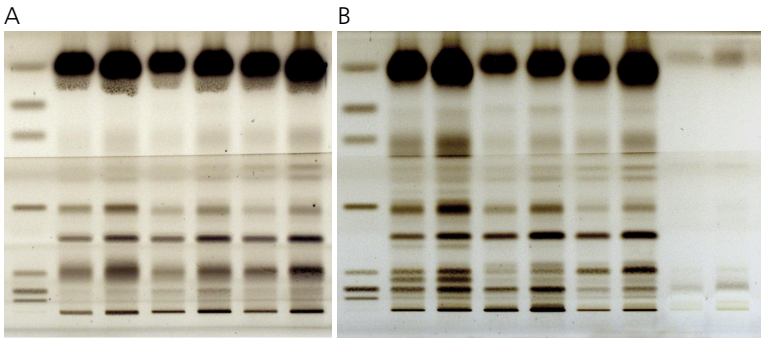


5

Standardsubstanzen (Bahn 1: Cholesterol, 2: Triolein, 3: Stearylpalmitat, 4: Ceramid VI, 5: Squalen, 6: Cholesterylpalmitat*, 7: Palmitinsäure, 8: 1,2-Dipalmitoyl-sn-glycerol) und verschiedene Hautlipidextrakte (Bahn 9: extrahiert mit Ethanol, 10: Cyclohexan – Ethanol 1:9, 11: Cyclohexan – Ethanol 1:4, 12: n-Hexan – Methanol 2:3).*

**entspricht eingekreister Zone, weitere Zonen sind Verunreinigungen*

Dabei stellte bei der Probenentnahme mit Klebstreifen und Wundklebern die selektive Extraktion der Hautlipide aus dem Trägermaterial die größte Herausforderung dar. Das direkte Aufbringen organischer Lösungsmittel war auf Ethanol begrenzt, um Hautreizungen zu vermeiden. Für quantitative Untersuchungen waren zur Probennahme die direkte Extraktion mit Ethanol, Abkratzen und Epiglu geeignet, da die Chromatographie weder durch die Haut- noch die Klebermatrix negativ beeinflusst wurde. Die Standardisierung der Probennahme erwies sich als schwierig, da die Tiefe der abgelösten Haut (Anzahl Schichten des Stratum corneum) und ein klar abgegrenztes Hautareal nicht einfach bestimmbar waren. Weitere Untersuchungen erfolgen hierzu.



6

Probennahme A) durch Abkratzen mit einer Rasierklinge, B) mit Epiglu Wundkleber; Bahn 1: Mischung aller Standardsubstanzen, Bahnen 2–7: Hautlipidproben, 8–9: Epiglu Blindprobe.

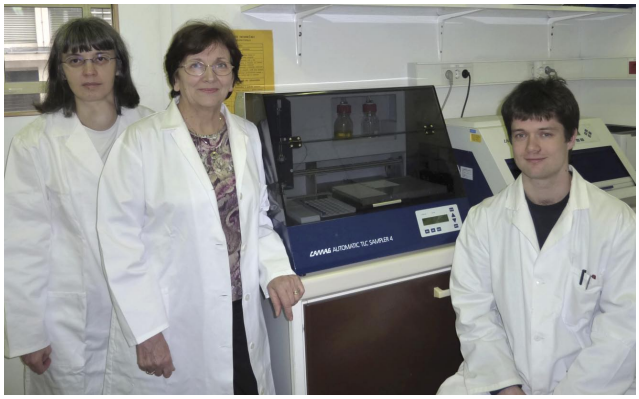
Die Methode wurde bezüglich Stabilität, Robustheit, Präzision der R_F-Werte und Standardsubstanzen sowie Linearität validiert. Alle Standardlösungen waren mind. 3 h stabil, sei es in Chloroform – Methanol 1:1 oder auf der Platte vor der Entwicklung; ebenso waren alle Standardsubstanzen und Hautextrakte im chromatographischen System stabil.
Das derivatisierte Chromatogramm veränderte sich während mind. 2 h nicht. Der Einfluss der relativen Feuchte auf die Trennung wurde in der ADC2 bei 5 %, 33 %, 47 % und 75 % untersucht. Für eine reproduzierbare Selektivität der Trennung sollte die relative Feuchte zwischen 33 % und 47 % konstant gehalten werden. Die Präzision des qualitativen Ergebnisses wurde anhand von R_F-Werten von sieben Substanzen von Platte zu Platte ermittelt (3 Platten wurden an einem Tag entwickelt, danach an zwei weiteren Tagen jeweils eine Platte). Die gemessenen R_F-Wert-Unterschiede waren <0.03 und lagen somit weit unter dem Akzeptanzkriterium von <0.05 R_F-Einheiten. Die Präzision der Gehaltsbestimmung (n = 9) wurde auf HPTLC-Platten Kieselgel 60 mit verschiedenen Schichtdicken (100 und 200 µm) und Partikelformen (unregelmässig und sphärisch (LiChrospher)) untersucht. Die LiChrospher-Platten mit einer Schichtdicke von 200 µm wiesen für alle Substanzen die beste relative Standardabweichung auf (%*RSD* ≤ 5 %). Der lineare Bereich fast aller ausgewählten Marker lag zwischen 100 und 350 ng/Zone, mit Ausnahme von Cholesterol zwischen 40–160 ng und 1,2-Dipalmitoyl-sn-glycerol zwischen 100–280 ng. Die Kalibrierkurven wiesen Korrelationskoeffizienten >0.9975 auf.

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

[1] K. Rothenbühler, Masterarbeit, Institut für Pharmazeutische Biologie, Universität Basel, 2009

[2] D. Handloser, V. Widmer, E. Reich, J. Liq. Chromatogr. Rel. Technol. 31 (2008) 1857

Trennung pflanzlicher Triterpenoide mittels HPTLC



Von links: Dr. Irena Vovk, Dr. Breda Simonovska und Mitja Martelanc*

Die Forschungsgruppe des lebensmittelchemischen Labors am Staatlichen Institut für Chemie in Ljubljana (www.ki.si) ist in Slowenien führend auf dem Gebiet der Planar-Chomatographie und bietet Consulting für Industrie und andere Institutionen an. Der Arbeitsschwerpunkt der Forschung & Entwicklung liegt im Bereich Nutrazeutika und bei der Entwicklung analytischer Methoden, die auf chromatographischen Trenntechniken basieren.

Einleitung

Triterpenoide repräsentieren eine grosse Klasse sekundärer Metabolite meist pflanzlicher Herkunft, die vor allem in Blattwachsen und epicuticularen Wachsen von Früchten vorkommen. Einige davon weisen positive Eigenschaften auf und wirken entzündungshemmend. Daher nimmt ihre Bedeutung als Bestandteil von Nahrungsergänzungsmitteln und Functional Food zu.

Üblicherweise wird die HPLC zur Bestimmung von Triterpenoiden in Pflanzenextrakten eingesetzt. Jedoch ist die Wahl der Detektionsbedingungen und der mobilen Phase eingeschränkt, da Triterpenoide keine Chromophore besitzen und es eine Vielzahl von möglichen Isomeren gibt. Gegenüber der HPLC bietet die HPTLC eine Vielfalt an mobilen Phasen und einzigartige Trennungen mit der Option zur *in situ*-Derivatisierung. Letztere ermöglicht eine charakteristische, spezifische Anfärbung und Fluoreszenz der getrennten Banden [1, 2] und dadurch ein einfaches, schnelles und kostengünstiges Screening, was die HPTLC bei der Untersuchung dieser Triterpenoide unverzichtbar macht. Abhängig von den Probeeigenschaften ist auch eine Quantifizierung möglich.

Standardlösungen

Standardlösungen und -gemische werden in *n*-Propanol angesetzt (0,1 mg/mL).

Probenvorbereitung

Eine ganze Tomate bzw. frische Kohl-, Rosmarin- oder Salbeiblätter werden in Dichlormethan eingetaucht. Pulverisierte Eichenrinde wurde mit 15 mL Dichlormethan für 15 min extrahiert. Nach Filtration und Abdampfen des Lösungsmittels werden die Rückstände in *n*-Propanol in folgenden Konzentrationen aufgenommen: 5 mg/mL (Kohl-, Rosmarinblätter), 30 mg/mL (Salbeiblätter), 10 mg/mL (Eichenrinde) und 2 mg/mL (Tomate) [2].

Schicht

HPTLC-Platte Kieselgel 60 (Merck), 20 x 10 cm, vorgewaschen durch Entwicklung mit Chloroform – Methanol 1:1 und HPTLC-Platte RP18 (Merck), 20 x 10 cm, vorgewaschen durch Entwicklung mit Aceton; anschliessende Trocknung auf dem DC-Plattenheizer bei 110 °C für 10 min.

Probenauftragung

Bandförmig mit Linomat, Bandlänge 6 mm, Abstand vom unteren Plattenrand 5 mm und vom seitlichen Plattenrand 12 mm, Bahnabstand 10 mm, Auftragevolumina 6 µL (Kohlblätter), 7 µL (Rosmarinblätter), 5 µL (Salbeiblätter), 4 µL (Eichenrinde) und 6 µL (Tomate), 2-8 µL für Standardlösungen

Chromatographie

In der Horizontal-Entwicklungskammer bei der Kieselgel-Platte mit *n*-Hexan – Ethylacetat 5:1 (Entwicklungsdauer 15 min), wobei der Konditioniertrog mit Fliessmittel benetzt wurde, und bei der RP18-Platte mit Ethylacetat – Acetonitril 3:2 oder Aceton – Acetonitril 5:1 (Entwicklungsdauer 17 min); Laufstrecken jeweils 8 cm

Postchromatographische Derivatisierung

Die getrockneten Platten wurden mit der Chromatogramm-Tauchvorrichtung für 2 s (Eintauchgeschwin-

Fortsetzung auf Seite 9

Online-Kopplung HPTLC-MS auf dem Weg zum Erfolg

Zu den CAMAG TLC/HPTLC-MS-Fortbildungstagen 2009 in Langenau, Offenburg, Basel, Münster, Berlin und Wien kamen Zuhörer aus den unterschiedlichsten Bereichen, in denen die DC/HPTLC eine wichtige und täglich eingesetzte Analysentechnik ist – so aus der Lebensmittelchemie, Pharmazie, Wasseranalytik, organischen Synthese- und Naturstoff-Forschung. Alle Veranstaltungen waren mit Teilnehmerzahlen zwischen 30 und 35 sehr gut besucht. Ziel war die praktische Erfahrung mit dem neuen TLC-MS-Interface, über dessen Markteinführung im Frühjahr 2009 in CBS 102 berichtet wurde.

8

Kompetente Vortragende präsentierten eindrucksvoll verschiedene Themenkreise mit Analysenbeispielen aus der täglichen Praxis. Den Anfang machte Dr. H. Luftmann (Universität Münster), der die historische Entwicklung des anspruchsvollen Anliegens schilderte, ein Interface zu entwickeln, das eine zuverlässige massenspektrometrische Identifizierung getrennter Substanzen direkt aus der Kieselgel-Trennschicht ermöglicht. Weitere Vorträge aus der Praxis widmeten sich der Wasseranalytik (Dr. W. Schulz, Zweckverband Landeswasserversorgung, Langenau), der pharmazeutischen und der Naturstoff-Analytik (Prof. Dr. H.-R. Schmutz, FH Nordwestschweiz, Muttenz) sowie der Lebensmittel- und Arzneimittelanalytik (PD Dr. G. Morlock, Universität Hohenheim, Stuttgart). Das behandelte Analytspektrum (z.B. Atrazin und seine Metaboliten, Metoprolol und seine Abbauprodukte, Coffein, Paracetamol, Acetylsalicylsäure, Ginsenoide, Isopropylthioxanthon, Lebensmittelfarbstoffe, Ergotamin, Pyridinol und Harman) war vielfältig.

Nach den Vorträgen fand eine Präsentation des Systems statt, bei der von Tagungsteilnehmern mitgebrachte DC/HPTLC-Platten vermessen wurden. Das TLC-MS-Interface wurde jeweils an das vor Ort verfügbare HPLC-MS-System gekoppelt. Je nach Ionisierbarkeit der Analyten sind für eine massenspektrometrische Identifizierung Substanzmengen im Piko- bzw. Nanogrammbereich pro getrennter Substanzzone auf der DC-/HPTLC-Platte ausreichend. Die vorgestellte Kopplungstechnik eignet sich nicht nur zur sicheren und schnellen Identifizierung durch die gemessenen Massenspektren, sondern kann auch für zuverlässige Quantifizierungen eingesetzt werden.

Einig waren sich alle: Sowohl die Zeitersparnis als auch die Kosteneffizienz zeichnen die neue Kopplungstechnik gegenüber der HPLC/MS-Technik aus.

Erfolg verpflichtet

Fortbildungsseminare zur Online-Kopplung HPTLC-MS auch in 2010:

- 16.04.2010 Universität Oldenburg, Prof. Dr. Christoffers, 26111 Oldenburg, Deutschland
- 03.06.2010 University of Leiden, Prof. Dr. Overkleeft, 2333 CC Leiden, The Netherlands
- 04.06.2010 University of Gent, Prof. Dr. De Spiegeleer, 9000 Gent, Belgium
- 29.06.2010 Technische Universität München, Prof. Dr. Schieberle, 85354 München, Deutschland

Bitte melden Sie Ihr Interesse an Dr. Konstantinos Natsias (info@camag-berlin.de). Sie erhalten dann ausführliche Unterlagen. Die Teilnahme ist kostenfrei.

CAMAG LITERATURDIENST
CAMAG BIBLIOGRAPHY SERVICE
PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

Das effiziente Lösen analytischer Fragestellungen mit der HPTLC ist Schwerpunkt dieser Ausgabe. Voraussetzung ist, dass das gesamte Instrumentarium optimal eingesetzt wird – eine Folgerung, die auch im Beitrag Coran (S. 5) gezogen wird.

Die HPTLC ist heute – 35 Jahre nach Einführung dieser Schichten – längst in der Praxis angekommen. Unsere zahlreichen Kundenkontakte zeigen allerdings auch, dass ein großer Teil der Prüfvorschriften in Industrie und Kontrollinstituten noch auf den klassischen DC-Schichten basiert. Dies liegt einerseits am mangelnden Kenntnisstand eines Teils der Anwender, andererseits am bürokratischen Aufwand, der erforderlich ist, eine DC-Methode auf HPTLC umzustellen. Aber wäre es nicht besser, die noch vorhandenen DC-Methoden Schritt für Schritt durch die HPTLC mit ihrem großen Potential zu ersetzen, als sich jahrelang im Routinebetrieb mit einer unzulänglichen Prüfvorschrift abzufinden? Bürokratie darf kein Hemmschuh für den analytischen Fortschritt sein!

Vorreiter sollten eigentlich die Universitäten sein. Doch gerade dort wird, wenn überhaupt, den Studierenden die Dünnschicht-Chromatographie oft nur in ihrem rudimentären Stand nahe gebracht. CAMAG ist bemüht, durch Seminare den Wissensstand zu aktualisieren. Wenn Sie als Lehrperson daran interessiert sind, melden Sie sich bitte unter info@camag-berlin.de

Eine Gelegenheit, Fortschritte in der HPTLC zu erfahren, ergibt sich beim International Symposium on High-Performance Thin-Layer Chromatography in Basel vom 6.–8. Juli 2011 (letzte gelbe Seite). Bitte reservieren Sie sich diesen Termin.

Mit freundlichen Grüssen

Gerda Morlock

Gerda Morlock
cbs@camag.com

Dear friends

The efficient solution of analytical tasks by HPTLC is the focus of this issue. An absolute prerequisite is a sound knowledge of the state-of-the-art technique – a conclusion drawn also by Professor Coran (p. 5).

HPTLC is now clearly established in the laboratory, 35 years after the introduction of the high-performance layers. But in the field, industry as well as regulatory laboratories, we meet many customers still using classical TLC layers and old fashioned procedures. To a large extent this is due to the fact that standard operating procedures are based on classical TLC layers, and that substituting an established procedure with HPTLC requires a lot of bureaucracy. Nonetheless, even this appears much better than struggling along for years with inadequate procedures and more importantly, suboptimal results.

Outriders for teaching state-of–the-art knowledge should be university institutes. But unfortunately, nowhere is it more apparent than at universities that TLC is just rudimentarily presented to students, if at all! CAMAG strives to upgrade the knowledge of contemporary HPTLC at universities by offering seminars. If you, as a teaching person are interested in a CAMAG seminar at your institute, contact us via info@camag.com.

An opportunity to see first hand the progress in HPTLC is the attendance of the International Symposium on High-Performance Thin-Layer Chromatography in Basle, July 6th–8th, 2011 (last yellow page). Make a note!

Sincerely,

Gerda Morlock

Gerda Morlock
cbs@camag.com

CAMAG

MARCH 2010 104

THE CBS CLASSIFICATION SYSTEM

1. **Reviews and books**
   a) Books on TLC
   b) Books containing one or several chapters on TLC
   c) Books containing frequent TLC information spread over several chapters of other information
2. **Fundamentals, theory and general**
   a) General b) Thermodynamics and theoretical relationship
   c) Relationship between structure and chrom. behaviour
   d) Measurement of physico-chemical and related values
   e) Optimization of solvent systems
   f) Validation of methods
3. **General techniques** (unless they are restricted to the application within one or two classification sections)
   a) New apparatus/techniques for sample preparation
   b) Separation material
   c) New apparatus for sample application/dosage
   d) New apparatus/techniques for chromatogram development
   e) New apparatus/techniques for pre- or post-chromatographic derivatization
   f) New apparatus/techniques for quantitative evaluation
   g) New apparatus/techniques for other TLC steps (distinguished from section 4)
4. **Special techniques**
   a) Automation of sample preparation/application
   b) Automation of complex chromatogram developing techniques
   c) Automation, computer application in quantitative chromatogram evaluation
   d) Combination of TLC with other chromatographic techniques
   e) Combination of TLC with other (non-chromatographic) techniques...MS, IR...etc.
5. **Hydrocarbons and halogen derivatives**
   a) Aliphatic hydrocarbons
   b) Cyclic hydrocarbons
   c) Halogen derivatives
   d) Complex hydrocarbon mixtures
6. **Alcohols**
7. **Phenols**
8. **Substances containing heterocyclic oxygen**
   a) Flavonoids
   b) Other compounds with heterocyclic oxygen
9. **Oxo compounds, ethers and epoxides**
10. **Carbohydrates**
    a) Mono- and oligosaccharides, structural studies
    b) Polysaccharides, mucopolysaccharides, lipopolysaccharides
11. **Organic acids and lipids**
    a) Organic acids and simple esters
    b) Prostaglandins
    c) Lipids and their constituents
    d) Lipoproteins and their constituents
    e) Glycosphingolipids (gangliosides, sulfatides, neutral glycosphingolipids)
12. **Organic peroxides**
13. **Steroids**
    a) Pregnane and androstane derivatives
    b) Estrogens
    c) Sterols
    d) Bile acids and alcohols
    e) Ecdysones and other insect steroid hormones
14. **Steroid glycosides, saponins and other terpenoid glycosides**
15. **Terpenes and other volatile plant ingredients**
    a) Terpenes
    b) Essential oils
16. **Nitro and nitroso compounds**
17. **Amines, amides and related nitrogen compounds**
    a) Amines and polyamines
    b) Catecholamines and their metabolites
    c) Amino derivatives and amides (excluding peptides)
18. **Amino acids and peptides, chemical structure of proteins**
    a) Amino acids and their derivatives
    b) Peptides and peptidic proteinous hormones
19. **Proteins**
20. **Enzymes**
21. **Purines, pyrimidines, nucleic acids and their constituents**
    a) Purines, pyrimidines, nucleosides, nucleotides
    b) Nucleic acids, RNA, DNA
22. **Alkaloids**
23. **Other substances containing heterocyclic nitrogen**
    a) Porphyrins and other pyrroles
    b) Bile pigments
    c) Indole derivatives
    d) Pyridine derivatives
    e) other N-heterocyclic compounds
24. **Organic sulfur compounds**
25. **Organic phosphorus compounds** (other than phospholipids)
26. **Organometallic and related compounds**
    a) Organometallic compounds
    b) Boranes, silanes and related non-metallic compounds
    c) Coordination compounds
27. **Vitamins and various growth regulators** (non-peptidic)
28. **Antibiotics, Mycotoxins**
    a) Antibiotics
    b) Aflatoxins and other mycotoxins
29. **Pesticides and other agrochemicals**
    a) Chlorinated insecticides
    b) Phosphorus insecticides
    c) Carbamates
    d) Herbicides
    e) Fungicides
    f) Other types of pesticides and various agrochemicals
30. **Synthetic and natural dyes**
    a) Synthetic dyes
    b) Chloroplasts and other natural pigments
31. **Plastics and their intermediates**
32. **Pharmaceutical and biomedical applications**
    a) Synthetic drugs
    b) Pharmacokinetic studies
    c) Drug monitoring
    d) Toxicological applications
    e) Plant extracts, herbal and traditional medicines
    f) Clinico-chemical applications and profiling body fluids
33. **Inorganic substances**
    a) Cations
    b) Anions
34. **Radioactive and other isotopic compounds**
35. **Other technical products and complex mixtures**
    a) Surfactants
    b) Antioxidants and preservatives
    c) Various specific technical products
    d) Complex mixtures and non-identified compounds
36. **Thin-layer electrophoresis**
37. **Environmental analysis**
    a) General papers
    b) Air pollution
    c) Water pollution
    d) Soil pollution
38. **Chiral separations**

XX. (abstract number underlined) refers to HPTLC related publication or application using HPTLC materials

1. Reviews and books

104 001 Tara MCGLINCHEY*, P.A. RAFTER, Fiona REGAN, D. GILLIAN, P. MCMAHON (*Department of Agriculture, Fisheries & Food, Central Meat Control, Backweston Laboratory Complex, Youngs Cross, Celbridge, Co., Kildare, Ireland): A review of analytical methods for the determination of aminoglycoside and macrolide residues in food matrices. Anal. Chim. Acta 624 (1), 1-15 (2008). Aminoglycosides and macrolides are important antibiotics for veterinary medicine and are widely used in the treatment of bacterial disease, and as feed additives for growth promotion. As a result the European commission set strict criteria for monitoring residues and requires testing for low levels of aminoglycosides and macrolides in foods. Therefore the development of fast, reliable, and sensitive methods for the extraction and subsequent analysis of these antibiotics is of great interest. The review discusses analytical methods for both extraction and determination of antibiotics in various food matrices focusing on the last 10 years. Extraction and clean-up methods such as deproteinization and solid-phase extraction are described, and various screening methods including TLC, EI, CE, microbiological assays, and LC combined with MS are reviewed. agricultural, food, analysis, HPTLC,

review 1, 28

2. Fundamentals, theory and general

104 002 Tatjana DJAKOVIC-SEKULIC*, Nada PERISIC-JANJIC, Evgenija DJURENDI (*Department of Chemistry, Faculty of Sciences, University of Novi Sad, Trg Dositeja Obradovi a 3, 21000 Novi Sad, Serbia): Retention data from reverse-phase high-performance thin-layer chromatography in characterization of some bis-salicylic acid derivatives. Biomed. Chromatogr. 23 (8), 881-887 (2009). HPTLC investigation of salicylic acid derivatives on RP-phase with various methanol - water and dioxane - water binary mixtures in order to establish relationships between chromatographic data and selected physico-chemical parameters that are related to ADME (absorption, distribution, metabolism and elimination). A linear correlation between RM values and the volume fraction of mobile phase modifier was observed. The obtained RM0 values were correlated with lipophilicity, solubility, human intestinal absorption, plasma-protein binding, and blood-brain barrier data.

HPTLC, QSRR, lipophilicity 2

104 003 L. KOMSTA*, K. SZTANKE, P. MACZKA, M. UCHEREK, R. SKIBINSKI, A. GUMIENICZEK (*Department of Medicinal Chemistry, Faculty of Pharmacy, Medical University of Lublin, Jaczewskiego 4, 20-090 Lublin, Poland; lukasz.komsta@am.lublin.pl): Determination of the lipophilicity of twenty-seven imidazo[2,1-c][1,2,4]triazine derivatives with strong biological activity by reversed-phase TLC. Comparison with results obtained by use of computational algorithms J. Planar Chromatogr. 22, 327-331 (2009) TLC of 27 novel imidazo[2,1-c][1,2,4]triazine derivatives together with 12 compounds with known literature log P values (as reference calibration data) on RP-18 with methanol - water binary mobile phases containing different proportions of methanol in horizontal chambers without chamber saturation. Detection under UV 254 nm. Using principal-component analysis the obtained results were compared with results from nine computational methods.

pharmaceutical research, qualitative identification 2c

104 004 T. SAMUEL*, A. MANIKANDAN, A. SINGH (*CGPSG College of Pharmacy, Peelamedu, Coimbatore, India): Standardisation of herbal medicine by high-performance thin-layer chromatography. Abstract No. 9923, IHCB (2009). HPTLC of several herbal formulations (tablets extracted with methanol) on silica gel with toluene - ethyl acetate 19:1. The fingerprint method was suitable for correct identification and for routine quality control of the herbal extracts.

pharmaceutical research quality control, herbal, HPTLC 2a

104 005 M. WAKSMUNDZKA-HAJNOS, D. MATOSIUK, A. PETRUCZYNIK, U. KIJKOWSKA-MURAK* (*Medical University of Lublin Department of Inorganic Chemistry 20-081 Lublin Poland): Determination of the lipophilicity of selected isoquinoline alkaloids by RP-TLC. Acta Chromatographica 24 (4), 563-573 (2008). TLC of nine alkaloids on RP-18 with mixtures of

aqueous acetone or aqueous dioxane with various mobile phase additives (ammonia, diethylamine, or tetrabutylammonium chloride) in order to suppress ionization of the alkaloids and/or reduce ionic interactions with surface silanol groups. Ion-pair TLC on RP-18 with aqueous acetone mixtures and various mobile phase additives (pentane sulfonic acid, octane sulfonic acid, or di-(2-ethylhexyl)orthophosphoric acid). Investigation of relationships between RM values and modifier concentration using a linear semilogarithmic equation for experimental data to calculate lipophilicity values RMW (RM for pure water), the slope, and the intercept with the x -axis. Comparison with retention data of standards with known lipophilicity (log P).

doping 2d, 22

3. General techniques

104 006 V.G. BEREZKIN*, N.Y. KULAKOVA, S.S. KHREBTOVA (*A. V. Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, Leninsky pr. 29, Moscow, 119991, Russia, berezkin@ips.ac.ru): Three-dimensional thin-layer chromatography on plates with open and closed adsorption layers. J. Planar Chromatogr. 22, 313-319 (2009). Presentation of two different methods of three-dimensional TLC using plates with open and sealed (closed) adsorption layers. In the suggested method the components of the test dye mixture initially migrate in the first direction, then in the second direction (different from the first) and in the third direction (different from first and second). TLC of dye mixture 1 (crystal violet, xylen cyanol, neutral blue, bromothymol dark blue, methanyl yellow, acridine orange, indophenol, ariabel red, Sudan blue II, Sudan IV, dimethylaminoazobenzene) with ethanol - acetic acid 10:1 (1D), acetone (2D), and toluene (3D) and of mixture 2 (dark violet, bright orange, yellow, dark red, violet) on silica gel with tetrahydrofurane - benzene 9:1 (1D), dichloromethane - benzene 3:1 (2D), and toluene (3D).

comparison of methods 3d

104 007 X. LIU*, T. KUBO, H. DIAO, J. BENJAMAS, T. YONEMICHI, N. NISHI (*College of Materials and Textiles, Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University, Xiasha Higher Education Zone, Hangzhou 310018, China): DNA/polyvinyl alcohol interpenetrating polymer network as stationary phase for thin-layer chromatography. Anal. Biochem. 393 (1), 67-72 (2009). A DNA/polyvinyl alcohol interpenetrating polymer network was produced by cross-linking polyvinyl alcohol with glutaraldehyde and subsequent cross-linking of DNA by UV irradiation. The polymer was then used to coat the surface of porous silica particles for TLC. Three typical DNA-binding compounds and eight amino acid enantiomers were used as model chemicals to investigate the chromatographic behavior of the modified TLC phase. Both classes of chemicals provided high separation efficiency. DNA-modified TLC phases could be used for various application fields, including efficacy evaluation of a medicine, toxicity assessment of a pollutant at the molecular level, as well as separation of enantiomers of dyes, amino acids, peptides, proteins, nucleotides, and drugs.

stationary phase 3b

104 008 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. Bio. Chromatogr. 21(1), 94-100 (2008). The method for detection of tyrosinase inhibiting substances involves spraying of the TLC layer with tyrosinase and l-tyrosine solutions successively. Positive results are detected as white spots against a brownish-purple background. The method is suitable either as a quick screening method for tyrosinase inhibitor detection or as a guiding procedure for an isolation of tyrosinase inhibitors from mixtures or natural product extracts. The technique is sensitive enough for results in the presence of 6 ng/zone glabridin.

review, postchromatographic derivatization 3d

4. Special techniques

104 009 P. ABU-RABIE*, N. SPOONER (*PreClinical Development Drug Metabolism and Pharmacokinetics, GlaxoSmithKline Research and Development, Park Road, Ware, Hertfordshire, SG12

ODP, United Kingdom; Paul.2.Abu-Rabie@gsk.com): Direct quantitative bioanalysis of drugs in dried blood spot samples using a thin-layer chromatography mass spectrometer interface. Anal. Chem. 81, 10275-10284 (2009). Direct quantitative bioanalysis of drugs from dried blood spot samples using a TLC-MS interface with or without HPLC separation. The method gave acceptable sensitivity, linearity, accuracy, and precision data for bioanalytical validations. The direct elution technique was shown to increase assay sensitivity for a range of analytes. Investigations were performed to optimize extraction time, minimize sample-to-sample carry-over, and compare chromatographic performance. On the basis of this preliminary assessment, it has been demonstrated that this TLC-MS interface has the potential to be an effective tool for the direct analysis of drugs in dried blood samples at physiologically relevant concentrations.

clinical chemistry research 4e

104 010 S.-C. CHENG (Cheng Sy-Chyi), M.Z. HUANG (Huang Min-Zong), J. SHIEA* (Shiea Jentaie) (*National Sun Yat-Sen University-Kaohsiung Medical University Joint Research Center, Kaohsiung, 804, Taiwan; jetea@mail.nsysu.edu-tw): Thin-layer chromatography/laser-induced acoustic desorption/electrospray ionization mass spectrometry. Anal. Chem. 81, 9274-9281 (2009). TLC of dye standards (FD&C Green No. 3, FD&C Red No. 3, eriochromcyanin R), drug standards (3,4-methylene-dioxy-N-methamphetamine, lysergic acid diethylamide, flunitrazepam), and rosemary essential oil on 1) RP-18 with 65 % acetone containing 1.5 % formic acid and 2) on silica gel with chloroform - methanol 9:1. Rosemary essential oil was also separated on silica gel with toluene - ethyl acetate 9:1. Detection under UV 254 nm and in daylight. The combination of laser-induced acoustic desorption and electrospray ionization mass spectrometry (LIAD/ESI/MS) can be used to rapidly characterize chemical compounds separated on a TLC plate.

qualitative identification 4e

104 011 N. GOTO-INOUE*, T. HAYASAKA, T. TAKI, Tania VALDES GONZALEZ, M. SETOU (*Department of Molecular Anatomy, Hamamatsu University School of Medicine, Hamamatsu, Shizuoka 431-3192, Japan): A new lipidomics approach by thin-layer chromatography-blot-matrix-assisted laser desorption/ionization imaging mass spectrometry for analyzing detailed patterns of phospholipid molecular species. J. Chromatogr. A 1216 (42), 7096-7101 (2009). Presentation of a TLC-blot-MALDI-IMS method which combines TLC and IMS (imaging mass spectrometry) for use in lipidomics. In comparison with common staining methods the method allows highly sensitive detecion of whole lipids and individual molecular species. Linearity for all lipids ranged approximately over one order of magnitude. Precision (*% RSD*) was <16 %. The TLC step allows precise separation of complex lipid mixtures into individual lipid classes before MS analysis is performed.

qualitative identification 4e

104 012 E. HARRY, J. REYNOLDS, A. BRISTOW, I. WILSON, C. CREASER* (*Centre for Analytical Science, Department of Chemistry, Loughborough University, Loughborough LE11 3TU, UK, c.s.creaser@lboro.ac.uk): Direct analysis of pharmaceutical formulations from non-bonded reverse-phase thin-layer chromatography plates by desorption electrospray ionisation ion mobility mass spectrometry. Rapid Commun. Mass Spectrom. 23, 2597-2604 (2009). RP-TLC of pharmaceutical formulations containing paracetamol, ephedrine, codeine, and caffeine on hydrocarbon-impregnated silica with methanol - water 1:1. Detection by desorption electrospray ionization (DESI) combined with ion mobility mass spectrometry (IM-MS). The limit of detection was 9, 16, 34, 239 and 225 µg/cm2 for codeine, caffeine, ephedrine and paracetamol, respectively.

pharmaceutical research, quality control, qualitative identification 4e

104 013 L. KOMSTA (Department of Medicinal Chemistry, Medical University of Lublin, Jaczewskiego 4, 20-090 Lublin, Poland): A comparative study on several algorithms for denoising of thin-layer densitograms. Anal. Chim. Acta 641 (1-2), 52-58 (2009). Comparison of classical filtering techniques (Savitzky-Golay, Adaptive Degree Polynomial Filter, Fourier denoising, Butterworth and Chebyshev IIR filters) and wavelet shrinkage (31 mother wavelets, 3 thresholding techniques and

11 decomposition levels) with the original noisy signal and a reference signal which was denoised experimentally by averaging 64 measurements. The best similarity to the reference signal was obtained with filters, however the signal was slightly oversmoothed. The wavelet shrinkage method gave less denoised signals. There was a significant influence of the thresholding technique and decomposition level, and best conditions were at level 2 or 3 and soft thresholding), whereas changing of the mother wavelet almost did not change the result. The presented results can be used as general recommendations for denoising densitometric fingerprints before applying further chemometric algorithms. The best choices were: Savitzky-Golay filter of appropriate window width (optimized against autocorrelation) or wavelet shrinkage with Haar wavelet, soft thresholding and high decomposition level.

HPTLC, quantitative analysis, qualitative identification, densitometry 4c

104 014 F. SCHULTE, J. MAEDER, L. W. KROH, U. PANNE, Janina KNEIPP* (*Chemistry Department, Humboldt Universität zu Berlin, Brook-Taylor-Strasse 2, 12489 Berlin, Germany; janina.kneipp@chemie.hu-berlin.de): Characterization of pollen carotenoids with in situ and high-performance thin-layer chromatography supported resonant Raman spectroscopy. Anal. Chem. 81, 8426-8433 (2009). HPTLC of zeaxanthin, cryptoxanthin, beta-carotene, lutein and pollen extracts on silica gel with tetrahydofuran - methylene chloride - *n*-hexane by automated multiple development. Quantitative determination by absorbance measurement at 425 nm, which has to be accomplished within 5 min after development due to the fast bleaching of the carotenoid color. The analysis of carotenoids in pollen extracts was confirmed by resonance Raman data measured directly on the HPTLC plates.

HPTLC, AMD, densitometry, quantitative analysis, biochemical research 4e, 30b

104 015 M. SHARIATGORJI, Z. SPACIL, G. MADDALO, L. CARDENAS, L. ILAG* (*Department of Analytical Chemistry, Stockholm University, 106 91 Stockholm, Sweden, Leopold.ilag@anchem.su.se): Matrix-free thin layer chromatography/laser desorption ionization mass spectrometry for facile separation and identification of medicinal alkaloids. Rapid Commun. Mass Spectrom. 23, 3655-3660 (2009). TLC of berberine and palmatine in roots of Berberis barandana on silica gel with butanol - acetic acid - water 14:3:4. Detection under UV 366 nm. Bands were cut out for further analysis by laser desorption ionization mass spectrometry. The hR_F value of berberine and palmatine was 56 and 46, respectively.

herbal, qualitative identification 4e

104 016 C. SIMOES-PIRES, B. HMICHA, A. MARSTON, K. HOSTETTMANN* (*Laboratory of Pharmacognosy and Phytochemistry, University of Geneva, 1211 Geneva 4, Switzerland, kurt.hostettmann@unige.ch): A TLC bioautographic method for the detection of alpha- and beta-glucosidase inhibitors in plant extracts. Phytochem. Anal. 20, 511-515 (2009). Bioautography of alpha-D-glucosidase (1) and beta-D-glucosidase (2) in buffer solution (sodium acetate 4.1 % in water pH=7.5) sprayed onto a silica gel plate. Incubation at room temperature for 60 min for (1) and at 37 °C for 20 min for (2). For detection of the active enzyme, solutions of 2-naphthyl-alpha-D-glucopyranoside or 2-naphthyl-beta-D-glucopyranoside and Fast Blue salt were mixed at a ratio of 1:1 for (1) or 1:4 for (2), and sprayed onto the plate to give a purple background coloration after 2-5 min. Methanol extracts of the aerial parts of Tussilago farfara and Urtica dioica were tested as enzyme inhibitors and visualized as white spots on the TLC plates.

pharmaceutical research, herbal, qualitative identification, AMD, bioautography 4e

104 017 G. STUBIGER, E. PITTENAUER, O. BELGACEM, P. REHULKA, K. WIDHALM, G. ALLMAIER* (*Institute of Chemical Technologies and Analytics, Vienna University of Technology, 1060 Vienna, Austria, guenter.allmaier@tuwien.ac.at): Analysis of human plasma lipids and soybean lecithin by means of high-performance thin-layer chromatography and matrix-assisted laser desorption/ionization mass spectrometry. Rapid Commun. Mass Spectrom. 23, 2711-2723 (2009). HPTLC in combination with matrix-assisted laser desorption/ionization mass spectrometry (MALDI-MS) was used for the analysis of complex lipid mixtures. For the separation of

lipids one-dimensional HPTLC on silica gel aluminum foil was used with a two-phase mobile phase. The combination with MALDI-MS allowed the identification of 70 distinct lipid species and the analysis of even minor lipid classes from only very small volumes of human plasma (50 µL).

pharmaceutical research, clinical chemistry research, food analysis, HPTLC, quantitative analysis 4e

5. Hydrocarbons and halogen derivatives

104 018 I. BELAI*, G. OROS, B. BORDAS (*Plant Protection Institute, Hungarian Academy of Sciences, 1525 Budapest, P.O. Box 102, Hungary; ibel@nki.hu): Quantitative structure-retention relationship and 3D molecular modeling studies in the unusual chromatographic behavior of triphenylmethane derivatives in RPTLC systems. J. Planar Chromatogr. 22, 255-263 (2009). TLC of 25 triphenylmethane derivatives using paraffin oil-coated silica gel and acetone-water mixtures, in which the acetone content varied between 40 and 70 % in increments of 10 %. Dual retention behavior is observed for triphenylmethane derivatives in reversed phase HPTLC when the composition of acetone-water mobile phase is varied. The physicochemical and molecular properties of triphenylmethane derivatives lead to an unusual retention behavior, which was investigatd by traditional quantitative structure-retention relationship modeling and by 3D molecular modeling. Lipophilicity was found to be the most important molecular property governing the retention of triphenylmethane derivatives.

qualitative identification 5b

8. Substances containing heterocyclic oxygen

104 019 H.M. KURTBAY, I. KAYNAK, S. S. BOZKURT, M. MERDIVAN* (*Department of Chemistry, Dokuz Eylul University, Faculty of Science and Arts, 35160 Izmir, Turkey; melek.merdivan@deu.edu.tr): Densitometric HPTLC analysis of the 5-hydroxymethylfurfural content of Turkish fruit wines and vinegars. J. Planar Chromatogr. 22, 363-366 (2009). HPTLC of 5-hydroxymethylfurfural in seven Turkish fruit wines and three Turkish vinegars on silca gel with toluene - ethyl acetate - 90 % formic acid 6:3:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 286 nm. The limit of detection and quantification was 0.05 and 0.13 ng/mL, respectively.

food analysis, toxicology, HPTLC, densitometry, quantitative analysis 8b

9. Oxo compounds, ethers and epoxides

104 020 Petra JAZBEC*, A. SMIDOVNIK, M. PUKLAVEC, M. KRIZMAN, J. SRIBAR, L. MILIVOJEVIC, M. PROSEK (*Department of Food Chemistry, National Intitute of Chemistry, Ljubljana, Slovenia; Petra.jazbec@ki.si): HPTLC and HPLC-MS quantification of coenzyme Q10 and cholesterol in fractionated chicken-breast tissue. J. Planar Chromatogr. 22, 395-398 (2009). HPTLC of coenzyme Q10, cholesterol and biological extracts on silica gel with petroleum ether - diethyl ether - acetic acid 85:15:1. Detection by dipping into 5 % phosphomolybdic acid in ethanol for 10 s followed by heating for 10 min at 110 °C. Quantitative evaluation in visible light.

food analysis, HPTLC, quantitative analysis, densitometry, biochemical research 9, 20

10. Carbohydrates

104 021 T. BERNARDI, Elena TAMBURINI* (*Department of Chemistry, University of Ferrara, Via L. Borsari 46, 44100 Ferrara, Italy; tme@unife.it): An HPTLC-AMD method for understanding the metabolic behavior of microorganisms in the presence of mixed carbon sources. The case of Bifidobacterium adolescentis MB 239. J. Planar Chromatogr. 22, 321-325 (2009). HPTLC of glucose, fructose, galactose, lactose, raffinose, 1-kestose, nystose, and fructosyl-nystose on diol phase by automated multiple development with acetonitrile - acetone - water and acetonitrile - water. Preliminary isocratic developments were performed at 22-25 °C at 65-75 % relative humidity in a twin trough chamber. Detection by vaporization with 37 % hydochloric acid vapor for 30 min followed by dipping in 4-aminobenzoic acid. Quantitative determination by fluorescence measurement at 313 nm.

HPTLC, densitometry, quantitative analysis, AMD, biochemical research 10a

104 022 J. STROKA*, I. DONCHEVA, B. SPANGENBERG (*European Commission Joint Research Centre, Institute for Reference Materials and Measurements, Food Safety and Quality Unit, Retieseweg 111, 2440 Geel, Belgium; joerg.stroka@ec.europa.eu): Determination of sucralose in soft drinks by high-performance thin-layer chromatography: Interlaboratory study. J. AOAC Int. 92, 1153-1159 (2009). HPTLC of sucralose on amino phase with acetonitrile - water 4:1 in a horizontal or standard development chamber without chamber saturation. For detection the plate was heated at 190 °C for 20 min, either in a drying oven or on a temperature-controlled heating plate. Quantitative determination by absorbance and fluorescence measurement at 254 nm. The results of the interlaboratory comparison show good precision characteristics. The fluorescence measurements of the sucralose derivatives indicated better method performance, compared with absorbance measurements in the UV.

food analysis, quality control, HPTLC, quantitative analysis, densitometry 10a

11. Organic acids and lipids

104 023 Ritu ARORA*, B. SINGH, R. SINGH, C. KATIYAR (*Guru Nanak Dev University, Amritsar, Punjab, India): Validated HPTLC method for the determination of cinnamic acid in crude plant materials, herbal extracts and pharmaceutical dosage forms containing Cinnamomum cassia. 60th Indian Pharmaceutical Congress PA-214 (2008). HPTLC of cinnamic acid (in plant raw material, herbal extracts and pharmaceutical dosage forms) on silica gel with chloroform - methanol 4:1 in a saturated twin trough chamber at room temperature (25 °C). Quantitative determination by absorbance measurement at 277 nm. The hR_F value for cinnamic acid was 50. The method was linear in the range of 700-1400 ng/spot.

pharmaceutical research, herbal, HPTLC, densitometry, quantitative analysis 11a

104 024 F. HASAN*, R. KHAR, F. AHMAD, M. ALI, M. RAZA (*Dept. of Pharmaceutics, Jamia Hamdard, Hamdard Nagar, New Delhi, India): Development of novel high-performance thin-layer chromatography method for estimation of lipid in egg oil. Abstract No. F-282, 61st (2009). HPTLC of lipids on silica gel in a saturated twin trough chamber with carbon tetrachloride - methanol - acetic acid 270:30:11 at room temperature (25 °C). Quantitative determination by fluorescence measurement at 366 nm. The hR_F value of cholesterol was 35. The calibration curve showed good linear relationship with r^2=0.999 in the range of 100-600 ng/spot (via peak area).

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

104 025 P. HAZAM*, D. SARKAR, B. DEY, S. DAS (*Himalayan Pharmacy Institute, Dept. of Pharmacognosy, Majhotar, Sikkim, India: A high-performance thin-layer chromatographic (HPTLC) method for the estimation of rosmarinic acid from Salvia officinalis (sage). 60th Indian Pharmaceutical Congress PG-256 (2008). HPTLC of rosmarinic acid in Salvia officinalis on silica gel with toluene - ethyl acetate - formic acid 5:4:1. Quantitative determination by absorbance measurement at 328 nm. Linearity was 0.1-1.0 ng/mL, recovery was 99.4 %. The method was found suitable for routine quality control of the Salvia officinalis raw material.

herbal, environmental, densitometry, HPTLC, quantitative analysis 11a

104 026 Y. KAWAI*, M. MIYOSHI, J. MOON, J. TERAO (*Department of Food Science, Graduate School of Nutrition and Biosciences, The University of Tokushima, Tokushima 770-8503, Japan): Detection of cholesteryl ester hydroperoxide isomers using gas chromatography-mass spectrometry combined with thin-layer chromatography blotting. Anal. Biochem. 360 (1), 130-137 (2007). Determination of cholesteryl ester hydroperoxides, especially cholesteryl linoleate hydroperoxide isomers, by combination of TLC blotting with diphenyl-1-pyrenylphosphine fluorescent detection (DPPP-TLC blotting) and GC-electron ionization-MS (GC-EI-MS). After DPPP-TLC blotting of cholesteryl ester hydroperoxides fluorescent zones were obtianed which were extracted and derivatized by hydrogenation, transmethylation, and trimethylsilylation. The resulting methyl ester/trimethylsilylether derivatives of hydroxyoctadecenoic acid were then analyzed by GC-EI-MS using selected ion monitoring of isomer-specific fragment ions originating from the alpha-cleavage of the trimethylsilyloxyl group.

agricultural, clinical routine analysis, postchromatographic derivatization, qualitative identification, quantitative analysis 11

104 027 H. LI (Li Haixing)*, T. QIU (Qiu Ting), Y. CAO (Cao Yusheng), J. YANG (Yang Jiyan), Z. HUANG (Huang Zhibing) (*Sino-German Joint Research Institute, Nanchang University, Nanchang 330047, China): Pre-staining paper chromatography method for quantification of gamma-aminobutyric acid. J. Chromatogr. A 1216 (25), 5057-5060 (2009). Paper chromatography of gamma-aminobutyric acid. The method consists of application, separation and detection and is clean, rapid, inexpensive and reproducible compared to the routine paper chromatography. The derivatization procedure with ninhydrin reagent was optimized regarding reagent concentration, derivatization temperature and time and Cu2+ concentration. Quantification of gamma-aminobutyric acid by combination of with vis spectrophotometry. The limit of detection was 0.05 mg/mL and the linear range was from 0.5 to 20.0 mg/mL. The determination coefficient was $r^2 = 0.998$. The method was accurate (*%RSD* < 2.6 %), and recoveries were 102.7-103.9 %.

quality control, traditional medicine, pharmaceutical research, quantitative analysis, qualitative identification, paper chromatography 11a

104 028 N. MANJU*, A. BINDU, N. ALEYKUTTY, J. SAJAN (*Department of Pharmaceutical Sciences, Cheruvandoor Campus, Ettumanoor, Kottayam, Kerala, India): Development of HPTLC method for quantification of gallic acid from the fruits of Phyllanthus emblica L. 60th Indian Pharmaceutical Congress PG-265 (2008). HPTLC of gallic acid in the ethyl acetate fraction of fruits of Phyllanthus emblica on silica gel with toluene - ethyl acetate - formic acid - methanol 30:30:8:2. Detection and quantitative determination of gallic acid by absorbance measurement at 280 nm. The proposed HPTLC method provided a good resolution of gallic acid from other constituents present in the ethyl acetate fraction of fruits of Phyllanthus emblica and can be used for the quantification of gallic acid.

herbal, HPTLC, densitometry, quantitative analysis 11a

104 029 D. MUKHERJEE*, T. BARMAN, S. RAJA, P. MUKHERJEE (*School of Natural Product Studies, Jadavpur University, Kolkata 700032, India): Estimation of betulinic acid in Nelumbo nucifera (Nymphaceae) rhizome and seed extract by validated HPTLC method. Abstract No. 9192, IHCB (2009). HPTLC of betulinic acid in hydro alcoholic extracts of Nelumbo nucifera rhizome and seed, on silica gel with chloroform - methanol - formic acid 49:1:1. Detection by spraying with anisaldehyde reagent. Quantitative determination by absorbance measurement at 420 nm. The method was linear in the range of 2-10 ng/spot, recovery was 98.1-98.4 %.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, quantitative analysis, postchromatographic derivatization 11a

104 030 B. NIMAVAT*, D. MOVALIA, S. MISHRA, H. TANK (*S. J. Thakkar Pharmacy College, Saurashtra University, Rajkot,Gujarat, India): Development of validated HPTLC method for quantitative estimation of oleanolic acid as marker in total methanolic extract of fruits of Randia dumetorum lam. 60th Indian Pharmaceutical Congress PA-217 (2008). HPTLC of oleanolic acid in total methanolic extract of fruits of Randia dumetorum lam. on silica gel with toluene - ethyl acetate - glacial acetic acid 70:30:1 in a twin trough chamber saturated for 10 min. Detection by treatment with 10 % sulphuric acid in methanol, followed by heating at 110 °C and immediate densitometric evaluation. Quantitative determination by absorbance measurement at 540 nm. The method was linear in the range of 50-500 ng/spot. Recovery was in the range of 99.4-100.8 %.

pharmaceutical research, traditional medicine, herbal, HPTLC, quantitative analysis 11a

104 031 P. PATEL*, R. SHAH, S. PATEL, Unnati SHAH (*Pharmanza Herbal Pvt. Ltd., Plot No. 214, Kania 388435, Gujarat, India; and Lachoo Memorial College of Science & Technology, Jodhpur, India): Method development and validation for the estimation of (-)hydroxy citric acid in fruits of Garcinia gummigutta D. by HPTLC. Abstract No. 9191, IHCB (2009). HPTLC of (-)hydroxy

citric acid (the main constituent of Garcinia gummigutta fruits) on silica gel with *n*-propanol - water - glacial acetic acid 50:50:1. Quantitative determination by absorbance measurement at 210 nm. The hR_F value was 46. The method was linear in the range of 100-1000 ng/spot, recovery was 99.8-100.9 %.

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

104 032 B. RAJ*, Salma KHANAM, L. JOSEPHINE (*Dept. of Pharmacognosy, Dayanand Sagar College of Pharmacy, Bangalore 560078, India, bincyraj@yahoo.com): HPTLC method for estimation of ursolic acid in Ocimum sanctum extract. Asian J. of Chem. 21(9), 6999-7004 (2009). HPTLC of ursolic acid in methanolic and aqueous extracts of Ocimum sanctum (Tulsi) on silica gel with toluene - ethyl acetate - acetic acid 55:45:2 with chamber saturation. Detection by treatment with Liebermann Burchard‘s reagent. Quantitative determination by absorbance measurement at 550 nm. The method was linear in the range of 100-400 ng/band. The amount of ursolic acid in aqueous and methanolic extracts was in the range of 0.2 and 0.4 %. Samples collected from different geographical regions were compared, samples from Kerala contained the highest levels of ursolic acid.

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

104 033 B. RAJ*, Salma KHANAM, S. JOHN, J. JENITA, Ayesha SIDDIQUA (*Dept. of Pharmacognosy, Dayananda Sagar College of Pharmacy, Kumaraswamy Layout, Bangalore 560078, India): Comparative HPTLC analysis of oleanolic acid in Ocimum sanctum collected from different geographical sources. Abstract No. 9226, IHCB (2009). HPTLC of oleanolic acid (in extracts of Ocimum sanctum collected from different geographical sources) on silica gel with toluene - ethyl acetate - glacial acetic acid 55:45:2. Detection by spraying with Liebermann-Burchard‘s reagent. Quantitative determination by absorbance measurement at 600 nm. Linearity was in the range of 1-4 µg, recovery was 100.4-102.1 %. Hydro alcoholic extracts of the plant contained high amounts of oleanolic acid, maximum contents were found in plants collected in the Kerala region.

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

104 034 M. SAJEWICZ, D. KRONENBACH, M. GONTARSKA, M. WRÓBEL, R. PIETKA, Teresa KOWALSKA* (*Institute of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland; kowalska@us.edu.pl): TLC in a search for structural limitations of spontaneous oscillatory in-vitro chiral conversion. Alpha-hydroxybutyric and mandelic acids. J. Planar Chromatogr. 22, 241-248 (2009). TLC of alpha-hydroxybutyric acids and mandelic acid on silica gel, prewashed with methanol - water 9:1 and impregnated by dipping for 2 s in 0.05 g/L aqueous copper acetate solution, with dioxane - water 9:1 at 22 +/- 2°C. Quantitative determination by absorbance measurement during 16 days at 326 nm. Spontaneous oscillatory in-vitro chiral conversion was observed for alpha-substituted propionic acids as well as chiral carboxylic acids with two and four carbon atoms.

qualitative identification, densitometry 11a

13. Steroids

104 035 R. XIA (Xia Rui)*, SH. DONG (Dong Shuying), B. CHE (Che Baoquan) (*Beijing Municip. Inst. Drug Cont., Beijing 100035, China): (Rapid identification of glucocorticoid in Chinese traditional medicinal formulations by thin-layer chromatography) (Chinese). Chinese J. Pharm. Anal. 28 (3), 470-471 (2008). TLC of glucocorticoid in TCM formulations extracted with chloroform - methanol 9:1 on silica gel with dichloromethane - diethyl ether - methanol - water 385:60:15:2. Detection by spraying with a solution of tetrazolium blue chloride. Successful separation and identification of five glucocorticoids.

doping, pharmaceutical research, traditional medicine, quality control, quantitative analysis, qualitative identification 13

104 036 B. YUAN (Yuan Baoqiang)*, L. Yang (Yang Lahu) (*Fuzhou Municip. Inst. Drug Cont., Fuzhou, Fujian 350001, China): (Analysis of dexamethasone acetate and the related compounds in the tablets) (Chinese). Chinese J. Pharm. Anal. 27 (3), 412-413 (2007). TLC of dexamethasone acetate on silica gel with dichloromethane - methanol 9:1. Detection under UV 254 nm.

pharmaceutical research, quality control, qualitative identification, quantitative analysis 13

15. Terpenes and other volatile plant ingredients

104 037 B. CHENGAIAH*, B. PRATAP, M. ALAGUSUNDARAM, M. RUTHU, V. SAROVAR REDDY (*Annamacharya College of Pharmacy, Rajampet, Kadapa, A.P., India): Identification of terpenes in stem of Ocimum basilicum Linn by HPTLC technique. 60th Indian Pharmaceutical Congress PA-221 (2008). HPTLC of terpenes in stem of Ocimum basilicum on silica gel with toluene - ethyl acetate 19:1. Evaluation of 5 spots under UV 254 nm and under visible light after treatment with anisaldehyde reagent followed by heating at 100 °C for 5 min: borneol (yellow, hR_F 24), menthol (red, hR_F 27), eugenol (blue, hR_F 50), thymol (violet, hR_F 57), safrole (blue, hR_F 93).

traditional medicine, herbal, HPTLC, qualitative identification 15a

17. Amines, amides and related nitrogen compounds

104 038 A. ALPMANN, Gertrud MORLOCK* (*University of Hohenheim, Institute of Food Chemeistry, Garbenstrasse 28, 70599 Stuttgart, Germany; gmorlock@uni-hohenheim.de): Rapid and cost-effective determination of acrylamide in coffee by planar chromatography and fluorescence detection after derivatization with dansulfinic acid. J. AOAC Int. 92, 725-729 (2009). HPTLC of acrylamide extracted from coffee samples on silica gel with ethyl acetate - tert. butyl methyl ether 4:1 in a twin trough chamber or automatic developing chamber. Pre-chromatographic in situ derivatization of the extracts (applied as area) by overspraying with dansulfinic acid produced the fluorescent dansylpropanamide band. Quantitative determination by fluorescence measurement at 254/>400 nm. The limit of quantification was 48 µg/kg. The linearity over the whole procedure showed determination coefficients between 0.9995 and 0.9825 ($n = 6$). The within-run precision (*% RSD*, n = 6) of the chromatographic method was 3%. Commercial coffee samples analyzed showed acrylamide contents between 52 and 191 µg/kg, which was in correlation with amounts reported in publications.

food analysis, toxicology, quality control, HPTLC, quantitative analysis, densitometry 17c

18. Amino acids and peptides, chemical structure of proteins

104 039 J.D. VASTA, M. CICCHI, J. SHERMA*, B. FRIED (*Lafayette College, Department of Chemistry, Easton PA 18042-1782 USA): Evaluation of thin-layer chromatography systems for analysis of amino acids in complex mixtures. Acta Chromatographica 21 (1), 29-38 (2009). Evaluation of different TLC systems for analysis of 21 amino acids in biological tissues and fluids. Detection by derivatization with ninhydrin reagent, and determination of R_F values by slit-scanning densitometry. The five most suitable systems were cellulose and silica gel plates developed with either 2-butanol - pyridine - acetic acid - water 39:34:10:26 or 2-butanol - pyridine - 25 % ammonia - water 39:34:10:26, and ion exchange plates developed with citrate buffer of pH 3.3. Detection with ninhydrin allowed the identification of all amino acids except for leucine and isoleucine in complex mixtures. Quantification is possible as well if the amino acid of interest is well separated from adjacent components of the mixture. The method is illustrated with example chromatograms on cellulose HPTLC layer showing the identification and separation of amino acids in snail tissue samples.

clinical chemistry research, HPTLC, quantitative analysis, qualitative identification, densitometry 18a

104 040 M. VEGA*, M. ARANDA (*University of Concepcion, Faculty of Pharmacy, Department of Food Science, Nutrition and Dietetic, Barrio Universitario s/n Casilla 237, PO 403-0249 Concepcion, Chile; mveha@udec.cl): Determination of available lysine by planar chromatography: A useful tool for protein quality evaluation in fish feed. J. AOAC Int. 92, 699-702 (2009). HPTLC of a dinitrophenyl-lysine derivative (produced by incubation with 1-fluoro-2,4-dinitrobenzene and hydolyzation with hydrochloric acid) on silica gel (prewashed with methanol) with *n*-pro-

panol - 25 % ammonia 7:3 in a twin trough chamber. Quantitative determination by absorbance measurement at 360 nm. The method was linear (r^2 = 0.9991) in the range from 12.5 to 125.0 ng/band. Repeatability *(%RSD)* and intermediate precision *(%RSD)* in matrix were 0.8 % and 3.6 %, respectively. Recoveries of spiked samples at two levels ranged from 72 to 85 % with *RSD* from 3 to 8%. This method is a reliable, high throughput and low cost analytical method for the salmon feed industry.

food analysis, quality control, HPTLC, densitometry, quantitative analysis 18a

20. Enzymes

104 020 Petra JAZBEC et al., see section 9

22. Alkaloids

104 041 K. DHALWAL*, V. SHINDE, K. MAHADIK (* Dept. of Pharmacognosy and Phytochemistry, Poona College of Pharmacy, Bharathi Vidyapeeth University, Erandwane, Pune 411038, India): Effective and sensitive methods for quantitative analysis of alkaloids in sida species by using HPLC and HPTLC. Abstract No. 9402, IHCB (2009). HPLC and HPTLC methods were developed for the simultaneous estimation of vasicine and vasicinone in Sida cordifolia and Sida acuta roots. HPTLC of vasicine and vasicinone on silica gel with ethyl acetate - methanol - ammonia 40:10:1. Quantitative determination by absorbance measurement at 300 nm. Linearity was 320-960 ng/spot (vasicine) and 80-400 ng/spot (vasicinone). Linearity by HPLC was 4-20 µg/mL. The HPTLC method was more suitable because of high throughput and low analysis time.

pharmaceutical research, quality control, herbal, HPTLC, densitometry, comparison of methods 22

104 042 Danuta RAJ*, A. KOKOTKIEWICZ, M. LUCZKIEWICZ (*Department of Pharmacognosy, Wroclaw Medical University, pl. Nankiera 1, 50-140 Wroclaw, Poland; dankaraj@wp.pl): Densitometric HPTLC analysis of indolizidine alkaloids in the herb and in-vitro culture of Securinega suffruticosa. J. Planar Chromatogr. 22, 371-376 (2009). HPTLC of indolizidine alkaloids and extracts on silica gel and RP-18 (both prewashed with chloroform-methanol 1:1) with 11 mobile phases in a horizontal chamber without chamber saturation. The best resolution was achieved on silica gel with chloroform - methanol 20:1. Detection under UV 254 nm and after spraying with Dragendorff reagent. Quantitative determination of securinine and allosecurinine by absorbance measurement at 254 nm. The limit of detection and quantification was 11 and 36 ng/zone for securinine and 12 and 41 ng/zone for allosecurinine, respectively.

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

104 043 H. WIEDENFELD*, G. HOESCH, E. ROEDER, TH. DINGERMANN (*Pharmazeutisches Institut der Universität, An der Immenburg 4, 53121 Bonn, Germany; wiedenfeld@uni-bonn.de): Lycopsamine and cumambrin B from Eupatorium maculatum. Pharmazie 64, 415-416 (2009). TLC of the pyrrolizidine alkaloid lycopsamine and the guaianolide cumambrin B on silica gel with dichloromethane - methanol - 25 % ammonia 85:14:1. Detection see A. R. Mattocks, Detection of pyrrolizidine alkaloids on thin-layer chromatograms, J. Chromatogr. 27, 505-508 (1967).

pharmaceutical research, herbal, qualitative identification 22

104 005 M. WAKSMUNDZKA-HAJNOS et al., see section 2

104 044 L. ZHANG (Zhang Lu)*, H. LI (Li Hongyu), Y. WEI (Wei Yuhui) (*State Key Lab of Minist. Educ. of Drought & Grassplot Zoology, Coll. Life Sci., Lanzhou Univ., Lanzhou 730000, China): (Analysis of the alkaloids in the seeds of Sophora moorcroftiana (Benth.) Baker by TLC and HPLC) (Chinese). Chinese J. Pharm. Anal. 28 (7), 1071-1074 (2008). TLC of the alkaloids matrine, oxymatrine, sophocarpine, sophoridine, cytosinethe in seed extracts of Sophora moorcroftiana on silica gel with chloroform - methanol - ammonia 50:6:1. Detection under UV 254 nm.

Comparison with reference standards showed that extracts contained matrine, oxymatrine, sophocarpine, sophoridine, and cytosine.

pharmaceutical research, traditional medicine, quality control, herbal, densitometry, quantitative analysis, qualitative identification 22

27. Vitamins and various growth regulators

104 045 A. MOHAMMAD*, A. ZEHRA (*Aligarh Muslim University, Analytical Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh, India): Specific separation of thiamine from hydrophilic vitamins with aqueous dioxane on precoated silica TLC plates. Acta Chromatographica 20 (4), 637-642 (2008). TLC of thiamine hydrochloride from riboflavin, nicotinic acid, calcium D-pantothenate, pyridoxine hydrochloride, cyanocobalamin, and ascorbic acid on silica gel with dioxane - water 1:1. Detection under UV light. Examination of the effect of impurities (metal cations and inorganic anions) on the chromatography of thiamine hydrochloride. The detection limit for thiamine hydrochloride was 90 ng/spot and *%RSD* of thiamine hydrochloride was 14.9 % (n = 5).

pharmaceutical research, quality control, food analysis, quantitative analysis, qualitative identification 27

104 046 A. MOHAMMAD*, A. ZEHRA (*Analytical Research Laboratory, Department of Applied Chemistry, Faculty of Engineering and Technology, Aligarh Muslim University, Aligarh 202002, India; mohammadali4u@rediffmail.com): Simultaneous separation and identification of cyanocobalamin, thiamine, and ascorbic acid on polyoxyethylene sorbitan monooleate-impregnated silica layers with water as mobile phase. J. Planar Chromatogr. 22, 429-433 (2009). TLC of the water-soluble vitamins cyanocobalamine, thiamine, and ascorbic acid on silica gel (impregnated with a 2 % solution of the non-ionic surfactant Tween 80) with double distilled water in glass jars saturated for 30 min at 30 +/- 2° C. Detection of all substances except folic acid and cyanocobalamin by exposure to iodine vapor and evaluation in daylight.

quality control, qualitative identification 27

104 047 M. SUE-CHU, S. KRISTENSEN, H.H. TONNESEN* (*University of Oslo, School of Pharmacy, Department of Pharmaceutics, P. O. Box 1068, Blindern, 0316 Oslo, Norway; h.h.tonnesen@farmasi.nio.no): Photoinduced color changes in two different qualities of riboflavin in the solid state and various tablet formulations. Photoreactivity of biologically active compounds. Pharmazie 64, 428-435 (2009). TLC of riboflavin and lumichrome (7,8-dimethylbenzo[g]pteridine-2,4-(1H,3H)-dione) on silica gel with acetic acid - acetone - methanol - benzene 1:1:4:14 Detection under visible light and UV 254 and 366 nm.

pharmaceutical research, food analysis, qualitative identification 27

28. Antibiotics, Mycotoxins

104 242 R. BHUSHAN et al., see section 38

104 048 U. HUBICKA, J. KRZEK*, H. WOLTYNSKA, B. STACHAZ (*Collegium Medicum of Jagiellonian University, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Krakow, Poland; jankrzek@cm-uj.krakow.pl): Simultaneous identification and quantitative determination of selected aminoglycoside antibiotics by thin-layer chromatography and densitometry. J. AOAC Int. 92, 1068-1075 (2009). TLC and HPTLC of six antibiotics (amikacin, gentamicin, kanamycin, neomycin, netilmicin, and tobramycin) in pharmaceutical preparations on silica gel with methanol - 25 % ammonia - chloroform 3:2:1. Detection by derivatization with 0.2 % ethanolic ninhydrin reagent. Quantitative determination by absorbance measurement at 500 nm. The limit of detection and quantification was 250 and 500 ng/zone for amikacin, 500 and 1000 ng/zone for kanamycin, 480 and 800 ng/zone for neomycin, 380 and 630 ng/zone for netilmycin, 480 and 800 ng/zone for tobramycin and 1000 and 1650 ng/zone for gentamicin. Precision was good with *%RSD* of 0.3-0.6 %.

quality control, pharmaceutical research, HPTLC, densitometry, quantitative analysis 28a

104 049 S. JOSHI*, A. SHARMA, M. S. M. RAWAT, C. DHIMAN (*Department of Chemistry, K. L. D. A. V. (P. G.) College, Roorkee-247667. India; shajoshi@yahoo.com): Development of conditions for rapid thin-layer chromatography of beta-lactam antibiotics. J. Planar Chromatogr. 22, 435-437 (2009). TLC of penicillins (benzylpenicillin, ampicillin, and amoxicillin) and cephalosporins (cephalexin, cefoperazone, ceftriaxone, cefixime, and cefadroxil) in powder extracts on silica gel (impregnated with 0.2 % ammonium chloride) with propanol - acetic acid 4:1 and butanol - acetic acid - water 4:1:2. Detection under UV light at 365 nm.

quality control, pharmaceutical research, qualitative identification 28a

104 001 Tara MCGLINCHEY et al., see section 1

104 050 P.N. RANJANE*, S.V. GANDHI, S.S. KADUKAR, K.G. BOTHARA, (*Department of Pharmaceutical Analysis, A.I.S.S.M.S. College of Pharmacy, Kennedy Road, Near R.T.O., Pune 411 001, India): HPTLC determination of cefuroxime axetil and ornidazole in combined tablet dosage form. J. Chromatogr. Sci. 48 (1), 26-28 (2010). HPTLC of cefuroxime axetil and ornidazole in combined tablet dosage form on silica gel with toluene - *n*-butanol - triethylamine 17:4:1. The hR_F value of ornidazole was 51 and of cefuroxime axetil 67. Quantification by densitometry at 285 nm. Linearity was between 100 and 500 ng/band for both cefuroxime axetil and ornidazole. The method was used for the assay of the compounds in pharmaceutical formulations. The content of cefuroxime axetil was 102.4 % and of ornidazole 101.0 %.

pharmaceutical research, quality control, HPTLC, autoradiography, densitometry, quantitative analysis, qualitative identification 28a

104 051 A.R. ROTE*, S.P. PINGLE* (*Department of Pharmaceutical Chemistry, M. G. V.'s Pharmacy College, Panchavati (Pune University) Mumbai, Agra Road, Nashik 422003, Maharashtra, India): Reverse phase-HPLC and HPTLC methods for determination of gemifloxacin mesylate in human plasma. J. Chromatogr. B 877 (29), 3719-3723 (2009). HPTLC of gemifloxacin mesylate in human plasma, extracted with chloroform - acetic acid 59:1, on silica gel with ethyl acetate - methanol - ammonia 8:4:3. The hR_F value of gemifloxacin mesylate was 33. Quantification by densitometry at 254 nm. The calibration curve was established in the range of 50 to 600 ng/spot. Recovery of gemifloxacin mesylate was between 80.0 and 86.2 %. The stability of gemifloxacin mesylate in plasma was confirmed with samples submitted to three cycles of freeze-thawing at <20 °C, and with samples stored on the bench for 12 h.

clinical chemistry, research, HPTLC, densitometry, quantitative analysis, qualitative identification, comparison of methods 28a

104 052 D.H. SHEWIYO*, E. KAALE, P.G. RISHA, B. DEJAEGHER, J. SMEYERS-VERBEKE, Y. VANDER HEYDEN (*Directorate of Laboratory Services, Tanzania Food and Drugs Authority, P.O. Box 77150, Dar es Salaam, Tanzania): Development and validation of a normal-phase high-performance thin layer chromatographic method for the analysis of sulfamethoxazole and trimethoprim in co-trimoxazole tablets. J. Chromatogr. A 1216 (42), 7102-7107 (2009). HPTLC of co-trimoxazole tablets (combination of sulfamethoxazole and trimethoprim) on silica gel with toluene - ethyl acetate - methanol 100:57:43. Detection under UV 254 nm. The hR_F values were 30 and 61 for trimethoprime and sulfamethoxazole, respectively. Quantification by densitometry at UV 254 nm. Cochran's criterion test indicated homoscedasticity of variances for the calibration data. The F-tests for lack-of-fit indicated that straight lines were adequate to describe the relationship between spot areas and concentrations for each compound. Repeatability and precision *(%RSD)* was 1.0 and 0.8 % for sulfamethoxazole as well as 1.3 % and 1.6 % for trimethoprim, respectively. Recovery was 99.0 % and 99.7 % for sulfamethoxazole and trimethoprim, respectively.

pharmaceutical research, quality control, HPTLC, qualitative identification, quantitative analysis, densitometry 28a

104 053 U. VINCENT, G. GIZZI, C. VON HOLST*, J. DE JONG, J. MICHARD (*European Commission, Joint Research Centre, Insitutute for Reference Materials and Measurements, Food Safety and

Quality Unit, Retieseweg, 2440 Geel, Belgium, christoph.von-holst@ec.europa.eu): Validation of an analytical method for the determination of spiramycin, virginiamycin, and tylosin in feeding-stuffs by thin-layer chromatography and bio-autobiography. Food Addit. Contam. 24, 351-359 (2007). Inter-laboratory validation of the analytical method (published in the SIMBAG-FEED report 4.6) based on TLC coupled to bio-autography for the detection of tylosin, spiramycin and virginiamycin in feeding-stuffs for poultry, pig, cattle and calf. The detection limit of spiramycin was 2 mg/kg and the method has a target concentration of 1 mg/kg for tylosin and virginiamycin. The method showed high specificity and offers the possibility for screening before LC/MS analysis.

quality control, quantitative analysis 28a

104 054 L. YANG (Yang Lihong)*, CH. HU (Hu Changqin), W. LIU (Liu Wenying) (*China Pharm. Univ., Nanjing 210009, China): (Determination of gentamycin and the related compounds by thin-layer chromatography) (Chinese). Chinese J. Pharm. Anal. 26 (2), 221-224 (2006). HPTLC of gentamycin and related compounds on silica gel with chloroform - methanol - 25 % ammonia 5:7:6. The main compound is well separated from the impurities. Quantification by densitometry at 485 nm. Linearity was between 4.0 - 40 ng/spot ($r^2 = 0.99$) and the limit of detection was at the low ng level.

quality control, pharmaceutical research, HPTLC, densitometry, quantitative analysis, qualitative identification 28a

29. Pesticides and other agrochemicals

104 055 V.R. CHANDEGAONKAR, D.B. SHINDE, D.V. MANE* (*Department of Chemistry, Chhatrapati Shivaji College, Omerga, (MS) 413606 India; manedv.2007@rediffmail.com): Thin-layer chromatographic detection and identification of the insecticide imidacloprid in biological materials. J. Planar Chromatogr. 22, 459-460 (2009). TLC of imidacloprid (1-[(6-chloro-3-pyridinylmethyl)]-4,5-dihydro-N-nitro-1H-imidazol-2-amine) and biological extracts on silica gel with chloroform - acetone 7:3 or hexane - acetone - ethanol 8:1:1 with chamber saturation. Detection by spraying with 5 % dimethylaminobenzaldehyde in hydrochloric acid, followed by heating at 100 °C for 10 min. Imidacloprid was detected as a pink zone under visible light.

agricultural, qualitative identification, postchromatographic derivatization 29a

104 056 V.R. CHANDEGAONKAR, J.N. SANGSHETTI, D.B. SHINDE, D.V. MANE* (*Department of Chemistry, Chhatrapati Shivaji College, Omerga (MS) 431606, India; manedv.2007@rediffmail.com): A new chromogenic spray reagent for detection and identification of monocrotophos. J. Planar Chromatogr. 22, 457-458 (2009). TLC of monocrotophos and biological extracts, dimethoate, endosulfan, carbaryl, and cypermethrin on silica gel with chloroform - acetone 7:3 with chamber saturation. Detection by spraying with 5 % sodium hydroxide solution followed by 5 % benzil reagent (5 g benzil in 100 mL acetone) and heating at 100 °C for 10 min. Monocrotophos was detected as a pink zone in daylight.

toxicology, agricultural, qualitative identification 29b

104 057 W. SCHWACK*, T. ZEISLER, C. STIEFEL (*University of Hohenheim, Institute of Food Chemistry, Garbenstrasse 28, 70599 Stuttgart, Germany; wschwack@uni-hohenheim.de): Determination of dialkyl phosphates as breakdown products of organophosphorus insecticides in fruit juices by HPTLC with fluorescence detection. J. AOAC Int. 92, 691-697 (2009). HPTLC of dialkyl phosphate standards (dimethyl phosphate, diethyl phosphate, dimethyl thiophosphate, diethyl thiophosphate and dibutyl phosphate as internal standard) on amino phase, prewashed with methanol, with dichloromethane in a twin trough chamber. Quantitative determination by fluorescence measurement at 366/>400 nm. The limit of quantification was between 0.8 and 1.4 ng/zone. Fluorescence enhancement was achieved by dipping the plate into a 50 % solution of paraffin oil in *n*-hexane, increasing the sensitivity and resulting in an LOQ of 0.5-0.6 ng/zone.

agricultural, toxicology, quality control, food analysis, HPTLC, densitometry, quantitative analysis 29b

104 058 T. TOSTI, G. RAKIC, M. NATIC, D. MILOJKOVIC-OPSENICA, S. HUSINEC, V. SAVIC, Z. TESIC* (*Faculty of Chemistry, University of Belgrade, P. O. Box 51, 11158 Belgrade, Serbia; ztesic@chem.bg.ac.rs): TLC retention behavior of brodifacoum, bromadiolone, and coumatetralyl and their impurities on different adsorbents. J. Planar Chromatogr. 22, 333-343 (2009). TLC of brodifacoum, bromadiolone, coumateralyl and impurities on silica gel, alumina, cellulose, and RP-18 with 46 different mobile phases in a twin trough chamber saturated for 30 min. The most selective phases on silica gel were chloroform - ethyl acetate - *n*-hexane in various ratios, ethyl methyl ketone- toluene 1:9, and chloroform - toluene 7:3; on alumina the best mobile phase was dichloromethane - *n*-hexane 3:2. Detection under UV light at 254 nm.

toxicology, qualitative identification 29f

104 059 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Medical University of Lublin, 4 Staszica Street, 20-081 Lublin, Poland, tomasz.tuzimski@umlub.pl): Application of SPE-HPLC-DAD and SPE-HPTLC-DAD to the analysis of pesticides in lake water. J. Planar Chromatogr. 22, 235-240 (2009). HPTLC of pesticides (clofentezine, neburon, chlorfenvinphos, lenacyl, trifluralin, thiram, procymidone, flufenoxuron, tralkoxydim, propaquizafop, dinoseb) and water samples (sample preparation by solid phase extraction) on silica gel with ethyl acetate - *n*-heptane 1:4 and 3:7 in a horizontal chamber. Quantitative determination by diode array densitometry in the range of 200 to 600 nm. The limit of detection was between 40 and 650 ng/spot and the limit of quantification was between 120 and 1920 ng/spot.

environmental, toxicology, HPTLC, quantitative analysis 29a

30. Synthetic and natural dyes

104 060 Gertrud MORLOCK*, C. OELLIG (*University of Hohenheim, Garbenstrasse 28, 70599 Stuttgart, Germany; gmorlock@uni-hohenheim.de): Rapid planar chromatographic analysis of 25 water-soluble dyes used as food additives. J. AOAC Int. 92, 745-756 (2009). HPTLC of 25 dyes (brilliant black BN, tartrazine, ponceau 6 R, resorcin yellow, fast yellow AB, orcein, allura red, green S, amaranth, quinoline yellow, acid blue, erythrosine, sunset yellow FCF, indigo carmine, ponceau 4R, azorubine, brilliant blue FCF, carmine, scarlet GN, copper chlorophyll, enocyanine, chlorophyllin trisodium copper salt, curcumin, riboflavin-5-phosphate, riboflavin) on silica gel in a horizontal developing chamber with ethyl acetate - methanol - water - acetic acid 65:23:11:1 for 40 samples in 12 min. Relative humidity was 21 +/- 3 % at a temperature of 20 +/- 3 °C. Alternatively, a twin trough chamber or automatic developing chamber can be used. Quantitative determination by absorbance measurement at 11 different wavelengths. Repeatabilities (*%RSD*, n = 4) near the limit of quantification showed precisions of mostly <2.7 %, ranging between 0.2 and 5.2 %. Correlation coefficients (R >0.9987) and RSD values (<4.2 %) of the calibration curves were highly satisfactory using classical quantification. However, digital evaluation of the plate image was also used for quantification, which resulted in RSD values of the calibration curves of mostly <3.0%, except for two <6.0%. The method was applied for the analysis of some energy drinks and bakery ink formulations, directly applied after dilution. By recording of absorbance spectra in the visible range, the identities of the dyes found in the samples were ascertained by comparison with the respective standard bands (correlation coefficients >0.9996). If necessary for confirmation, online mass spectra were recorded within a minute.

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

104 014 F. SCHULTE et al., see section 4e

104 061 ZH. WANG (Wang Zhiyong)*, B. WANG (Wang Bingquan), H. ZOU (Zou Hong) (*Dep. Chem., Capital. Norm. Univ., Beijing 10037, China): (Identification of the category of black gel pen ink by thin-layer chromatography) (Chinese). Physical Testing and Chem. Anal., Part B: Chem. Anal. 45 (1), 14-18 (2009). TLC of ink extracts on silica gel with *n*-butanone - ethanol - water - acetic acid 14:4:6:1. Detection in visble light and identification by comparison of spot colors and hR_F values. The method was used for the identification of 37 different black ink samples from gel pens from different sources, which could then be classified into 13 categories.

quality control, qualitative identification 30a

32. Pharmaceutical and biomedical applications

104 062 S. AGARWAL*, A. AIL, Y. DU, S. ABUJA (*Dept. of Pharmaceutics, Faculty of Pharmacy, Jamie Hamdard University, New Delhi 110062, India, agarwal_sp@yahoo.com): Determination of artemisinin in bulk and pharmaceutical dosage forms using HPTLC. Ind. J. Pharma. Sci. 71(1), 98-100 (2009). HPTLC of artemisinin on silica gel with toluene - ethyl acetate with chamber saturation for 30 min. Quantitative determination by absorbance measurement at 520 nm. The method was linear over the range of 100-600 ng/band, recovery was in the range of 98.8-100.5 %.

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

104 063 S. AHMAD* , M. SINGH, M. KAUR, A. AHMED (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Quantitative estimation of glycyrrhizic acid in the tablets of Yashtimadhu by HPTLC. 60th Indian Pharmaceutical Congress PG-260 (2008). HPTLC of glycyrrhizic acid on silica gel with chloroform - glacial acetic acid - methanol - water 15:8:3:2. Quantitative determination by absorbance measurement at 254 nm. The hR_F value of glycyrrhizic acid was 28. The method was linear in the range of 50-500 ng/spot. The sample analyzed by the proposed method contained 87.8 µg glycyrrhizic acid per tablet, equivalent to 0.015 % w/w of the tablet formulation.

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

104 064 S. AHMAD*, A. KAMAL, R. PARVEEN, F. AHMAD, K. SALEEM (*JMI, Dept. of Biosciences, New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of strychnine and brucine in seeds of Strychnos Nux Vomica. 60th Indian Pharmaceutical congress PA-235 (2008). HPTLC of strychnine and brucine (in seeds of Strychnos Nux Vomica) on silica gel with chloroform - methanol - ammonia 38:2:1. Quantitative determination by absorbance measurement at 258 nm (strychnine, hR_F value 29) and 271 nm (brucine, hR_F value 21). The method was linear in the range of 100-1000 ng/spot (both compounds). The method was suitable for standardization of several herbal formulations containing nux vomica as an ingredient.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 065 S. AHMAD*, M. SINGH, R. PARVEEN, Y. KAMAL (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Quantitative estimation of withaferin a in the tablets of Ashwagandha by HPTLC. 60th Indian Pharmaceutical Congress PG-262 (2008). HPTLC of withaferin A in Ashwagandha on silica gel aluminum layer with toluene - ethyl acetate - formic acid 5:5:1. Quantitative determination by absorbance measurement at 214 nm. The chromatograms of the tablet formulation showed a zone corresponding to standard withaferin A (hR_F value 37) indicating the presence of the same in the herbal formulation. Linearity was in the range 50 to 500 ng/spot. The concentration of withaferin A was 8.07 µg/tablet and 0.00134 % w/w in the tablet formulation.

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

104 066 M. ALAVALA (KLE University‘s College of Pharmacy, Belgaum, Karnataka, India): New stability indicating high-performance liquid chromatography and high-performance thin-layer chromatography method for the estimation of olopatadine hydrochloride. Abstract No. F-309, 61st IPC (2009). HPTLC of olopatadine HCl on silica gel with methanol - chloroform - 25 % ammonia 80:20:1. The hR_F value was 46. Quantitative determination by absorbance measurement at 301 nm. The method was linear in the range of 100-900 ng/band.

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

104 067 B. ARUN*, A. SUGANTHI, A. FATHIMUNNISA, T. RAVI (*College of Pharmacy, Sri Ramakrishna Institute of Paramedical Science, Coimbatore, Tamil Nadu, India): Development of validated HPTLC method for the estimation of buclizine hydrochloride in tablet dosage form. Abstract No. F-276, 61st IPC (2009). HPTLC of buclizine hydrochloride on silica gel with methanol - chloroform - ammonia 8:1:1 %. Quantitative determination by absorbance measurement at 234 nm.

The calibration curve was linear in the range of 100-700 ng/spot. The limit of detection and limit of quantification were 20 and 100 ng/spot, respectively.

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

104 068 J. BAGYALAKSHMI*, S. VIJAYARAJ, Sajna JOHN, T. RAVI (*Sri Ramkrishna Instiute of Paramedical Science, College of Phamracy, Coimbatore, Tamilnadu, India): Development and validation of simultaneous estimation of paracetamol, aceclofenac and rabeprazole in combined tablet dosage formulation by HPTLC method. 60th Indian Phamraceutical Congress PA-222 (2008). HPTLC of paracetamol, aceclofenac and rabeprazole on silica gel with ethyl acetate - methanol - glacial acetic acid 90:10:1. Quantitative determination by absorbance measurement at 275 nm. Linearity was in the range of 100-500 µg/mL, 20-100 µg/mL and 2-10 µg/mL for paracetamol, aceclofenac and rabeprazole respectively. Recovery was in the range of 98.9-100.1 % for all three compounds.

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

104 069 N. BAIRWA*, A. TRIVEDI, S. MISHRA (*The M. S. University of Baroda, Pharmacy Dept., Vadodara, Gujarat, India): Simultaneous estimation of markers in a haematinic herbomineral formulation using high-performance thin-layer chromatography. 60th Indian Pharmaceutical Congress PG-251 (2008). HPTLC of glycyrrhetinic acid and piperine in haematinic herbomineral capsule formulation on silica gel with toluene - ethyl acetate - glacial acetic acid 25:15:1. Quantitative determination by absorbance measurement at UV 254 nm. The method was linear in the range of 0.8-2.4 ng/spot (glycyrrhetinic acid) and 10-50 ng/spot (piperine). Recovery was 96.3-98.6 %. traditional medicine

herbal, HPTLC, densitometry, quantitative analysis 32e

104 070 V. BALI*, M. ALI, J. ALI (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): A novel and rapid HPTLC method for the analysis of rosuvastatin calcium. 60th Indian Pharmaceutical Congress PA-219 (2008). HPTLC of rosuvastatin calcium on silica gel with toluene - ethyl acetate - formic acid 9:7:1. Quantitative determination by absorbance measurement at 254 nm. The hR_F value was 32. The method was linear in the range of 100-100 ng/spot, recovery was 99.7 %.

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

104 071 V. BHAVNANI*, V. DIWALE, P. DESHMUKH, A. DHIWARE (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): Validated method development for estimation of famotidine and domperidone in combined tablet dosage form. Abstract No. F-335, 61st IPC (2009). HPTLC of famotidine and domperidone on silica gel with toluene - methanol - triethyl amine 12:6:1. The hR_F value was 23 and 67 for famotidine and domperidone, respectively. Quantitative determination by absorbance measurement at 290 nm. The method was linear in the range of 100-500 ng/band. Recovery was 98.6-98.9 % for both drugs.

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

104 072 D. BHAWSAR*, P. RATHI, M. PURANIK, P. YEOLE (*Institute of Pharmaceutical Education and Research, Wardha, Maharashtra, India): Development and validation of HPTLC technique for simultaneous estimation of gatifloxacin and ornidazole in solid dosage forms. Abstract No. F-347, 61st IPC (2009). HPTLC of gatifloxacin and ornidazole on silica gel with dichloromethane - methanol - 25 % ammonia 95:10:3. The hR_F value was 16 and 60 for gatifloxacin and ornidazole, respectively. Quantitative determination by absorbance measurement at 302 nm. The method was linear in the range of 20-200 ng/band for gatifloxacin and 50-500 ng/band for ornidazole. Recovery was between 100.4 and 101.9 for both drugs.

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

104 073 R. BIDAWAI*, N. RAUT, D. WANKHEDE, N. GAIKWAD (*University Dept. of Pharmaceutical Science, RTM Nagpur University, Nagpur, Maharashtra, India): Validated stability indicating

HPTLC method for the estimation of olmesartan medoxomil in bulk and pharmaceutical dosage form. 60th Indian Pharmaceutical Congress PA-226 (2008). HPTLC of olmesartan medoxomil (an angiotensin-II antagonist) on silica gel with toluene - acetonitrile - methanol - ethyl acetate - acetic acid (mobile phase ratio not specified by the authors). The hR_F value was 56. Quantitative determination by absorbance measurement at 262 nm. The linearity was between 300-800 ng/spot. The method was suitable for separation of olmesartan medoxomil from degradation products obtained by forced stress conditions (acid, alkali, peroxide, light, heat).

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

104 074 A. CHABUKSWAR*, S. JAGDALE, S. KUMBHAR, R. WAVHALE (*Bombay College of Pharmacy, Mumbai, Maharashtra,India): Simultaneous HPTLC estimation of certain antihypertensive drugs in tablet dosage form. 60th Indian Pharmaceutical Congress PA-230 (2008). HPTLC of amlodipine besylate (AB) and telmisartan (TMS) on silica gel with ethyl acetate - 1,4-dioxane - methanol - 25 % ammonia 30:3:6:3. The hR_F value was 16 for TMS and 33 for AB. Quantitative determination by absorbance measurement at 323 nm. The method was linear in the range of 100-500 µg/mL (TMS) and 200-1000 µg/mL (AB), average recovery was 100.2-100.4 %.

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

104 075 Mrinalini DAMLE*, K. BOTHARA, Kirti TOPAGI (*Dept. fo Chem. A.I.S.S.M.S. College of Pharma, Kennedy Road, R.T.O. Pune 411044, India., mcdamle@rediffmail.com): Stability-indicating HPTLC method for determination of nebivolol hydrochloride and valsartan. Ind. J. Pharma. Sci. 8(4), 198-201(2009). HPTLC of nebivolol hydrochloride and valsartan on silica gel with ethyl acetate - methanol - acetic acid 12:2:1 in a twin trough chamber saturated for 15 min. Quantitative determination by absorbance measurement at 240 nm for valsartan and 280 nm for nebivolol hydrochloride. The method was found to be linear in the range of 600-1400 ng/band for valsartan 1200-2800 ng/band for nebivolol. The sample were subjected to different stress conditions (acid, alkali, oxidation, photolysis, thermal) and all degradation products were well separated from the main compounds.

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

104 076 R.S. DAREKAR, A.B. KHETRE, S.M. SINGH, M.C. DAMLE* (*Department of Pharmaceutical Chemistry, AISSMS College of Pharmacy, Near R. T. O., Kennedy Road, Pune 411 001, India, mcdamle@rediffmail.com): HPTLC quantitation of 2-hydroxy-4-methoxybenzaldehyde in Hemidesmus indicus R. Br. root powder and extract. J. Planar Chromatogr. 22, 453-456 (2009). HPTLC of 2-hydroxy-4-methoxybenzaldehyde and biological extracts on silica gel with toluene - ethyl acetate - acetic acid 7:2:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 277 nm.

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

104 077 P. DESHPANDE*, G. SHRIDHARAN, L. ANANDI, D. JADHAV, M. DAMLE, S. GANDHI (*Dept. of Pharmaceutical Analysis, AISSMS College of Pharmacy, Kennedy Road, Pune, India, santoshvgandhi@rediffmail.com): Validated method development for estimation of atorvastatin calcium and fenofibrate in fixed dose combination by HPTLC. The Pharma Review 7(39), 151-153 (2009). HPTLC of atorvastatin calcium and fenofibrate on silica gel (pre-washed with methanol) with chloroform - methanol 4:1 over 20 mm with chamber saturation. The hR_F value of atorvastatin calcium was 29 and of fenofibrate 77. The method was linear in the range of 200-1000 ng/band for atorvastatin calcium and 320-1600 ng/band for fenofibrate.

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

104 078 S.R. DHANESHWAR*, V.K. BUSARI, M.V. MAHADIK (*Bharati Vidyapeeth University, Poona College of Pharmacy, Department of Pharmaceutical Chemistry, Pune, Maharashtra, India 411038; sunil.dhaneshwar@gmail.com): Application of a stability-indicating thin-layer chromatographic method to the determination of tenatoprazole in pharmaceutical dosage forms. J.

AOAC Int. 92, 387-393 (2009). TLC of tenatoprazole - before and after acid and alkali hydrolysis, oxidation and photodegradation - on silica gel, prewashed with methanol and dried at 110 °C for 5 min, with toluene - ethyl acetate - methanol 6:4:1 in a twin trough chamber saturated with the mobile phase for 30 min at 25 °C. Quantitative determination by absorbance measurement at 306 nm. The limit of detection and limit of quantitation were 50 and 100 ng/spot, respectively.

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

104 079 V.V. DIGHE, G.A. CHAREGAONKAR* (*S. P. Mandali's Ramnarain Ruia College, Matunga, Mumbai 400 019, India; gaurichareGaonkar@gmail.com): HPTLC analysis of myristicin and safrole in seed powder of Myristica fragrans Houtt. J. Planar Chromatogr. 22, 445-448 (2009). HPTLC of myristicin, safrole and extract of seeds on silica gel, prewashed with methanol, with toluene in an automatic developing chamber saturated for 20 min. Quantitative determination by absorbance measurement at 210 nm for myristicin and at 290 nm for safrole.

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

104 080 Avani DODIYA, Shweta PAWAR, C. PATEL (*School of Pharmacy and Technology Management, NMIMS University, Mumbai, Maharashtra, India): Simultaneous determination of nebivolol hydrochloride and hydrochlorothiazide in tablets by high-performance thin-layer chromatography. Abstract No. F-280, 61st IPC (2009). HPTLC of nebivolol HCl and hydrochlorothiazide on silica gel with toluene - ethyl acetate - methanol - 25 % ammonia 30:27:17:2. The hR_F value was 38 and 68 for hydrochlorothiazide and nebivolol, respectively. Quantitative determination by absorbance measurement at 281 nm. The method was linear in the range of 500-3000 ng/band for nebivolol and 1000-6000 ng/band for hydrochlorothiazide. Recovery was 100 %.

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

104 081 N. DUBEY*, N. DUBEY, R. MEHTA, A. K. SALUJA (*Devi Ahilya Vishwa Vidyalaya, School of Pharmacy, Indore, India; nidhidubeympharm@yahoo.com) : Determination of psoralen and plumbagin from its polyherbal oil formulations by an HPTLC densitometric method. J. AOAC Int. 92, 779-784 (2009). HPTLC of psoralen and plumbagin and extracts of ayurvedic polyherbal oil formulations on silica gel at 22 °C and 55 % humidity with toluene - ethyl acetate 3:1 in a twin trough chamber with chamber saturation. UV spectra were recorded from 200 to 600 nm; densitometric measurements were performed at 302 nm (for psoralen) and 275 nm (for plumbagin).

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

104 082 N. DUBEY*, N. DUBEY, R. MEHTA, A. SALUJA (*Devi Ahilya Vishwavidyalaya (DAVV), School of Pharmacy, Indore, Madhya Pradesh, India; nidhidubeympharm@yahoo.com): Estimation of catechin in Ayurvedic oil formulations containing Acacia catechu. J. AOAC Int. 92, 1021-1026 (2009). HPTLC of catechin and extracts from polyherbal oil formulations on silica gel using chloroform - acetone - 0.1 % formic acid 77:15:8 % in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 296 nm. The limit of detection and quantification was 6 and 20 ng/spot, respectively.

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

104 083 Nidhi DUBEY*, N. DUBEY, R. MEHTA, A. SALUJA (*Devi Ahilya Vishwavidyalaya, School of Pharmacy, Indore, M.P., India): Rapid densitometric determination of Allium sativum in polyherbal oil formulations. 60th Indian Pharmaceutical Congress PA-202 (2008). HPTLC of allyl disulphide (an active ingredient of Allium sativum, garlic) on silica gel with *n*-hexane. The hR_F value was 52. Quantitative determination by absorbance measurement at 298 nm. The linearity range was 200-1200 ng/spot. Several polyherbal formulations containing garlic were analyzed with the proposed method using allyl disulphide as marker.

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

104 085 R.R. DURÓN, L. CENICEROS ALMAGUER, N.C. CAVAZOS ROCHA, P.G.S. FLORES, Noemi WAKSMAN DE TORRES* (*Universidad Autónoma de Nueve León, Departamento de Química Analítica, Facultad de Medicina, PO Box 2316, Sucursal Tecnológico, 64841, Monterrey Nuevo León, Mexico; nwaksman@fm.uanl.mx): Comparison of high-performance liquid chromatographic and thin-layer chromatographic methods for determination of aloin in herbal products containing Aloe vera. J. AOAC Int. 91, 1265-1270 (2008). TLC of aloin A and extracts of dried herbal products applied bandwise on silica gel with ethyl acetate - methanol - water 100:17:10 at 25 °C with chamber saturation. Detection by spraying with 1 % methanolic diphenylboric acid 2-aminoethylester (natural products reagent), followed by 5 % ethanolic polyethylene glycol 400 and visualization under UV light at 365 nm. The hR_F value of aloin (as a mixture of aloin A and B stereoisomers) was 38. Linearity was between 0.5 and 10 mg/L. Precision *(%RSD)* of the hR_F value of the aloin band was 0.88. The limit of detection was 1 µg/band. Recovery (by standard addition) was 87.6 % for aloin.

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

104 086 Halina EKIERT*, A. SZEWCZYK, A. KUS (*Jagiellonian University, Collegium Medicum, Faculty of Pharmacy, Chair and Department of Pharmaceutical Botany, 9 Medyczna Street, 30-688 Kraków, Poland; mfekiert@cyf-kr.edu.pl): Free phenolic acids in Ruta graveolens L. in vitro culture. Pharmazie 64, 694-696 (2009). Preparative TLC and HPTLC of protocatechuic acid, vanillic acid, syringic acid, and p-coumaric acid and methanolic extracts on silica gel. Detection under UV light at 254 nm.

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

104 087 Izabella FECKA (Wroclaw Medical University, Department of Pharmacognosy, Pl. Nankierea 1, 50-140 Wroclaw, Poland; izabela@farmgn.am.wroc.pl): Development of chromatographic methodes for determination of agrimoniin and related polyphenols in pharmaceutical products. J. AOAC Int. 92, 410-418 (2009). HPTLC of agrimoniin, pedunculagin, ellagic acid, gallic acid and catechin and plant extracts on silica gel, RP-18 and amino phase in a horizontal chamber. The best resolution and selectivity were achieved with diisopropyl ether - acetone - formic acid - water 4:3:2:1, tetrahydrofuran - acetonitrile - water 3:1:6, and acetone - formic acid 3:2. Polyphenols were detected under UV light at 254 nm and in visible light after spraying with 1 % methanolic iron(III) chloride or bis-diazotized sulfanilamide and after treatment with a vanillin-hydrochloric acid reagent.

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

104 088 J. FISCHEDICK*, R. GLAS, A. HAZEKAMP, R. VERPOORTE (*Division of Pharmacognosy, Leiden University, Gorlaeus Laboratories, 2333 CC Leiden, The Netherlands, jtfische@gmail.com): A qualitative and quantitative HPTLC densitometry method for the analysis of cannabinoids in Cannabis sativa. Phytochem. Anal. 20, 421-426 (2009). HPTLC of delta-9-tetrahydrocannabinol in the flowertops of Cannabis sativa on silica gel with chloroform with chamber saturation for 20 min. Quantitative determination by absorbance measurement at 206 nm. Derivatization by dipping in Fast Blue B solution for 5 s. The hR_F value of delta-9-tetrahydrocannabinol was 47 and selectivity regarding matrix was given. Linearity was given between 50 and 500 ng/zone. The limit of quantification and detection was 50 and 10 ng/zone, respectively. The intra- and inter-day repeatability (*%RSD*, n = 9) were not higher than 5.0 %. Recovery was 85.8 % for delta-9-tetrahydrocannabinol in decarboxylated Cannabis samples. The method was shown to be comparable within a small degree of error (0.5 %) to results from a validated HPLC method.
toxicology, quality control,

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

104 089 M. GAME*, M. JADHAO, V. WANKHADE, G. GHENGE (*Vidyabharti College of Pharmacy, Amravati, Maharashtra, India): Estimation of andrographolide in herbal powder and polyherbal asava by HPTLC. Abstract No. F-299, 61st IPC (2009). HPTLC of herbal powder and polyherbal formulation containing Andrographis paniculata on silica gel with benzene - ethyl acetate 1:1.

Quantitative determination by absorbance measurement at 222 nm. The calibration curve for andrographolide was linear in the range of 360-660 ng/band.

quality control, herbal, HPTLC, densitometry 32e

104 090 D. GANDHI*, N. PATEL, P. MEHTA (*Institue of Pharmacy, Nirma Univeristy, Ahmedabad, Gujarat, India): Development and validation of HPTLC method for simultaneous determination of levodopa and carbidopa in their combined dosage form. Abstract No. F-246, 61st IPC (2009). HPTLC of levodopa and carbidopa on silica gel with acetone - chloroform - *n*-butanol - acetic acid - water 50:45:42:35:25. Quantitative determination by absorbance measurement at 283 nm. The method was linear in the range of 200-700 ng/band for both compounds, with a recovery of 98.7-99.9 %.

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

104 091 S.V. GANDHI*, S.I. KHAN, R.T. JADHAV, S.S. JADHAV, G.A. JADHAV (*AISSMS College of Pharmacy, Department of Pharmaceutical Analysis, Pune, India; santoshvgandhi@rediffmail.com): High-performance thin-layer chromatographic determination of rabeprazole sodium and domperidone in combined dosage form. J. AOAC Int. 92, 1064-1067 (2009). HPTLC of rabeprazole sodium and domperidone in tablets on silica gel (prewashed with methanol and dried at 110 °C for 5 min) with toluene - acetone - methanol 9:9:1 in a twin trough chamber saturated with the mobile phase for 5 min. Quantitative determination by absorbance measurement at 285 nm. The hR_F value of rapebrazole sodium and domperidone was 53 and 32, respectively. Linearity was between 50 and 800 ng/band for both substances.

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

104 092 M. GANESH*, S. SWANT, R. JAMBHALE, A. KASABE (*Arvind Gawali College of Pharmacy, Satara, Maharashtra, India): Extraction and estimation of theobromine in marketed tea by HPTLC and UV method. Abstract No. F-277, 61st IPC (2009). HPTLC of theobromine in different extracts of tea (Camelia sinensis) on silica gel with ethyl acetate - methanol 27:3. Quantitative determination by absorbance measurement at 274 nm. The maximum content of theobromine in tea samples was 2.3 %. Linearity was in the range of 3-15 µg/zone. The limit of detection and quantification was 30 and 140 ng/spot, respectively.

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

104 093 A. GANTAIT* , K. MUKHERJEE, S. PONNUSANKAR, P. MUKHERJEE (*Jadavpur University, School of Natural Product Studies, Kolkata, India): A validated method for standardization of Centella asiatica extract. 60th Indian Pharmaceutical Congress PG-244 (2008). HPTLC of Centella asiatica extract with asiaticoside as marker on silica gel with chloroform - glacial acetic acid - methanol - water 15:8:3:2 with chamber saturation. Detection by spraying with anisaldehyde reagent, followed by heating in oven and immediate quantitative determination by absorbance measurement at 607 nm. The hR_F value of asiaticoside was 81. Linearity was in the range of 0.96-3.36 µg/spot. Hydroalcoholic extracts contained approx. 3.2 % of asiaticoside.

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

104 094 A GANTAIT*, N. NEMA, A. SAHU, S. PANDIT, S. BHADRA, P. MUKHERJEE (*School of Natural Product Studies, Jadavpur University, Kolkata 700032, India): A validated method for quantification of glycyrrhizin in Glycyrrhiza glabra extract by HPTLC. Abstract No. 9162, IHCB (2009). HPTLC of glycyrrhizin in methanolic (70 %) extracts of Glycyrrhiza glabra on silica gel with chloroform - methanol - water 130:72:15. Quantitative determination by absorbance measurement at 254 nm and at 420 nm after spraying with anisaldehyde reagent. The method was linear in the range of 0.8-3.8 µg/spot. The limit of detection and quantification was 0.16 and 0.52 µg/spot, respectively. Glycyrrhizin was used as bioactive marker for quality control.

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

104 095 A. GANTAIT*, P. ROY, S. PANNUSANKAR, P. MUKHERJEE, B. SAHA (*School of Natural Product Studies, Jadavpur, Kolkata 700032, India): Standardization of Tinospora cordifolia extract through HPTLC densitometry. Abstract No. 91334, IHCB (2009). Standardization of Tinospora cordifolia extract by HPTLC of syringic acid on silica gel with chloroform - methanol 8:1. The hR_F value of syringic acid was 53. Quantitative determination by absorbance measurement at 254 nm.

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

104 096 Vidya GAWANDE*, Manisha PURANIK, A. CHANDEWAR (*Pataldhamal Wadhwani College of Pharmacy, Yavatmal, Maharashtra, India): Development of validated HPTLC method for simultaneous estimation of domperidone in combination with esomeprazole magnesium in solid dosage form. 60th Indian Pharmaceutical Congress PA-220 (2008). HPTLC of domperidone and esomeprazole on silica gel with chloroform - methanol 9:1 with chamber saturation for 30 min. The hR_F value was 25 for domperidone and 46 for esomeprazole. Quantitative determination by absorbance measurement at 295 nm. The method was linear in the range of 60-300 ng/spot for domperidone and 80-400 ng/spot for esomeprazole.

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

104 097 S. GHODKE*, A. RATHORE, L. SATHIYANARAYANAN, K. MAHADIK (*Bharati Vidyapeeth University, Poona College of Pharmacy, Pune, Maharashtra, India): Validated HPTLC method for simultaneous estimation of isotretinoin and rrythromycin in bulk drug and topical gel form. Abstract No. F-243, 61st IPC (2009). HPTLC of isotretinoin and erythromycin on silica gel with toluene - dimethyl sulfoxide - methanol 65:2:25. Quantitative determination by absorbance measurement at 340 nm before derivatization for isotretinoin and at 410 nm for erythromycin after derivatization with 10 % sulfuric acid followed by heating at 100 °C for 15 min. The hR_F value was 38 and 55 for isotretinoin and erythromycin, respectively. The linearity was in the range of 30-150 ng/band for isotretinoin and 1200-6000ng/band for erythromycin. Recovery was between 96.9 and 99.7 for isotretinoin and between 97.2 and 102.6 % for erythromycin. Intra-day and inter-day relative standard deviations for both components were <2.0 %.

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

104 098 S. GOHIL*, S. PATEL, N. PATEL, D. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganapat University, Mehsana, India): Estimation of atomoxetine hydrochloride by HPTLC method in pharmaceutical formulations. 60th Indian Pharmaceutical Congress PA-212 (2008). HPTLC of atomoxetine HCl on silica gel with acetone - methanol - triethylamine 30:15:2. Quantitative determination by absorbance measurement at 275 nm. The method was linear in the range of 300-2100 ng/spot and was suitable for routine quality control of pharmaceutical formulations.

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

104 099 P. HAMRAPURKAR*, N. LONE, K. KAMAT, T. SAMBARE (*Principal K. M. Kundnani College of Pharmacy, Mumbai, Maharashtra, India): Quantitative determination of aloe-emodin in Rheum emodi using HPTLC. 60th Indian Pharmaceutical Congress PA-224 (2008). HPTLC of aloe-emodin in extract of Rheum Emodi (prepared by supercritical fluid extraction) on silica gel with toluene - acetone - formic acid 80:20:1. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 100-400 ng/spot. Compared with other extraction techniques supercritical fluid extraction was more effcient and less time consuming.

herbal, HPTLC, quantitative analysis, qualitative identification 32e

104 100 T. HONG, M.L. JEONG, M. ZAHN, B.A. FAY, K. LEE, H. HWANGBO, E. PARK, M. KIM, W. MA* (*Unigen Inc., Quality Control/Quality Assurance Department, 2660 Willamette Dr, NE, Lacey, WA 98516, USA; WenwenM@unigen.net): Detection of the potential adulterant Teuc-

rium chamaedrys in Scutellaria baicalensis raw material and extract by high-performance thin-layer chromatography. J. AOAC Int. 92, 785-788 (2009). HPTLC of plant extracts and herbal preparations applied bandwise on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:25 after preconditioning for 5 min. Detection by immersion in natural products reagent (diphenylboric acid 2-aminoethylester) followed by polyethylene glycol 400 reagent for 2 s. After air-drying the plates were evaluated under UV 366 nm.

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

104 101 F. HOU (Hou Feng)*, F. LIU (Liu Fang), Q. MO (Mo Qiwu) (*Guangzhou Meichen Pharm. Co. Ltd., Guangzhou 510075, China): (Study of the quality standard for Conghuang Bushen capsules) (Chinese). J. Chinese Trad. & Herb. Drugs 40 (8), 1249-1252 (2009). TLC of extracts of the TCM drug on silica gel with 1) methanol - acetic acid - water 18:1:4; 2) petroleum ether (60-90 °C) - ethyl acetate 1:1; 3) toluene - ethyl acetate - methanol 5:5:3; 4) petroleum ether (60-90 °C) - ethyl acetate - formic acid 15:5:1. Detection 1) by spraying with potassium iodobismuthate reagent; 2) under UV 254 nm; 3) by spraying with 5 % $AlCl_3$ in ethanol and evaluation under UV 365 nm.

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

104 102 X. HOU (Hou Xiaotao)*, L. MU (Mu Liqun), L. HUANG (Huang Lifen), J. ZHOU (Zhou Jiangyu) (*Guangxi Inst. TCM, Nanning, Guangxi 530001, China): (Study on the quality standard for Yixuean Pills) (Chinese). Chinese J. Hospit. Pharm. 29 (8), 686-688 (2009). TLC of the extracts of Yixuean pills on silica gel with 1) chloroform - ethyl acetate - acetone - formic acid 60:25:25:4; 2) *n*-hexane - chloroform - methanol 15:5:2; 3) ethyl acetate - formic acid - acetic acid - water 15:1:1:2. Detection 1) under UV 254 nm; 2) by exposure to iodine vapor and under UV 254 nm; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration evaluation under visible light and UV 254 nm.

cosmetics, pharmaceutical research, traditional medicine, quality control, qualitative identification, quantitative analysis, review, densitometry 32c

104 103 J. HUANG (Huang Jiefen)*, H. LI (Li Huixia), H. DENG (Deng Huimin) (*Guangzhou Zhongyi Pharm. Co. Ltd., Guangzhou 510530, China): (Study of the quality standard for Xinyi Biyan pills) (Chinese). J. Chinese Trad. & Herb. Drugs 40 (8), 245-247 (2009). TLC of the TCM drug extracts on silica gel with 1) petroleum ether (90-120 °C) - toluene - formic acid 20:40:1; 2) chloroform - ethyl acetate - methanol - water - formic acid 3:10:2:2:2; 3) ethyl acetate - formic acid - water 12:2:3. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C; 2) by exposure to iodine vapor; 3) under UV 254 nm.

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

104 104 X. HUANG (Huang Xiaoyu)*, N. HUANG (Huang Nojia) (*Wannianqing Pharm. Co., Guangdong Prov., Shantou, Guangdong 515031, China): (Simultaneous identification of Fructus Schisandrae sphenantherae, Rhizoma Acori tatarinowii, Rhizoma Chuanxiong and vitamin E in Naolibao pills by thin-layer chromatography) (Chinese). J. Chinese Pharm. Standard 10 (4), 284-286 (2009). TLC of Naolibao pill extracts on silica with *n*-hexane - ethyl acetate 5:1. Qualitative identification by detection under UV 254 nm and 365 nm. The method is simple, rapid, reliable, and suitable for the quality control of the TCM formulation.

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

104 105 Demiana I. NESSEEM*, C.G. MICHEL, A.A. SLEEM, T.S. EL-ALFY (*Pharmaceutics Department, National Organization for Drug Control & Research (NODCAR), 6 Abou Hazem St. Pyramids Ave, Cairo, Egypt; demianaesseem@yahoo.com): Formulation and evaluation of antihyperglycemic leaf extracts of Zizyphus spina-christi (L.) WILLD. Pharmazie 64, 104-109

(2009). TLC of christinin-A as marker and leaf extracts on silica gel with chloroform - methanol - water 13:8:2 and butanol - acetic acid - water 4:1:5 (upper phase). Detection by spraying with p-anisaldehyde reagent.

pharmaceutical research, herbal, qualitative identification 32e

104 106 P.S. JAIN (R.C. Patel College of Pharmacy, Karwand Naka, Shirpur Dist. Dhule 425 405 (M.S.) India): Stability-indicating HPTLC determination of ambroxol hydrochloride in bulk drug and pharmaceutical dosage form. J. Chromatogr. Sci. 48 (1), 45-48 (2010). HPTLC of ambroxol hydrochloride on silica gel with methanol - triethylamine 2:3. The hR_F value of ambroxol was 53. Quantification by absorbance measurement at 254 nm. Linearity was given in the range of 100-1000 ng/spot with $r^2 = 0.9966$ (via peak area). The limits of detection and quantitation were 10 and 30 ng/spot, respectively. Ambroxol hydrochloride was susceptible to degradation under oxidation and thermal stress conditions. The method is suitable for purity testing of the drug as it detects the related impurities.

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

104 107 V. JAITAK*, A. GUPTA, V. KAUL, P. AHUJA (*Natural Plant Products Div. Institute of Bioresource Technology, Palampur 176061, H.P., India, vkaul2002@yahoo.co.in): Validated high-performance thin-layer chromatography method for steviol glycosides in Stevia rebaudiana. J. Pharm. Biomed. Anal. 47, 790-794 (2008). HPTLC of the steviolbioside, stevioside and rebaudioside A in Stevia rebaudiana leaves on silica gel with ethyl acetate - ethanol - water 20:5:3. Detection by spraying with acetic anyhdride - sulphuric acid - ethanol 1:1:10 reagent. Quantitative determination by absorbance measurement at 510 nm. Linearity was in the range of 160-960 ng/spot for steviolbioside, 1-6 µg/spot for stevioside and 0.5-3 µg/spot for rebaudioside A with good correlation coefficients (0.998-0.999). The method was used for the assay of steviol glycosides in S. rebaudiana leaves collected from ten different locations.

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

104 108 P. JHA*, S. KHAN, R. PARVEEN, S. AHMAD (*Jamia Hamdard, Faculty of Pharmacy (Hamdard University), New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of omeprazole in pharmaceutical dosage form. 60th Indian Pharmaceutical congress PA-234 (2008). HPTLC of omeprazole on silica gel with chloroform - methanol 9:1. The hR_F value was 39. Quantitative determination by absorbance measurement at 302 nm. The method was linear in the range of 5-3000 ng/spot, recovery was 99.5 %. The method was suitable for routine quality control of formulations.

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

104 109 Maithilee JOSHI*, A. NIKALJE, M. SHAHED, M. DEHGAN (*Y. B. Chavan College of Pharmacy, Dr. Rafiq Zakaria Campus, Rauza Bagh, Aurangabad 431001, India, ana@k.st): HPTLC method for the simultaneous estimation of emtricitabine and tenofovir in tablet dosage form. Ind. J. Pharma. Sci. 71(1), 95-97 (2009). HPTLC of emtricitabine and tenofovir on silica gel with chloroform - methanol 9:1. Quantitative determination by absorbance measurement at 265 nm. The calibration curve was linear between 200 and 1000 ng with a regression coefficient of 0.9995.

pharmaceutical research, densitometry, HPTLC, quantitative analysis 32a

104 110 S.S. KADUKAR, S.V. GANDHI*, P.N. RANJANE, S.S. RANHER (*Department of Pharmaceutical Analysis, AISSMS College of Pharmacy, Kennedy Road, Near R. T. O., Pune 411 001, Maharashtra, India; santoshvgandhi@rediffmail.com): HPTLC analysis of olmesartan medoxomil and hydrochlorothiazide in combination tablet dosage forms. J. Planar Chromatogr. 22, 425-428

(2009). HPTLC of olmesartan medoxomil and hyrochlorothiazide on silica gel, prewashed with methanol, with chloroform - methanol - toluene 6:4:5 in a twin trough chamber saturated for 15 min. Quantitative determination by absorbance measurement at 258 nm.

pharmaceutical research, quality control, HPTLC 32a

104 111 R. KAKDE*, D. SATONE, N. BAWANE (*Department of Pharmaceutical Sciences, RTM Nagpur University, Nagpur-440 033, Maharashtra, India; drkakde@yahoo.com): HPTLC method for simultaneous analysis of escitalopram oxalate and clonazepam in parmaceutical preparations. J. Planar Chromatogr. 22, 417-420 (2009). HPTLC of escitalopram oxalate and clonazepam on silica gel, prewashed with methanol, in a twin trough chamber saturated for 20 min at 25 °C with methanol - toluene - triethylamine 10:35:1. Quantitative determination by absorbance measurement at 253 nm.

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

104 112 P. KAKULTE*, M. DESHPANDE, S. CHAUDHRI, V. KASTURE (*Amrutvahini College of Pharmacy, Ahemadnagar, Maharashtra, India): High-performance thin-layer chromatographic determination of ambroxol in human plasma by liquid-liquid extraction and its use in stability study. Abstract No. F-286, 61st IPC (2009). HPTLC of ambroxol (extracted from plasma with diethylether, after centrifugation the organic layer was evaporated and the residue was taken up in 1 mL of methanol) on silica gel with acetonitrile - methanol - triethylamine 41:5:4. Quantitative determination by absorbance measurement at 254 nm.

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

104 113 R. KANT*, M. GUPTA (*Delhi Institute of Pharmaceutical Science and Research New Delhi, India): HPTLC method development and its validation for determination of ranolazine in pharmaceutical formulations. Abstract No. F-259, 61st IPC (2009). HPTLC of ranolazine on silica gel with methanol - toluene 9:11 in a twin trough chamber with chamber saturation for 30 min. The hR_F value was 64. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 2-14 µg/band. Recovery was 98.3-101.4 %.

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

104 114 A. KAUR*, V. RAVICHANDRAN, P. JAIN, R. AGRAWAL (*Pharmaceutical Chemistry Research Lab. Dept. of Pharmaceutical Sciences, Dr. Hari Singh Gour Univ. Sagar, MP 470003, India, dragrawal2001@yahoo.co.in): High-performance thin-layer chromatography method for estimation of conessine in herbal extract and pharmaceutical dosage formulations. J. Pharm. Biomed. Anal. 46, 391-394 (2008). TLC of conessine on silica gel with toluene - ethyl acetate - diethyl amine 13:5:2 in a twin trough chamber saturated at 25 °C. Detection by treatment with modified Dragendorff's reagent. Quantitative determination by absorbance measurement at 520 nm. The hR_F value of conessine was 82. Linearity was in the range of 1-10 µg/zone with a correlation coefficient of 0.9998 via peak area.

pharmaceutical research, herbal, densitometry, quantitative analysis 32e

104 115 H. KHAN*, M. ALI, A. AHUJA, S. AHMAD, J. Ali (*Jamia Hamdard, Faculty of Pharmacy, New Delhi, India): Stability indicating TLC method for simultaneous estimation of aceclofenac and paracetamol in bulk drugs and in their fixed dose combinations. 60th Indian Pharmaceutical Congress PA-218 (2008). TLC of aceclofenac and paracetamol on silica gel with toluene - isopropylalcohol - ammonia 8:7:1. The hR_F value of aceclofenac was 24 and of paracetamol 68. Quantitative determination by absorbance measurement at 254 nm. Linearity was in the range of 25-2000 ng/band with correlation coefficients of 0.9998 for aceclofenac and 0.9996 for paracetamol. The limits of detection and quantification were 25 and 150 ng/band for aceclofenac and 50 and 200 ng/band for paracetamol. Both drugs were subjected to acid and alkali hydrolysis, oxidative degradation, and photodegradation. The degradation products were well resolved from the pure drug.

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

104 116 P. KHATWANI*, S. KULKARNI (*Bombay College of Pharmacy, Mumbai, Maharashtra, India): A sensitive high-performance thin-layer chromatography method for estimation of wedelolactone from Eclipta alba by different methods of extraction. Abstract No. F-272, 61st IPC (2009). HPTLC of wedelolactone on silica gel with toluene - ethyl acetate 9:1. Quantitative determination by fluorescence measurement at 366 nm. The method was linear in the range of 500-8000 ng/mL. Recovery was in the range of 100.2-101.0 %. The plant material was extracted using percolation, maceration, hot solvent extraction, supercritical fluid extraction, microwave assisted extraction, ultra sonication, and an orbital shaker. Quantification of wedelolactone in the extracts showed highest levels for Soxhlet extraction and lowest levels for supercritical fluid extraction.

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

104 117 A. KHODKE*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Pune, Maharashtra, India): A vlidated stability indicating HPTLC method for simultaneous estimation of irbesartan and hydrochlorothiazide. Abstract No. F-269, 61st IPC (2009). HPTLC of hydrochlorothiazide and irbesartan on silica gel with acetonitrile - chloroform 5:6. The hR_F value was 27 and 45 for irbesartan and hydrochlorothiazide, respectively. Quantitative determination by absorbance measurement at 270 nm. The sample was exposed to different stress conditions (acid, alkali, oxidative, photodegradation, thermal). Neither of the compounds showed degradation under thermal and photodegradation conditions, but both compounds showed significant degradation under acid, alkali and hydrolytic conditions. Degraded products were well resolved from the parent compounds.

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

104 118 E. KILINC*, F. AYDIN (*Department of Chemistry, Faculty of Art and Science, University of Dicle, Diyarbakir, 21280, Turkey; ekilinc@dicle.edu.tr): Stability-indicating HPTLC analysis of flurbiprofen in pharmaceutical dosage forms. J. Planar Chromatogr. 22, 349-354 (2009). HPTLC of flurbiprofen (2-(3-fluoro-4-phenyl)phenylpropanoic acid) and degradation products on silica gel, prewashed with methanol, with chloroform - acetone - xylene 5:2:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement (the authors report no wavelength). Linearity was between 50 and 600 ng/band. The limit of detection and quantification was 10 and 32 ng/band, respectively.

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

104 119 S. KOTHARI*, D. PATEL, N. SHAH, B. SUHAGIA (*Shri B. M. Shah College pf Pharma. Education and Research, Modasa, Gujarat, India): HPTLC method for simultaneous estimation of telmisartan and hydrochlorothiazide from their combination drug product. Abstract No. F-378, 61st IPC (2009). HPTLC of telmisartan and hydrochlorothiazide on silica gel with chloroform - toluene - methanol 2:5:5. The hR_F value was 53 and 75 for telmisartan and hydrochlorothiazide respectively. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 240-640 ng/band for telmisartan and 200-700 ng/band for hydrochlorothiazide.

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

104 120 Miroslawa KRAUZE-BARANOWSKA*, I. MALINOWSKA, D. GLOD, M. MAJDAN, A. WILCZANSKA (*Department of Pharmacognosy, Medical University of Gdansk, Gen. J. Hallera 107, 80-416, Poland, Krauze@amg.gda.pl): UTLC of flavonols in Sambucus nigra flowers. J. Planar Chromatogr. 22, 385-387 (2009). Ultrathin-layer chromatography of quercetin, rutin, and quercetin-3-O-glucoside on monolithic silica gel (size 30 mm x 18 mm) with binary and tertiary mobile phases in a cylindrical glass chamber previously saturated for 1 min. The migration distance was 20 mm and development time was 2 min. The investigated mobile phases were ethyl

acetate - *n*-hexane 1:4 and 3:7, tetrahydrofurane - hexane 2:3 and 3:2, tetrahydrofurane - methanol - hexane 3:3:4, and hexane - acetone - methyl ethyl ketone 3:3:4. The best separation was achieved with acetone - methyl ethyl ketone - hexane 3:4:3. Densitometric evaluation at 366 nm.

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

104 121 M. KRISHNA*, V. MURAGAN, P. MUSMADE, S. VENKATARAM (*Dayananda Sagar College of Pharmacy, Bangalore, Karnataka, India): Stability indicating high-performance thin-layer chromatography method for determination of triamcinalone acetonide in bulk drug and pharmaceutical dosage forms. 60th Indian Pharmaceutical Congress PA-211 (2008). HPTLC of triamcinalone acetonide on silica gel aluminum foil with toluene - ethyl acetate - ammonia 33:67:1 %.The hR_F value of triamcinalone acetonide was 38. Quantitative determination by absorbance measurement at 240 nm. The method was linear in the range of 100-2000 ng/spot; recovery was 99.5 %. The stability indicating method has been successfully applied to forced degradation studies of triamcinalone acetonide (acid, alkali, hydrogen peroxide, photo degradation thermal and neutral hydrolysis) and resolved degradation products and excipients from triamcinalone acetonide.

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

104 122 M. KUMAR*, B. SRINIVASAN (*Delhi Institute of Pharmaceutical Sciences and Research (DIPASR), New Delhi, India): Stability indicating HPTLC method for the determination of cinitapride hydrogen tartrate in bulk drug and pharmaceutical formulations. Abstract No. F-240, 61st IPC (2009). HPTLC of cinitapride hydrogen tartrate on silica gel with methanol - toluene 17:3. The hR_F value was 71. Quantitative determination by absorbance measurement at 265 nm. The linearity was in the range of 90-450 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidative, photodegradation, dry and wet heat) and degradation products were well separated from the main component.

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

104 123 R. KUNDU*, S. DHOLE, M. CHARDE, A. KASTURE (*J. L. Chaturvedi College of Pharmacy, Nagpur, Maharashtra, India): High-performance thin-layer chromatographic method for simultaneous estimation of benzhexol hydrochloride and trifluperazine hydrochloride in pharmaceutical preparations. Abstract No. F-248 61st IPC (2009). HPTLC of benzhexol HCl and trifluperazine HCl on silica gel with methanol - acetone - toluene - 25 % ammonia 10:10:70:1. The hR_F value was 37 and 82 for trifluperazine and benzhexol respectively. Quantitative determination by absorbance measurement at 210 nm. The method was linear in the range of 40-800 ng/band for benzhexol and 100-2000 ng/band for trifluperazine. Recovery was 99.7-99.9 % for both compounds.

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

104 124 N. LADKAT*, M. ANRUTKAR, J. JAGADE, A. KALE, S. PAWAR, A. BHOSALE (*Poona Dist. Education Asso. Seth Govind Raghunath Sable College of Pharmacy, Pune, Maharashtra, India): HPTLC estimation of cefixime and cloxacillin in tablet dosage form. Abstract No. F-260, 61st IPC (2009). HPTLC of cefixime and cloxacillin on silica gel, prewashed with methanol, with *n*-butanol - methanol - water - formic acid 80:60:40:3. The hR_F values were 28 and 45 for cefixime and cloxacillin, respectively. Quantitative determination by absorbance measurement at 293 nm for cefixime and 343 nm for cloxacillin. The linearity range was 150-600 ng/band for both compounds.

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

104 125 Q. LI (Li Qongya)*, J. WANG (Wang Jiaxin), Z. MA (Ma Zuo), SH. CHEN (Chen Shuhe), Y. LIU (Liu Yanwen), (*Joint State Key Lab of Minist. Educ. of Hubei Coll TCM & Head Off. of TCM Compound, Wuhan, Hubei 430061, China): (Qualitative and quantitative study of Alternanthera philoxeroides (Mart.) Griseb) (Chinese). Chinese J. Pharm. Anal. 28 (5), 732-734 (2008). TLC of Alternanthera philoxeroides extracts on silica gel with chloroform - methanol 40:1. The method is suitable for the quality control of the TCM drug and its formulations.

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

104 126 H. LI* (Li Hui), J. HU (Hu Jiangyu), H. OUYANG (Ouyang Hui), Y. LI (Li Yanan), H. SHI (Shi Hui), C. MA (Ma Chengjin), Y. ZHANG (Zhang Yongkang) (*Jishou University, Hunan Provinc Key Laboratory of Forest Products and Chemical Industry Engineering, Hunan Zhangjiajie, 427000, People's Rebublic of China, and Jishou University, College of Chemistry and Chemical Engineering, Hunan Jishou 416000, People's Republic of China; lihuijsdx@163.com): Extraction of aucubin from seeds of Eucomma ulmoides Oliv. using supercritical carbon dioxide. J. AOAC Int. 92, 103-110 (2009). Analytical and preparative TLC of aucubin and herbal extracts after extracton with supercritical carbon dioxide on silica gel with methanol - chloroform - petroleum ether - ethyl acetate 1:3:3:1. Visualization by spraying with 30 % sulfuric acid.

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

104 127 M. LIU (Liu Ming)*, G. LI (Li Gengsheng), H. WANG (Wang Huisheng) (*Henan Provin. Acad. Chinese Trad. Med. & Pharm., Zhengzhou, Henan 450004, China): (Study of the quality standard of Rehmannia glutinosa (Gdertn) Iibosch, a Chinese traditional herbal drug) (Chinese). Chinese J. Phram. Anal. 27 (9), 1311-1313 (2007). TLC of drug extracts on silica gel with trichloromethane - methanol - water 12:8:1. Detection by spraying with 5 % vanillin - sulfuric acid reagent and evaluation in daylight and under UV light.

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

104 128 H. LU (Lu Hui)*, H. XIE (Xie Hongping), SH. YANG (Yang Shilin), B. GU (Gu Bing) (*Pharm. Coll., Suzhou Univ., Suzhou, Jiangsu 215123, China): (Identification of leaf of Vitex negundo L. var. cannabifolia (Sieb. et Zucc.) Hand. -Mazz. and Oleum Viticis Negundo by thin-layer chromatography) (Chinese). J. Chinese Trad. Med. & Pharm. (Shizhen Guoyi Guoyao) 20 (11), 2799-2800 (2009). TLC of TCM drug extracts on silica gel with petroleum ether (60-90 ºC) - ethyl acetate 100:3. Detection by spraying with a solution of 5 % vanillin - 10 % sulfuric acid in ethanol 1:10 followed by heating at 105 °C until coloration, evaluation in visible and UV light.

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

104 129 K. LUNIYA*, R. MANTRI, S. DUBEY, S. BHARANI (*A.I.S.S.M.S. College of Pharmacy, Pune Maharashtra, India): Validated method development for estimation of naproxen sodium as bulk drug and in tablet dosage form by HPTLC. Abstract No. F-283, 61st (2009). HPTLC of naproxen sodium on silica gel with toluene - ethyl acetate - acetic acid 15:3:2. The hR_F value was 63. Quantitative determination by absorbance measurement at 230 nm. The method was linear in the range of 100-500 ng/band.

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

104 130 ROSIDAH, M. YAM*, A. SADIKUN, M. AHMAD, G.AKYIREM, M. ZAINI (*Department of Human Anatomy, Faculty of Medicine and Health Sciences, University Putra Malaysia, Serdang 43400, Selangor, Malaysia, yammunfei@yahoo.com) : Toxicology evaluation of standardized methanol extract of Gynura procumbens. J. Ethnopharmacol. 123, 244-249 (2009). HPTLC of kaempferol-3-O-rutinoside and astragalin in leaves of Gynura procumbens on silica gel with acetic acid - methanol - dichloromethane 1:3:7. Quantitative determination by absorbance measurement at 366 nm. The hR_F values of kaempferol-3-O-rutinoside and astragalin were 43 and 72, respectively, and selectivity regarding matrix was given. Linearity was given between 16 and 1000 µg/mL and the correlation coefficients were >0.987.

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

104 131 A. MADAN*, B. PATEL (*K. B. Institute of Pharmaceutical Education and Research Gandhinagar, Gujarat, India): HPTLC method for simultaneous determination of rabeprazole and itopride in capsules and its validation. Abstract No. F-244, 61st IPC (2009). HPTLC of rabeprazole and itopride on silica gel with ethyl acetate - methanol - benzene - chloroform 2:4:3:1. The hR_F value was 42 and 61 for rabeprazole and itopride, respectively. Quantitative determination by absorbance measurement at 276 nm. The method was linear in the range of 200-300 ng/band for rabeprazole and 1500-2250 ng/band for itopride.

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

104 132 V. MADHAVAN*, S. YOGANARSIMHAN, R. TIJARE (*M. S. Ramaiah College of Pharmacy,Bangalore, India): Pharmacognostical and phytochemical studies of roots tubers of Asparagus gonoclados Baker. Abstract No. 9966, IHCB (2009). HPTLC of root tuber extracts of Asparagus gonoclados Baker on silica gel with ethyl acetate - methanol - water 15:3:2. Detection by spraying with anisaldehyde reagent. Quantitative determination by absorbance measurement at 425 nm. Shatavarin IV was used as marker.

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

104 133 U. MALLAVADHANI*, G. SAHU (*Herbal Drugs and Bio-Remedies, Institute of Minerals and Material Technology (CSIR), Bhubaneswar-751 013, Orissa, India; uvmavadani@yahoo.com): A rapid HPTLC method for standardization of Ficus bengalensis Linn. J. Planar Chromatogr. 22, 377-380 (2009). HPTLC of stigmast-5-en-3beta-O-D-glucoside and bark extracts on silica gel with chloroform - methanol - water 33:7:4 in a saturated twin trough chamber. Detection by derivatization with anisaldehyde reagent followed by heating at 105 °C for 5 min. Quantitative determination by absorbance measurement at 515 nm.

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

104 135 K. MANGUKIA*, T. VAJA, Hasumati RAJ, Sadhana RAJPUT (*N. R. Vekaria Institute of Pharmacy & Research Centre Junagadh, Gujarat, India): Development and validation of stability-indicating HPLC and HPTLC methods for analysis of ezetimibe in pure form and in pharmaceutical formulation. Abstract No. F-320, 62st IPC (2009). HPTLC of ezetimibe on silica gel with ethyl acetate - toluene - methanol - formic acid 10:10:1:1. Quantitative determination by absorbance measurement at 231 nm. The method was linear in the range of 301-3610 ng/band. Recovery was 99.3-100.4 % The drug was exposed to different stress conditions (acid, base, oxidative, thermal) and all degradation products were well resolved from the main compound.

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

104 136 T. MANI*, S. BADAMI, N. MAHADEVAN, S. MANIMARAN, B. SURESH (*Bharathi College of Pharmacy, Bharathi Nagara, Karnataka, India): Determination of harmalin in Passiflora edulis leaves by HPTLC. Abstract No. 9274, IHCB (2009). HPTLC of harmalin from ethanolic leaf extracts of Passiflora edulis on silica gel with ethyl acetate - acetic acid - formic acid - water 100:11:11:27. The hR_F value of harmalin was 39. The method was linear in the range of 200-1600 ng/spot, recovery was 98.4-99.2 %.

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

104 137 S. MANIMARAN*, M. CHAITANYA, T. PRAVEEN, S. DHANABAL (*JSS College of Pharmacy, Ootacamund, Tamil Nadu, India): Method validation and estimation of isovitexin content in Passiflora incarnata Linn by HPTLC technique. 60th Indian Pharmaceutical Congress PG-258 (2008). HPTLC of isovitexin in Passiflora incarnata raw material and extract on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:26. The plant was found to contain 0.087 % w/w of isovitexin.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 138 R. MANTRI*, M. SENGAR, U. PATIL, S. GANDHI (*A.I.S.S.M.S. College of Pharmacy, Pune, Maharashtra, India): High-performance thin-layer chromatographic determination of diclofenac sodium and thiocolchicoside in fixed dose combination. Abstract No. F-284, 61st IPC (2009). HPTLC of diclofenac sodium and thiocolchicoside on silica gel with toluene - ethyl acetate - methanol 5:3:2. The hR_F value was 17 and 53 for thiocolchicoside and diclofenac sodium, respectively. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 50-300 ng/band for both drugs. Recovery was 100.5-101.1 %.

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

104 139 A. MASLANKA. J. KRZEK*, M. STOLARCZYK (*Department of Inorganic and Analytical Chemistry, Jagiellonian University, Collegium Medicum, 9 Medyczna Street, 30-688 Cracow, Poland, jankrzek@cm-uj.krakow.pl): Simultaneous analysis of hydrochlorothiazide, triamterene, furosemide, and spironolactone by densitometric TLC. J. Planar Chromatogr. 22, 405-410 (2009). TLC of hydrochlorothiazide, triamterene, furosemide, and spironolactone on silica gel with hexane - ethyl acetate - methanol - water - acetic acid 42:40:15:2:1 with chamber saturation. Quantitative determination by absorbance measurement at 264 nm. The limit of detection for the different compounds was between 22 and 150 ng/band, and the limit of quantification was between 68 and 450 ng/band.

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

104 140 Annie MATHEW*, R. RAVINDRA (*C. U. Shah College of Pharmacy, S.N.D.T. Women's University, Juhu Road, Santacruz (W) Mumbai, India): Quantitative HPTLC analysis of dially disulfide in garlic oil macerate. Abstract No. 9286, IHCB (2009). HPTLC of dially disulfide in garlic oil macerate on silica gel with *n*-hexane - isopropyl alcohol - formic acid 196:4:3. Quantitative determination by absorbance measurement at 210 nm. The method was linear in the range of 16-48 µg/spot.

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

104 141 S. MATHUR*, D. SHARMA, P. SAINI, R. SINGH (*R & D Div., Indian Pharmacopoeia Commission, Ministry of Health & Family Welfare, Govt. of India, Ghaziabad, U.P., India): Simultaneous estimation of domperidone and paracetamol in bulk and its tablets dosage forms by HPTLC method. Abstract No. F-395, 61st IPC (2009). HPTLC of domperidone and paracetamol on silica gel with acetone - toluene - methanol 2:2:1 with chamber saturation at room temperature. The hR_F value was 52 and 74 for domperidone and paracetamol, respectively. Quantitative determination by absorbance measurement at 248 nm (paracetamol) and 285 nm (domperidone). The method was linear in the range of 16-48 ng/band. Recovery was 99.5-101.2 %.

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

104 142 Dipali MEHETRE*, A. CHABUKSWAR, B. KUCHEKAR, A. KATEGAONKAR (*MAEER's Maharashtra Institute of Pharmacy, Pune, Maharashtra, India): Validation of HPTLC method for simultaneous quantitation of olmesartan medoximal and amlodipine besylate in bulk drug and formulation. Abstract No. F-258, 61st IPC (2009). HPTLC of olmesartan medoximal and amlodipine besylate on silica gel with chloroform - methanol - toluene - acetic acid 80:10:1:1. Quantitative determination by absorbance measurement at 254 nm. The linearity range was 800-5000 ng/band for olmesartan and 200-1400 ng/band for amlodipine. Recovery was in the range of 98-102 % for both drugs.

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

104 143 A. MISHRA*, R. BHOMIA, S. VASANTHARAJU, A. KARTHIK, S. SAYED, K. BHAT (*L. M. College of Pharmacy, Ahmedabad, Gujarat, India): Simultaneous estimation of salbutamol silphate and guaiphenesin in their combined liquid dosage form by HPTLC method. Abstract No. F-238, 61st IPC (2009). HPTLC of salbutamol silphate and guaiphenesin, used as pharmaceutical

syrup against cough, on silica gel with ethyl acetate - methanol - 25 % ammonia 15:3:2. The hR_F value was 47 and 65 for salbutamol and guaiphenesin, respectively. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 200-1000 ng/band for salbutamol and 10-15 µg/band for guaiphenesin.

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

104 144 A. MISHRA*, R. BHOMIA, S. VASANTHARAJU, A. KARTHIK, S. SYED, K. BHAT (*Manipal College of Pharmaceutical Sciences, Manipal Univeristy, Manipal, Karnataka, India): Stability-indicating HPTLC method for the estimation of tolterodine in bulk drug. Abstract No. F-237, 61st IPC (2009). HPTLC of tolterodine on silica gel with toluene - methanol - 25 % ammonia 250:250:1. The hR_F value was 40. Quantitative determination by absorbance measurement at 284 nm. The method was linear in the range of 200-1000 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidation, thermal) and degradation under alkaline conditions was observed. The degradation products were well separated from the main component.

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

104 145 S. MISHRA*, Sunita CHAUDHARY, K. GADHVI (*Saraswati Institute of Pharmaceutical Sciences, Ahmedabad, Gujarat, India): Estimation of glycyrrhizic acid and withanolide A in polyherbal formulation by HPTLC. 60th Indian Pharmaceutical Congress PA-233 (2008). HPTLC of glycyrrhizic acid on silica gel with toluene - ethyl acetate - glacial acetic acid 25:15:1. The hR_F value of glycyrrhizic acid was 52, linearity was in the range of 500-1000 ng/mL and recovery was 99.2 %. Withanolide A was separated with toluene - ethyl acetate - glacial acetic acid 20:20.1. The hR_F value of withanolide A was 44, linearity was in the range of 400-1000 ng/mL and recovery was 99.4 %.

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

104 146 K. MODT*, N. PATEL, R. GOYAL (*Dept. of Pharmacology, Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa, Gujarat, India): A sensitive HPTLC method for the estimation of L-dopa from Muccuna pruriens Linn and a formulation containing M. pruriens. Abstract No. 9425, IHCB (2009). HPTLC of L-dopa in Mucuna pruriens seed extract and formulations on silica gel with *n*-butanol - acetic acid - water 4:1:1. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 100-1200 ng/spot with an average recovery of 100.3 %.

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

104 147 P. MUCAJI*, M. NAGY, T. LIPTAJ, N. PRÓNAYOVÁ, E. SVAJDLENKA (*Department of Pharmacognosy and Botany, Faculty of Pharmacy, Comenius University, Odbojárov 10, 832 32 Bratislava, Slovak Republic, mucaji@fpharm.uniba.sk): Separation of a mixture of luteolin-7-rutinoside and luteolin-7-neohesperidoside isolated from Ligustrum vulgare L. J. Planar Chromatogr. 22, 301-304 (2009). Analytical and preparative TLC of luteolin-7-rutinoside and luteolin-7-neohesperidoside on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:23, on cellulose with 30 % acetic acid, and on polyamide with chloroform - methanol - 2-butanone - acetyl acetone (= pentane-2,4-dione) 9:4:3:1. Detection by spraying with natural products reagent followed by treatment with PEG 4000, or by aniline phthalate.

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

104 148 A. MUJTABA*, S. BABOOTA, J. ALI, K. KOHLI (*Dept. of Pharmaceutics, Faculty of Pharmacy, Hamdard University, New Delhi, India): Development and validation of novel HPTLC method for quantitative estimation of ondansetron HCl in bulk and pharmaceutical dosage form. Abstract No. F-274, 61st IPC (2009). HPTLC of ondansetron HCl on silica gel with chloroform - ethyl acetate - methanol - 25 % ammonia 90:50:40:1. The hR_F value was 52. Quantitative de-

termination by absorbance measurement at 254 nm. The method was linear in the range of 100-1400 ng/band. Recovery was 99.3 %.

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

104 149 B. NARWATE*, P. GHULE, P. MOHITE, R. UGALE (*Dept. of Pharmaceutical Chem., MES College of Pharma., Sonai, Tal.-Newasa, Dist.-Ahmednagar 414105 (M.S.) India., balaji_narwate@rediffmail.com): A high-performance thin-layer chromatographic determination of clopidogrel bisulphate in tablets. Ind. J. Pharma. Sci. 8(4), 211-212 (2009). TLC of clopidogrel bisulphate on silica gel with carbon tetrachloride - ethyl acetate - ammonia 50:3:2. Quantitative determination by absorbance measurement at 230 nm. Linearity was between 300 and 1500 ng. In comparison with the labeled claim the amount of clopidogrel in tablets was 99.2 %. The recovery was 99.2 % (via peak area).

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

104 150 J. NIRMAL*, S. MAHESHWARI, H. RAJ, S. RAJPUT (*N.R. Vekaria Institute of Pharmacy & Research Centre, Junagadh, Gujarat, India): Development and validation of stability-indicating HPLC and HPTLC methods for analysis of pravastatin in pure form and application of the methods for estimation of pharmaceutical formulation. Abstract No. F-315, 61st IPC (2009). HPTLC for pravastatin on silica gel with ethyl acetate - toluene - acetonitrile - formic acid 60:35:5:2. Quantitative determination by absorbance measurement at 237 nm. The method was linear in the range of 318-3816 ng/band. Recovery was 99.9-101.2 %. Stability tests showed that degradation products resulting under acid stress conditions were well resolved from the main component.

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

104 151 K. NISHAMOL*, A. BINDU, N. ALEYKUTTY, S. JOSE (*M. G. Univeristy, Dept. of Pharmaceutical Sciences, Ettumanoor, Kottayam, Kerala, India): Development of a high-performance thin-layer chromatography method for the quantitative estimation of rutin in the fresh leaves of Moringa pterygosperma Gaertn. 60th Indian Pharmaceutical Congress PG-263 (2008). HPTLC of rutin in methanolic extracts of fresh leaves of Moringa pterygosperma on silica gel with ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:26. Quantitative determination by absorbance measurement at 254 nm. The method provided good resolution of rutin from other constituents of the plant.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 152 D.N. OLENNIKOV (Laboratory of Medical and Biological Research, Department of Biologically Active Substances, Institute of General and Experimental Biology, Siberian Division, Russian Academy of Sciences, Sakh'yanovoy St 6, 670047, Ulan-Ude, Russia; oldaniil@rambler.ru): Densitometric HPTLC analysis of aloenin in aloe pharmaceuticals. J. Planar Chromatogr. 22, 359-362 (2009). HPTLC of aloenin in aloe juice, tablets, and liquid extracts on silica gel with ethyl acetate - 95 % ethanol - water 20:3:1 at room temperature in a saturated chamber. Detection by immersion for 1 s in freshly prepared 5 % sodium hydroxide solution in 95 % ethanol, followed by heating at 100 °C for 5 min. Quantitative determination by absorbance measurement at 365 nm.

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

104 153 A. OSMAN*, M. OSMAN (*National Organization for Drug Control and Research, 6 Abu Hazem St Pyramids, PO Box 29, Cairo, Egypt; afaf_osmanelteti@yahoo.com): Specrofluorometry, thin-layer chromatography, and column high-performance liquid chromatography determination of rabeprazole sodium in the presence of its acidic and oxidized degradation products. J. AOAC Int. 92, 1373-1381 (2009). TLC of rabeprazole sodium and its degradation products on silica gel with isopropanol - 30 % ammonia 40:1 with chamber saturation. Quantitative determination by absorbance measurement at 284 nm.

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

104 154 M. PAI*, Rajashree GUDE, Swati KENY (*Goa College of Pharmacy, Panaji, Goa, India): Development and validation of a new sensitive method for the quantitative analysis of ranitidine hydrochloride and domperidone in antiulcer combination by using HPTLC. 60th Indian Pharmaceutical Congress PA-215 (2008). HPTLC of ranitidine HCl and domperidone in combined dosage form on silica gel with ethyl acetate - methanol - ammonia 100:10:1 in a twin trough chamber saturated for 10 min. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 100-500 ng/µL for both compounds with a recovery of 102.5-100.8 %.

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

104 155 H.J. PANCHAL*, B.N. SUHAGIA, N.J. PATEL (*Patel College of Pharmaceutical Education and Research, Ganpat Vidyanagar, Kherva, Mehsana 382711, Gujarat, India; hiral.panchal@ganpatuniversity.ac.in): Simultaneous HPTLC analysis of atorvastatin calcium, ramipril, and aspirin in a capsule dosage form. J. Planar Chromatogr. 22, 265-271 (2009). HPTLC of atorvastatin calcium, ramipril, and aspirin and extracts of pharmaceutical formulations on silica gel, prewashed with methanol, with methanol - benzene - ethyl acetate - glacial acetic acid 9:140:100:1 in a twin trough chamber, saturated with mobile phase for 30 min at room temperature. Quantitative determination by absorbance measurement at 210 nm. The limit of detection was 5 ng/zone for atorvastatin calcium, 3 ng/zone for ramipril, and 19 ng/zone for aspirin.

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

104 156 M. PANDE*, Shweta GONDKAR, J. RAO, S. YADAV (*Poona College of Pharmacy, Bharati Vidyapeeth University, Pune, Maharashtra, India): Simultaneous determination of tenofovir and emtricitabine in the bulk drug and tablet dosage form by HPTLC method. Abstract No. F-253, 61st IPC (2009). HPTLC of tenofovir and emtricitabine on silica gel with toluene - methanol - ethyl acetate - acetic acid 40:20:50:1. Quantitative determination by absorbance measurement at 273 nm. The hR_F value was 52 for tenofovir and 40 for emtricitabine. Linearity of tenofovir and emtricitabine was in the range of 120-600 ng/spot and 80-560 ng/spot, respectively. The recovery was 99.9 and 99.5 % for tenofovir and emtricitabine, respectively.

quality control, HPTLC, densitometry, quantitative analysis 32a

104 157 P. PARMAR*, Ankita MEHTA (L. M. College of Pharmacy, Navrangpura, Gujarat 380009, India, parul1383@gmail.com): Development and validation of HPTLC method for the estimation of clotrimazole in bulk drug and tablet formulation. Ind. J. Pharma. Sci. 71(4), 451-454 (2009). HPTLC of clotrimazole in bulk drug and tablet dosage form on silica gel with cyclohexane - toluene - methanol - triethyl amine 80:20:5:2. Quantitative determination by absorbance measurement at 262 nm. The calibration curve was linear between 200 to 1000 ng/spot for clotrimazole. The limit of detection and limit of quantification for clotrimazole were 50 ng/spot and 200 ng/spot, respectively.

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

104 160 A. PATEL*, B. DHANYA, A. SEN, A. SETH (*Dept. of Pharma., Sumandeep Vidyapeeth University, Vadodara, Gujarat, India): Development and validation of a HPTLC method for estimation of doxazosin mesylate in tablet dosage form. Abstract No. F-251, 61st IPC (2009). HPTLC of doxazosin mesylate on silica gel with acetone - toluene - 25 % ammonia 60:40:1. The hR_F value was 65. Quantitative determination by absorbance measurement at 251 nm. Linearity was in the range of 20-100 ng/band. Recovery was 103.3 %.

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

104 159 B. PATEL*, A. MODH, P. MEHTA, H. BHATT (*Institute of Pharmacy, Nirma University Science and Technology, Ahmedabad, Gujarat, India): Development and validation of spectrophotometric and HPTLC method for simultaneous estimation of levocetirizine dihydrochloride and

montelukast sodium in their combined dosage form. Abstract No. F-311, 61st IPC (2009). HPTLC of levocetirizine dihydrochloride and montelukast sodium on silica gel with chloroform - methanol 93:7. The hR_F value was 21 and 65 for levocetrizine and montelukast, respectively. Quantitative determination by absorbance measurement at 345 nm. The method was linear in the range of 100-350 ng/band for levocetrizine and 600-1100 ng/band for montelukast.

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

104 161 C. PATEL*, B. PATEL, P. PATEL, C. PATEL (*Shri Sarvajanik Pharmacy College, Hemchandracharya North Gujarat University, Mehsana, Gujarat, India): Development and validation of a simultaneous HPTLC method for the estimation of atorvastatin calcium and amlodipine besilate in tablet dosage form. 60th Indian Pharmaceutical Congress PA-213 (2008). HPTLC of atorvastatin calcium and amlodipine besilate on silica gel with chloroform - toluene - methanol - water 55:10:20:2. Quantitative determination by absorbance measurement at 242 nm. The calibration curve was found to be linear between 400 and 1200 ng/spot for both atorvastatin calcium and amlodipine besilate. The limit of detection and the limit of quantification for atorvastatin calcium were 100 and 400 ng/spot, and for amlodipine besilate 200 and 400 ng/spot, respectively.

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

104 173 D. PATEL*, S. HEMALATHA, SURESH B., S. DHANABAL (*Dept. of Pharmacognosy. J.S.S. College of Pharmacy, Oacamund 643001, Tamil Nadu, India): Phytochemical standardization and fingerprinting analysis of Berberis aristata extract by HPTLC. Abstract No. 9185, IHCB (2009). HPTLC of hydro alcoholic extracts of Berberis aristata on silica gel with benzene - ethyl acetate - diethyl amine 6:3:1. Detection by spraying with $AlCl_3$ reagent (for estimation of flavonoids) or with Folin Ciocalteu reagent (for total phenolic content). The fingerprint profile was optimized using two different mobile phases: *n*-butanol - acetic acid - water 14:1:5 and *n*-propanol - formic acid - water 90:1:9. The extract showed 6 different spots and was found to contain 13.5 % w/w of berberin.

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

104 162 D. PATEL*, B. SHAH, B. PATEL (*K. B. Institute of Pharmaceutical Education and Research Gandhinagar, Gujarat, India): Simultaneous estimation of atorvastatin calcium, ramipril and aspirin in capsule dosage form by HPTLC. Abstract No. F-245, 61st IPC (2009). HPTLC of atorvastatin (AT) calcium, ramipril (RA) and aspirin (AS) on silica gel with benzene - ethyl acetate - toluene - methanol - acetic acid 40:45:10:5:1. Quantitative determination by absorbance measurement at 220 nm. The hR_F values were 45, 28 and 72 for AT, RA and AS, respectively. The linearity ranges were 0.5-2.5 µg/band (r^2=0.998) for AT, 0.5-2.5 µg/band (r^2=0.9978) for RA and 0.75-3.75 µg/band (r^2=0.9946) for AS with mean recoveries of 100.3, 99.1 and 98.9 for AT, RA and AS, respectively.

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

104 164 F. PATEL*, J. DONGA, N. PATEL, V. GANDHI (*Dharmaj Degree Pharmacy College, Dharmaj, Gujarat, India): Determination of camylofin dihydrochloride in bulk and tablet by liquid chromatography and HPTLC. Abstract No. F-267, 61st IPC (2009). HPTLC of camylofin dihydrochloride on silica gel with chloroform - ethyl acetate - methanol - 25 % ammonia 50:30:20:1. Quantitative determination by absorbance measurement at 215 nm. Linearity was in the range of 1500-7500 ng/band. The proposed method is suitable for routine quality control of bulk drug and tablets.

pharmaceutical research, HPTLC, densitometry, quantitative analysis 32a

104 165 J. PATEL*, K. BHAT, F. SHAIKH, S. PANDYA (*Babaria Institute of Pharmacy, Varnama, Vadodara, Gujarat, India): Simultaneous determination of strychnine and piperine in their combined

herbal dosage form by HPTLC. 60th Indian Pharmaceutical Congress PA-192 (2008). HPTLC of strychnine and piperine in herbal extracts and herbal formulations on silica gel with toluene - ethyl acetate - diethyl amine 7:2:1. Quantitative determination by absorbance measurement at 283 nm. Linearity was 400-2000 ng/spot, recovery was in the range of 99.5-101.0 both for strychnine and piperine.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 158 J.B. PATEL, S.K. LAHIRI, M.B. SHAH* (*Department of Pharmacognosy, L. M. College of Pharmacy, Navarangpura, Ahmedabad (Gujarat), 380009, India; mbshah2007@rediffmaol-com): Development of a new method for identification af Withania somnifera root, and a method for quantitative analysis of withaferin A in young and old roots. J. Planar Chromatogr. 22, 283-286 (2009). HPTLC of withaferin and extracts of the powdered root on silica gel, prewashed with methanol, with toluene - ethyl acetate - acetone 2:3:3 in a twin trough chamber saturated with mobile phase for 30 min. Detection by spraying with anisaldehyde reagent followed by heating for 15 min at 105 °C; characteristic orange fluorescence was observed for whithaferin. Quantitative determination by absorbance measurement at 214 nm. The limit of detection and quantification for withaferin A was 258 and 782 ng/zone, respectively.

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

104 170 M. PATEL*, P. MANDLEKAR, S. MULGUND, K. JAIN (*Sinhgad College of Pharmacy, Pune, Maharashtra, India): Simultaneous HPTLC determination of ramipril hydrochlorothiazide and telmisartan in combined tablets. Abstract No. F-239, 61st IPC (2009). HPTLC of hydrochlorothiazide, ramipril and telmisartan on silica gel with ethyl acetate - chloroform - methanol 6:3:1. Quantitative determination by absorbance measurement at 215 nm. The method was suitable for routine quality control of combined formulations.

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

104 163 N. PATEL*, G. PATEL, H. BHATT, C. SHASTRY (*Shree Dhanvantry Pharmacy College, Surat, Gujarat, India): HPTLC method for simulteneous determination of aspirin and atorvastatin in pharmaceutical formulation. Abstract No. F-293, IPC (2009). HPTLC of aspirin and atorvastatin on silica gel with *n*-hexane - acetone - butyl acetate - formic acid 60:30:12. Quantitative determination by absorbance measurement at 242 nm. For both drugs, the method was linear in the range of 3-7 µg/band and the recovery was between 99.3 and 101.0 %.

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

104 171 P. PATEL*, R. MASHRU (*Babaria Institute of Pharmacy, Vadodara, Gujarat, India): Two dimensional thin layer chromatography (2D-TLC) for resolution of isomers of (±) bupropion HCl. Abstract No. F-254, 61st IPC (2009). HPTLC of bupropion HCl on silica gel with quinine sulphate - methanol - water 13:20:12 in the first direction. Quinine sulphate was used as a chiral selector at a concentration of 3.5 mM. The two separated bands were detected under UV 366 nm. The hR_F values of I (-) and d (+) isomers of bupropion HCl were 90 and 84, respectively. After the second run in the second dimension with methanol - water 80:13 they were better resolved with hR_F values of 88 and 76.

HPTLC, qualitative identification 32a

104 172 P. PATEL*, R. MASHRU, T. PATEL (*Babaria Institute of Pharmacy, Varnama, Vadodara, Gujarat, India): Development and validation of a direct HPTLC method for separation of isomers of (±) bupropion HCl using quinine sulphate as a chiral selector in mobile phase. 60th Indian Pharmaceutical Congress PA-216 (2008). HPTLC of isomers of bupropion HCl on silica gel with quinine sulphate - methanol - water 13:20:12 (quinine sulphate served as a chiral selector). Evaluation under UV 366 nm. Linearity was in the range of 10-100 µg/spot for d(+)- and l(-)-isomers of bupropion. The isomer ratio was 80 % d(+)-bupropion and 20 % l(-)-bupropion.

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

104 169 P. PATEL*, N. PATEL, R. GOYAL (*Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa, Gujarat, India): Quality control of polyherbal formulations used in diabetes mellitus. 60th Indian Pharmaceutical Congress PG-246 (2008). HPTLC of biomarkers such as curcumin, charantin, and swertiamarin in some 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 swertiamarin). Quantitative determination by absorbance measurement at 536 for charantin (hR_F value 33), 425 nm for curcumin (hR_F value 89), and 238 nm for swertiamarin (hR_F value 54).

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

104 167 R. PATEL*, Mrunali PATEL, K. BHATT, B. PATEL (*A. R. College of Pharmacy and G. H. Patel Institute of Pharmacy, Vallabh Vidyanagar, Gujarat, India): Development and validation of HPTLC method: its application in qualification of olanzapine in mucoadhesive microemulsion formulations and invitro study. Abstract No. F-235, 61st IPC (2009). HPTLC of olanzapine on silica gel with methanol - ethyl acetate 4:1. The hR_F value was 35. Quantitative determination by absorbance measurement at 285 nm. The method was linear in the range of 100-600 ng/band. The method was suitable for analysis of formulations and in-house prepared mucoadhesive microemulsions.

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

104 166 R.B. PATEL*, M.R. PATEL, M.B. SHANKAR, K.K. BHATT (*Sardar Patel Univrsity, A. R. College of Pharmacy & G. H. Patel Institute of Pharmacy, Vallabh Vidyanagar 388120, Gujarat, India; rashmru@gmail.com): Simultaneous determination of alprazolam and fluoxetine hydrochloride in tablet formulations hy high-performance column liquid chromatography and high-performance thin-layer chromatography. J. AOAC Int. 92, 1082-1087 (2009). HPTLC of alprazolam and fluoxetine hydrochloride in pure powder and formulations on silica gel with acetone - toluene - ammonia 12:7:1 in a twin trough chamber saturated for 30 min. Quantitative determination by absorbance measurement at 230 nm. There was no significant difference in the determined content of alprazolam and fluoxetine by HPTLC and HPLC methods (assay results compared by applying the paired t-test).

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

104 168 S. PATEL*, N. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganapat University, Mehsana, Gujarat, India): HPTLC estimation of amitriptyline HCl, trifluoperazine HCl, risperidone and alprazolam in pharmaceutical products using single mobile phase. 60th Indian Phamraceutical Congress PA-210 (2008). HPTLC of amitriptyline HCl, trifluoperazine HCl, risperidone and alprazolam on silica gel with carbon tetrachloride - acetone - triethylamine 80:20:3. Quantitative determination by absorbance measurement at 250 nm in the range of 50-1200 ng/spot for amitriptyline HCl, 50-1200 ng/spot for trifluoperazine HCl, 100-2400 ng/spot for risperidone, and 25-600 ng/spot for alprazolam. The limit of quantification for amitriptyline HCl and trifluoperazin HCl was 50 ng/spot, for risperidone 100 ng/spot, and for alprazolam 25 ng/spot.

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

104 174 P. PATIDAR *, S. MANIMARAN, N. SONI, S. DHANABAL (*J S S College of Pharmacy, Ooty, Tamil Nadu, India): Simultaneuos HPTLC estimation of quercetin and rutin from Tylophora indica and Tephrosia purpurea. 60th Indian Pharmaceutical Congress PG-261 (2008). HPTLC of quercetin and rutin in ethanolic extracts of aerial parts of Tylophora indica and Tephrosia purpurea on silica gel with ethyl acetate - formic acid - acetic acid - water 100:11:11:26. Quantitative determination by fluorescence measurement at 366/>400 nm. The extract of Tephrosia purpurea

contained 1.56 % of quercetin and 1.40 % of rutin, whereas Tylophra indica contained 4.30 % of quercetin.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 175 N.G. PATRE, L. SATHIYANARAYANAN, M.V. MAHADIK, S.R. DHANESHWAR* (*Department of Pharmaceutical Chemistry, Bharati Vidyapeeth University, Poona College of Pharmacy, Center for Advanced Pharmaceutical Research, Erandwane, Pune 411038, Maharashtra State, India; Sunil.dhaneshwar@gmail.com): A validated, stability-indicating HPTLC method for analysis of doxofylline. J. Planar Chromatogr. 22, 245-348 (2009). HPTLC of doxofylline (7-(1,3-dioxalan-2-ylmethyl)theophylline) in bulk drug and in formulations on silica gel, prewashed with methanol, with toluene - methanol 4:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 275 nm.

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

104 176 M. PENG (Peng Minjie)*, D. LI (Li Duowei), Y. WANG (Wang Yichao), SH. JIA (Jia Shaoliang) (*Inst. Life Sci., North-West University, Xi'an 710069, China): (Determination of scalreol in its formulations by thin-layer chromatography) (Chinese). Chinese J. Pharm. Anal. 28 (9), 1554-1556 (2008). TLC of scalreol on silica gel with *n*-hexane - ethyl acetate - formic acid 32:16:1. Detection by spraying with vanillin - sulfuric acid - ethanol 1:1:18. Quantification by densitometry at 520 nm. Linearity was between 10 and 50 µg/zone with a determination coefficient of 0.9994. Recovery was 100.1 % (n = 6, RSD = 0.9 %). Repeatability (*%RSD*, n = 6) was 2.1 % within plate and 2.3 % plate-to-plate.

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

104 177 CH. PIAO (Piao Chunmei)*, X. QU (Qu Xiangling), X. ZHOU (Zhou Xunrong) (*Affiliated Hosp. No.2, Guizhou Inst. TCM, Guiyang, Guizhou 550003, China): (Identification procedure for Tongmaitang Yanming capsuls) (Chinese). Chinese J. Hosp. Pharm. 29 (4), 1246-1247 (2009). TLC of TCM drug extracts on silica gel with 1) chloroform - methanol - acetone 10:1:1; 2) toluene - chloroform - acetone - methanol - formic acid 4:6:8:1:4; 3) chloroform - methanol 4:1. Detection 1) after exposure to ammonia vapor under UV 254 nm; 2) under UV 254 nm; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration.

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

104 178 L. POTALE*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Kenned Raod, Pune, Maharashtra, India): A validated stability indicating HPTLC method for simultaneous estimation of telmisartan and rampril. Abstract No. F-271, 61st IPC (2009). HPTLC of telmisartan and rampril on silica gel with methanol - chloroform 1:6. The hR_F value was 38 and 68 for rampril and telmisartan, respectively. Quantitative determination by absorbance measurement at 210 nm. The sample was exposed to different stress conditions (acid, alkali, oxidative, photo degradation and thermal). Both drugs did not degrade under acidic and photolytic conditions, but showed significant degradation under alkaline and thermal conditions. Both compounds were well separated from different degradation products under experimental conditions.

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

104 179 Kirti PRABHU*, R. LOBO, Richa AGRAWAL, A. SHIRWAIKAR, A. SHIRWAIKAR, Mamatha BALLAL (*Dept. of Pharmacognosy, Manipal College of Pharmaceutical Science, Manipal University, Manipal, India): Application of a stability-indicating HPTLC method for the quantitative determination of hesperidin in pharmaceutical dosage form. Abstract No. 9324, IHCB (2009). HPTLC of hesperidin in orange peel extract and formulation on silica gel with ethyl acetate - methanol - water 100:17:13. Quantitative determination by absorbance measurement at 287 nm. The method was linear in the range of 10-1000 ng/spot. Hesperidin was subjected to degradation studies (acid, alkali, hydrolysis, oxidation, and thermal stress) and was found susceptible to diffe-

rent stress condition. The method was suitable for determination of hesperidin and its degradation products in bulk drug as well as formulations.

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

104 180 S.L. PRABU*, T. SINGH, C.D. KUMAR, A. JOSEPH, K.K. SRINAVASAN (*Manipal College of Pharmaceutical Sciences, Manipal 576104, India; slaxmanvel@gmail.com): High-performance thin-layer chromatographic method for analysis of racecadotril in the bulk drug. J. Planar Chromatogr. 22, 277-281 (2009). HPTLC of racecadotril (2-{2(acetylsulfanylmethyl)-3-phenylpropanoyl}amino acetic acid benzyl ester) and its degradation products in the bulk drug and in a pharmaceutical formulation on silica gel with *n*-hexane - ethyl acetate 7:3 in a twin trough chamber saturated for 30 min. Quantitative determination by absorbance measurement at 230 nm. The limit of detection and quantification was 50 and 100 ng/band, respectively.

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

104 181 N. PRAJAPATI*, V. YADAV, S. PANCHOLI (*S. K. Patel College of Pharmaceutical Education and Research, Mehsana, Gujarat, India): Development and validation of HPTLC method for determination of repaglinide and rosiglitazone maleate in combined dosage form. Abstract No. F-263, 61st IPC (2009). HPTLC of repaglinide (REPA) and rosiglitazone (ROSI) on silica gel with benzene - methanol - acetone - acetic acid 80:10:9:1. Quantitative determination by absorbance measurement at 266 nm. Lienarity was in the range of 800-2800 ng/band and 400-2400 ng/band for REPA and ROSI, respectively. Recovery was 101.7 and 99.5 % for REPA and ROSI, respectively.

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

104 182 P. PUSHPALATHA*, R. K. SARIN, M. A. RAO, T. R. R. BAGGI (*Central Forensic Science Laboratory, Directorate of Forensic Science, Ministry of Home Affairs, Government of India, Ramanthapur, Hyderabad 500 013, India; sarinrk@yahoo.com): A new thin-layer chromatographic method for analysis of zolpidem and zoplicone. J. Planar Chromatogr. 22, 449-451 (2009). TLC of zolpidem and zoplicone on silica gel with methanol - triethylamine 39:1, acetonitrile - triethylamine 39:1, chloroform - methanol - triethylamine 38:2:1, and acetonitrile - methanol - triethylamine 34:4:1 with chamber saturation. Detection by spraying with chloranilic acid reagent (0.5 chloranilic acid in dioxane) and evaluation of colored zones in daylight.

toxicology, qualitative identification 32a

104 183 Y. QI (Qi Yanfei)*, Q. GONG (Gong Qing), M. LU (Lu Min) (*Zhejiang Provin. Inst. Food & Med., Hangzhou, Zhejiang 310004, China): (Study of the quality standard for Qingshen Jianfei pills) (Chinese). J. Chinese Trad. & Herb. Med. 39 (12), 1818-1829 (2008). TLC of the extracts of the TCM drug on silica gel with 1) chloroform - methanol - acetone - ammonia 2:1:4:4; 2) chloroform - methanol - acetone - formic acid 32:8:4:7; 3) ethyl acetate - butanone - formic acid - water 10:1:1:1; 4) chloroform - methanol - water 13:7:2. Detection by evaluation under UV 365 nm and after spraying with 1) potassium iodobismuthate reagent; 2) a 1:1 mixture of 1 % potassium ferricyanide and 1 % FeCl3; 3) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration.

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

104 184 T. QU (Qu Tingli), Y. DENG (Deng Yaning), L. HAU (Hau Lihong), ZH. ZHAO (Zhao Zhengbao)* (*Pharm. Coll., Shanxi Univ. Med., Taiyuan Shanxi 030001, China): (Identification of Shenlingbaizhu pills by thin-layer chromatography) (Chinese). J. Chinese Trad. Patent Med. 30 (12), Supl. 4-6 (2008). TLC of the TCM drug extracts on silica gel with 1) dichloromethane - ethyl acetate - methanol - water 15:40:22:10; 2) petroleum ether (60-90 °C) - diethyl ether 3:2; 3) petroleum ether (60-90 °C) - ethyl acetate 25:2; 4) *n*-butanol - acetic acid - water 4:1:2; 5) chloroform - diethyl ether 1:1. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed

by heating at 105 °C until coloration; 2) under UV 365 nm; 3) under UV 254 nm.

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

104 185 P. RAJA* , K. BHATT, V. JOSHI, K. AMIN (*A. R. College of Pharmacy and G. H. Patel Institute of Pharmacy, Anand, Gujarat, India): Development and validation of HPTLC method for simultaneous estimation of olmesartan medoxomil and hydrochlorothiazide in their combined tablet dosage form. 60th Indian Pharmaceutical Congress PA-232 (2008). HPTLC of olmesartan medoxomil and hydrochlorothiazide on silica gel with methanol - toluene - ethyl acetate 5:11:4 with chamber saturation for 30 min. The hR_F value of olmesartan medoxomil was 27 and of hydrochlorothiazide 44. The method was linear in the range of 300-1800 ng/spot for both drugs.

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

104 186 K.P. RANDAU, S. SPROLL, H. LERCHE, F. BRACHER* (*Department Pharmazie - Zentrum für Pharmaforschung, Ludwig-Maximilians-Universität, Butenandtstr. 5-13, 81377 München, Germany; Franz.Bracher@cup.uni-muenchen.de): Pernambucone, a new tropone derivative from Croton argyroglossum. Pharmazie 64, 350-351 (2009). Preparative TLC of pernambucone (3,8-dimethyl-5-isopropyl-2,3-dihydro-1H-azulene-1,6-dione), orobanone and extracts of stem bark on silica gel with hexane - ethyl acetate 4:1 and dichloromethane - methanol 39:1. Detection in visible light.

pharmaceutical research, herbal, preparative TLC 32e

104 187 P. RAO*, R. KUMAR, G. REDDY, R. BABOO (*A. M. Reddy Memorial College of Pharmacy, Guntur, A.P., India): Method development and validation of HPTLC method for estimation of quetiapine in bulk drugs and in tablet dosage form. Abstract No. F-261, 61st IPC (2009). HPTLC of quetiapine on silica gel with methanol - toluene 4:3. The hR_F value was 41. Quantitative determination by absorbance measurement at 235 nm. The method was linear in the range of 100-500 ng/band. Recovery was 98.9 %.

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

104 188 J. RAO*, S. YADAV, M. APNDE, S. GONDKAR (*Bharati Vidyapeeth University, Poona college of Pharmacy, Pune, Maharashtra, India): Stability indicating HPTLC method for tenofovir in the bulk drug and tablet dosage form. Abstract No. F-252, 61st IPC (2009). HPTLC of tenofovir on silica gel with *n*-butanol - acetic acid - water 4:1:1. The hR_F value was 58. Quantitative determination by absorbance measurement at 260 nm. Linearity was in the range of 120-600 ng/band. The compound was subjected to different stress conditions (acid, alkali, oxidation, photodegradation and thermal) and degradations products were well resolved from the main component.

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

104 189 K. REMYA*, A. BINDU, N. ALEYKUTTY, J. SAJAN (*Department of Pharmaceutical Science, Cheruvandoor Campus, Ettumanoor, Kottayam, Keral, India): High-performance thin-layer chromatographic method for quantitative determination of quercetin in tender leaves of Psidium guajava. 60th Indian Pharmaceutical Congress PG-264 (2008). HPTLC of quercetin in acetone extracts of Psidium guajava leaves on silica gel with toluene - acetone - formic acid 38:10:5. Quantitative determination of quercetin by absorbance measurement at 364 nm. The correlation coefficient was 0.9847. There was a good correlation between peak area and corresponding concentration of quercetin. The proposed HPTLC method provided a good resolution of quercetin from other constituents present in acetone extract of tender leaves of Psidium guajava and can be used for the quantification of quercetin.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 190 A. REN (Ren Ainu)*, Y. LI (Li Yun), M. JU (Ju Mingqiao) (*Jiangsu Provin. Acad. Med. & Pharm., Nanjing, 210028, China): (Study of the quality standard for Baozhi pills, a Chinese

traditional patent medicine) (Chinese). Chinese J. Pharm. Anal. 28 (1), 20-23 (2008). TLC of TCM drug extracts on silica gel with toluene - ethyl acetate - methanol - isopropanol - ammonia 12:6:3:3:1. Detection under UV light. The method is suitable for quality control of Baozhi pills.

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

104 191 A. RICHA*, S. KIRTI, L. RICHARD, S. ANNIE (*Dept. of Pharmacognosy, Manipal College of Pharmaceutical Science, Manipal, Karnataka 576104, India): Pharmacognostical, phytochemical and HPTLC fingerprinting evaluation of Dendrophthoe falcata leaf. Abstract No. 9712, IHCB (2009). HPTLC of extracts from leaves of Dendrophthoe falcata on silica gel with methanol - formic acid - water 40:3:57. Quantitative determination by absorbance measurement at 280 nm using quercetin as marker.

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

104 192 K.K. ROUT*, O.P. ROUT, S.K. MISHRA (*Pharmacognosy and Phytochemistry Division, University Department of Pharmaceutical Sciences, Utkal University, Vani Vihar, Bhubaneswar 751004, Orissa, India; kd_rout@yahoo.co.in): Standardization of Ayurvedic formulations containing Aloe vera by quantification of a marker compound. J. Planar Chromatogr. 22, 381-384 (2009). TLC of aloin in commercial Ayurvedic preparations on silica gel (prewashed with methanol) with ethyl acetate - methanol - water 50:7:2 in a twin trough chamber with chamber saturation for 5-7 min at 30 +/- 4 °C and a relative humidity of 57 +/- 3 %. Quantitative determination by absorbance measurement at 360 nm. The limit of detection and quantification was 10 and 20 ng/band, respectively.

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

104 193 P. SAINI*, C. JAIN, R. SINGH, S. MATHUR, G. SINGH, M. NASLAM (*Research & Development Div., Indian Pharmacopoeia Commission, Govt. of India, Ministry of Health & Family Welfare, Sector-23, Rajnagar, Ghaziabad 201002, India, ipclab@vsnl.net): A simple and sensitive HPTLC method for simultaneous determination of abacavir sulphate and lamivudine in tablet dosage form. J. Pharma. Research 8(4), 187-191 (2009). HPTLC of abacavir sulphate and lamivudine on silica gel with methanol - acetone - *n*-butyl acetate 1:1:2. Quantitative determination by absorbance measurement at 284 nm. The hR_F value of abacavir sulphate was 58 and of lamivudine 35. Linearity of abacavir sulphate and lamivudine was in the range of 240-1200 ng/spot and 120-600 ng/spot, respectively. The limit of detection and quantification of abacavir sulphate was 0.7 and 2 ng/spot, respectively, and of lamivudine 1 and 3 ng/spot, respectively.

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

104 194 A. SARASWATHY*, D. RAMASAMY, S. ARIMASAMY, D. NANDINI (*CSM Drug Research Inst. for Ayurveda and Siddha, Anna Hospital Campus, Arumbakkam, Chennai 600106, saraswathy20042000@yahoo.co.in): Development of HPTLC profile and heavy metal analysis of stem bark of three Ficus species. Indian Drugs 46(6), 493-496 (2009). HPTLC of chloroform extracts of the bark of Ficus racemosa, F. bengalensis, and F. religiosa on silica gel with toluene - ethyl acetate - formic acid 90:10:1. Detection under UV 254 nm and visible light after treatment with vanillin sulfuric acid reagent, followed by heating at 105 °C until coloration.

herbal, HPTLC, qualitative identification 32e

104 195 N. SARATHI*, M. GANDHIMATHI, R. SAKTHI, T. RAVI (*Sri Ramakrishna Institute of Paramedical Science, Coimbatore, Tamil Nadu, India): A rapid HPTLC analysis of oxcarbazepine in human plasma. 60th Indian Pharmaceutical Congress PA-227 (2008). HPTLC of oxcarbazepine (in acetonitrile extracts of human plasma) on silica gel with ethyl acetate - toluene - methanol 7:2:1. The hR_F values of oxcarbazepine and the internal standard chlorzoxazone were 54 and 86, respectively. The linearity range was 10-300 ng/mL, recovery from plasma was 75.2 %.

quality control, HPTLC, densitometry, quantitative analysis 32a

104 196 S. SATHE*, S. BARI (*Department of Pharmaceutical Chemistry, R. C. Patel College of Pharmacy, Karwand Naka, Shirpur Dhule, Maharashtra 425405, India, sbbari@rediffmail.com, shitalsathe@rediffmail.com): Quantitative analysis of losartan potassium and atenolol by high-performance thin-layer chromatography. Indian Drugs 46(1), 78-81 (2009). HPTLC of atenolol and losartan potassium in tablets on silica gel with toluene - methanol - triethylamine 12:8:1 with chamber saturation for 45 min. Quantitative determination by absorbance measurement at 230 nm. The hR_F value of atenolol was 45 and of losartan potassium 67. The method was linear in the range of 1000-4000 ng/spot for both compounds. The recovery was 98.8-98.9 %.

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

104 197 Sunita SEERAPU*, B. SRINIVASAN (*Delhi Institute of Pharmaceutical Sciences and Research (DIPSAR), New Delhi, India): Development and validation of analytical method on HPTLC for the determination of ivabradine hydrochloride as bulk drug and in pharmaceutical formulations. Abstract No. F-275, 61st IPC (2009). HPTLC of ivabradine HCl on silica gel with methanol - chloroform 1:1. The hR_F value was 59. Quantitative determination by absorbance measurement at 285 nm. Linearity was in the range of 100-800 ng/spot with r^2=0.9989 (via peak area).

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

104 198 M. SHAH*, A. PRAJAPATI, S. PATEL, N. PATEL (*Shree S. K. Patel College of Pharmaceutical Education and Research, Ganpat University, Ganpat Vidyanagar, Mehsana, Gujarat, India): Estimation of voriconazole in powder by HPTLC method. 60th Indian Pharmaceutical Congress PA-231 (2008). HPTLC of voriconazole on silica gel with toluene - ethyl acetate 1:3. Quantitative determination by absorbance measurement at 255 nm. The linearity range was 10-1200 ng/spot, recovery was 99.5 %. The method was suitable for routine quality control of formulations.

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

104 199 N. SHAH*, B. SUHAGIA, R. SHAH, N. PATEL (*Shri B. M. Shah College of Pharmaceutical Education & Research, Modasa 383315): HPTLC method for the simultaneous estimation of valsartan and hydrochlorothiazide in tablet dosage form. Ind. J. Pharma. Sci. 71(1), 72-74 (2009). HPTLC of valsartan and hydrochlorothiazide on silica gel with chloroform - methanol - toluene - acetic acid 60:20:10:1. Quantitative determination by absorbance measurement at 260 nm. The calibration curve was linear between 300 to 800 ng/spot for valsartan and 100 to 600 ng/spot for hydrochlorothiazide. The limit of detection and the limit of quantification for valsartan were 100 and 300 ng/spot, respectively, and for hydrochlorothiazide 30 and 100 ng/spot, respectively.

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

104 200 R. SHAH*, H. BHATT, G. PATEL, C. SHARASTRY (*Shree Dhanvantary Pharmacy College, Surat, Maharashtra, India): Simultaneous estimation of mosapride citrate and pantoprazole in solid dosage form by HPTLC method. Abstract No. F-236, 61st IPC (2009). HPTLC of pantoprazole and mosapride citrate on silica gel (pre-washed with methanol) with ethyl acetate - benzene - methanol - 25 % ammonia 48:35:15:2 at room temperature. Quantitative determination by absorbance measurement at 250 nm or 276 nm. The method was linear in the range of 3-7 µg/band for both drugs. Recovery was between 99.0 and 103.5 %.

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

104 201 R. SHAH*, H. PANCHAL, B. SUHAGLA, N. PATEL (*S. K. Patel College of Pharma. Education & Research, Mehsana, Gujarat, India): Simultaneous determination of atorvastatin calcium, ramipril and aspirin in capsule dosage form by HPTLC. 60th Indian Phamraceutical Congress PA-209 (2008). HPTLC of atorvastatin calcium, ramipril and aspirin on silica gel with methanol - benzene - ethyl acetate - glacial acetic acid 9:140:100:1. Quantitative determination by absor-

bance measurement at 210 nm. The hR_F value of ramipril was 6, of atorvastatin 38, and of aspirin 86. Linearity was 100-600 ng/band (atorvastatin), 50-300 ng/band (ramipril) and 500-3000 ng/band (aspirin). Recovery was 99.9-100.0 % for all three compounds. Salicylic acid, an impurity of aspirin, was observed in capsule dosage form with an hR_F value of 72. The method was suitable for analysis of combined dosage form.

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

104 202 K. SHAH*, I. SOJITRA, R. SHAH, U. VACHHANI (*Rofel Shri G. M. Bilakhia College of Pharmacy, Vpi, Gujarat, India): Development and validation of HPTLC analytical method for determination of L-dopa in Mucuna pruriens powdered extract and polyherbal formulations. Abstract No. F-262, 61st IPC (2009). HPTLC of L-dopa on silica gel with *n*-butanol - water - actic acid 4:1:1. The hR_F was 37. Detection by spraying with 0.5 % ethanolic ninhydrin solution, followed by heating at 120 °C for 2 min. Quantitative determination by absorbance measurement at 520 nm. The method was linear in the range of 600-1400 ng/band.

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

104 203 N. SHARMA, U. SHARMA, A. GUPTA, DEVLA, A. SINHA*, B. LAL, P. AHUJA (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology (CSIR), Palampur, Himachal Pradesh, India, aksinha08@rediffmail.com) : Simultaneous densitometric determination of shikonin, acetylshikonin, and beta-acetoxyisovaleryl-shikonin in ultrasonic-assisted extracts of four Arnebia species using reversed-phase thin layer chromatography. J. Sep. Sci. 32, 3239-3245 (2009). HPTLC of shikonin (1), acetylshikonin (2), and beta-acetoxyisovalerylshikonin (3) in four species of Arnebia on RP-18 with acetonitrile - methanol - 5 % formic acid 20:1:4. Quantitative determination by absorbance measurement at 520 nm. Linearity was in the range of 100-600 ng/zone for (1) and (2) and 100-1800 ng/zone for (3). The limits of detection for (1), (2) and (3) were 18, 15 and 12 ng/zone, respectively, while the limits of quantification were 60, 45 and 40 ng/zone, respectively.

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

104 204 A.A. SHIRKHEDKAR*, P.M. BUGDANE, S. SURANA (*R.C. Patel College of Pharmacy, Shirpur Dist: Dhule, (M.S.) 425 405 India): Stability-indicating TLC-densitometric determination of nebivolol hydrochloride in bulk and pharmaceutical dosage form. J. Chromatogr. Sci. 48 (2), 109-113 (2010). HPTLC of nebivolol hydrochloride on silica gel with toluene - methanol - triethylamine 19:6:1. The hR_F value of nebivolol hydrochloride was 33. Quantification by densitometry in the absorbance mode at 281 nm. Linearity was between 500 and 3000 ng/spot with r^2= 0.9994. The limit of detection and quantification was 63 and 191 ng/spot, respectively. Nebivolol hydrochloride was subjected to acid and alkali hydrolysis, oxidation, thermal degradation, and photodegradation. The degradation products were well-resolved from the main component.

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

104 205 A.A. SHIRKHEDKAR*, S. J. SURANA (*Department of Pharmaceutical Chemistry, R. C. Patel Institute of Pharmaceutical Education and Research, Shirpur Dist: Dhule (M.S.) India 425 405; atulshirkhedkar@rediffmail.com; sjsurana@yahoo.com): Simultaneous densitometric TLC analyis of atorvastatin calcium and fenofibrate in the bulk drug and in pharmaceutical formulations. J. Planar Chromatogr. 22, 355-358 (2009). TLC of atorvastatin calcium and fenofibrate on silica gel, prewashed with methanol, with toluene - methanol - triethylamine 35:15:1 in a twin trough chamber saturated for 25 min at room temperature and relative humidity of 60 +/- 5 %. Quantitative determination by absorbance measurement at 258 nm. The limit of detection and quantification for atorvastatin calcium was 25 and 77 ng/zone, respectively and for fenofibrate 292 and 886 ng/zone, respectively.

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

104 206 A.R. SHRIVASTAVA, C.R. BARHATE, C.J. KAPADIA* (*Department of Pharmaceutics, Bombay College of Pharmacy, Kalina, Santacruz, Mumbai 400 098, India; drcjkapadia@gmail.com): Stress degradation studies in valsartam using validated stability-indicating high-performance thin-layer chromatography. J. Planar Chromatogr. 22, 411-416 (2009). HPTLC of valsartan in bulk drug and in formulations on silica gel (prewashed with methanol) with toluene - ethyl acetate - methanol - formic acid 60:20:20:1 in a twin trough chamber saturated for 20 min. Quantitative determination by absorbance measurement at 250 nm. The limit of detection and quantification was 25 and 150 ng/band, respectively.

pharmaceutical research, quality control, HPTLC 32a

104 207 N. SINGH, S. KHATOON*, N. SRIVASTAVA, A.K. SINGH RAWAT, S. MEHROTA (*Pharmacognosy and Ethnopharmacology Division, National Botanical Research Institute, Rana Pratp Marg, Lucknow 226001, India; neha_somvanshi@yahoo.com, sayyadak@yahoo.com): Qualitative and quantitative standardization of Myrica esculenta Buch.-Ham stem bark by use of HPTLC. J. Planar Chromatogr. 22, 287-291 (2009). HPTLC of the biomarkers gallic acid, lupeol, oleanolic acid, and stigmasterol and plant extracts on silica gel with toluene - ethyl acetate - formic acid 5:5:1 for gallic acid and with toluene - ethyl acetate 4:1 for lupeol, oleanolic acid, and stigmasterol in a saturated twin trough chamber. Quantitative determination by absorbance measurement at 272 nm. Detection of oleanolic acid, lupeol, and stigmasterol by dipping in anisaldehyde reagent followed by heating at 110 °C for 5 min. Densitometric evaluation at 652 nm.

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

104 209 B. SINGH*, S. ANANDJIWALA, M. NIVSARKAR (*National Institute of Pharmaceutical Education and Research, Ahmedabad, Gujarat, India): TLC densitometric analysis of glycyrrhizin, glycyrrhetinic acid, apigenin, kaempferol and quercetin from Glycyrrhiza glabra using HPTLC. 60th Indian Pharmaceutical Congress PG-323 (2008). HPTLC of glycyrrhizin, apigenin and kaempferol in methanolic extracts and glycyrrhetinic acid and quercetin in hydrolized extracts of Glycyrrhiza glabra on silica gel with ethyl acetate - methanol - acetic acid - water 40:5:5:10 (for glycyrrhizin), toluene - ethyl acetate - methanol - formic acid 30:15:1:2 (for kaempferol and quercetin), ethyl actate - ethanol - water - ammonia 65:20:4:1 (for glycyrrhetinic acid). Quantitative determination by absorbance measurement at 254 nm (glycyrrhetinic acid and apigenin), 258 nm (glycyrrhizin) and 280 nm (kaempferol and quercetin). The plant was found to contain 1.07 % glycyrrhizin, 0.64 % glycyrrhetinic acid, 0.007 % apigenin, 0.03 % kaempferol and 0.24 % quercetin.

HPTLC, densitometry, quantitative analysis 32e

104 208 K. SINGH*, S. AGRAWAL, M. GUPTA (*Delhi Institute of Pharmaceutical Sciences and Research, New Delhi, India): Development and validation of improved HPTLC method for simultaneous determination of curcumin, demethoxycurcumin and bis-demethoxycurcumin. 60th Indian Pharmaceutical Congress PA-223 (2008). HPTLC of curcumin, demethoxycurcumin and bis-demethoxycurcumin on silica gel with chloroform - methanol 19:1. The hR_F values were 25, 38, and 61 for bis-demethoxycurcumin, demethoxycurcumin, and curcumin respectively. Quantitative determination by absorbance measurement at 420 nm. The method was linear in the range of 50-400 ng/spot (curcumin), 10-150 ng/spot (demethoxycurcumin), and 5-40 ng/spot (bis-demethoxycurcumin). Recovery was in the range of 99.2-100.5 % for all three compounds.

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

104 210 P. SINHA*, M. DAMLE, K. BOTHARA (*AISSMS College of Pharmacy, Pune, Maharashtra, India): A validated stability indicating method for determination of aspirin and clopidogrel bisulphate. 60th Indian Pharmaceutical Congress PA-208 (2008). TLC of aspirin and clopidogrel bisulphate on silica gel with carbon tetrachloride - acetone 5:2. The hR_F value of aspirin was 15 and of clopidogrel bisulphate 80. Quantitative determination by absorbance measurement at 220 nm. The method was linear in the range of 2-6 µg/spot for aspirin and 3-6 µg/spot for clopidogrel.

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

104 211 Krystyna SKALICKA-WOZNIAK*, M. L. HAJNOS, K. GLOWNIAK (* Department of Pharmacognosy with Medicinal Plant Laboratory, Medical University of Lublin, Chodzki 1, 20-093 Lublin, Poland; kskalicka@pharmacognosy.org): High-performance thin-layer chromatography combined with densitometry for quantitative analysis of chlorogenic acid in fruits of Peucedanum alsaticum L. J. Planar Chromatogr. 22, 297-300 (2009). HPTLC of chlorogenic acid and of plant extracts on silica gel with ethyl acetate - formic acid - water 10:2:3 and ethyl acetate - formic acid - acetic acid - water 100:11:11:21 in a saturated horizontal chamber. Quantitative determination by absorbance measurement at 320 nm. Qualitative detection by derivatization with natural products reagent (1 % in methanol) followed by treatment with 5 % PEG 400 in ethanol.

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

104 212 Danuta SOBOLEWSKA*, Z. JANECZKO, I. PODOLAK, A. SZERLOMSKA (*Department of Pharmacognosy, Jagiellonian University, Collegium Medicum, Medyczna 9, 30-688 Cracow, Poland; dsobolew@cm-uj.krakow.pl): Densitometric analysis of diosgenin in methanolic extracts of Allium ursinum collected at different times during plant development. J. Planar Chromatogr. 22, 305-307 (2009). TLC of diosgenin in methanolic extracts of fresh leaves and bulbs on silica gel with *n*-hexane - acetone 4:1. Detection by spraying with 25 % sulfuric acid in methanol and heating at 100 °C for 2 min. Quantitative determination by absorbance measurement at 540 nm.

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

104 213 P. SONAWANE*, M. DHOKA, V. GAWANDE, P. VAIDYA (*All India Shri Shivaji Memorial Society‘s College of Pharmacy, Pune, Maharashtra, India): Simultaneous estimation of cefixime trihydrate and erdosteine in pharmaceutical dosage form by HPTLC method. Abstract No. F-256, 61st IPC (2009). HPTLC of cefixime trihydrate and erdosteine in combined capsule dosage form on silica gel with ethyl acetate - acetone - methanol - water 15:5:5:3. Quantitative determination by absorbance measurement at 235 nm. The calibration curve was linear between 100 and 500 ng/band for cefixime and 150 to 750 ng/band for erdosteine. The limit of detection and quantification for cefixime was 0.37 µg/mL and 1.14 µg/mL, respectively and for erdosteine 0.33 µg/mL and 1.04 µg/mL, respectively.

quality control, HPTLC, densitometry, quantitative analysis 32a

104 214 N. SONI*, S. MANIMARAN, N. MURUGANANTHAM, S. DHANABAL, K. ELANGO (*Dept. of Phytopharmacy & Phytomedicine, JSS College of Pharmacy, Ootacamund,The Nilgiri, Tamil Nadu, India): Validated HPTLC method for the analysis of colchicine. Abstract No. 9933, IHCB (2009). HPTLC of colchicines in Gloriosa superba (collected from different parts of India) on silica gel with ethyl acetate - methanol 200:27. The hR_F value of colchicine was 29. Quantitative determination by absorbance measurement at 350 nm. The method was linear in the range of 50-1000 ng/spot. The sample collected from Kerala was found to contain highest level of colchicines (0.24 %).

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

104 215 B. SPARZAK, Miroslawa KRAUZE-BARANOWSKA*, L. POBLOCKA-OLECH (*Medical University of Gdansk, Department of Pharmacognosy with the Medicinal Plants Garden, Hallera 107, 80-416 Gdansk, Poland; krauze@amg.gda.pl): High-performance thin-layer chromatography densitometric determination of beta-sitosterol in Phyllantus species. J. AOAC Int. 92, 1343-1348 (2009). HPTLC of beta-sitosterol, beta-amyrin and plant extracts on silica gel with chloroform - *n*-hexane - methanol 13:6:1. Detection by spraying with vanillin-orthophosphoric acid reagent, 5 % phosphomolybdic acid, or anisaldehyde reagent, followed by heating at 110 °C for 5 min. Vanillin reagent provided the best results. Quantitative determination by absorbance measurement at 525 nm.

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

104 216 Malgorzata STAREK*, M. REJDYCH (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Kraków, Poland; mstarek@interia.pl): Densitometric analysis of celecoxib, etoricoxib and valdecoxib in pharmaceutical preparations. J. Planar Chromatogr. 22, 399-403 (2009). TLC of celecoxib, etoricoxib, and valdecoxib on silica gel with chloroform - acetone - toluene 12:5:2 with chamber saturation for 15 min at room temperature. Quantitative determination by absorbance measurement at 254 and 290 nm.

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

104 217 G.S. SUBRAMANIAN*, A. KARTHIK, A. BALIGA, P. MUSMADE, S. KINI (*Department of Pharmaceutical Quality Assurance, Manipal College of Pharmaceutical Sciences, Manipal University, Manipal, Karnataka, India 576104; ganrajesh@gmail.com): High-performance thin-layer chromatographic analysis of bicalutamide in bulk drug and liposomes. J. Planar Chromatogr. 22, 273-276 (2009). HPTLC of bicalutamide and leflunomide (as internal standard) on silica gel with toluene - ethyl acetate 4:5 in a twin trough chamber saturated for 30 min at room temperature. Quantitative determination by absorbance measurement at 273 nm. The limit of detection and quantification was 50 and 200 ng/band, respectively.

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

104 218 P. SUBRAMANIAN*, A. RAJENDRAN, V. MADHURAM, R. PADMA (*Drug Standardisation Unit O.U.B. 32, Raod, No.4, Habsiguda, Hyderabad 500007, India, veasha@rediffmail.com): HPTLC fingerprinting of some ethnomedicinally important Cassia species. Indian Drugs 46(6), 477-482 (2009). HPTLC of extracts of Cassia auriculata, C. obtusifolia, and C. uniflora and of chrysophanol and emodin on silica gel with toluene - ethyl acetate - formic acid 10:3:1. Detection under UV 254 nm. Based on the fingerprint and by comparison with chemical markers identification of each species was possible.

herbal, densitometry, HPTLC, qualitative identification 32e

104 219 A. SUGANTHI*, A. FATHIMUNNISA, T. RAVI (*College of Pharmacy, SRIPMS, Coimbatore, Tamil Nadu, India): HPTLC method for the simultaneous estimation of itopride hydrochloride and pantoprazole in pharmaceutical dosage form. Abstract No. F-264, 61st IPC (2009). HPTLC of pantoprazole and itopride hydrochloride on silica gel with *n*-butanol - chloroform - 25 % ammonia 7:2:1. The hR_F value was 54 and 75 for pantoprazole and itopride hydrochloride, respectively. Quantitative determination by absorbance measurement at 291 nm. The linearity of the method was 80-240 ng/band for pantoprazole and 300-900 ng/band for itopride.

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

104 220 A. SUGANTHI*, M. SRIKANTH, A. GOP, T. RAVI (*College of Pharmacy, Sri Ramakrishna Institute of Paramedical Sciences, Coimbatore, Tamilnadu, India): A validated HPTLC method for the estimation of fenoverine in capsule dosage form. Abstract No. F-270, 61st IPC (2009). HPTLC of fenoverine on silica gel with methanol - butyl acetate 1:4. The hR_F value of fenoverine was 67. The linearity was in the range of 50 to 500 ng/band with a correlation coefficient of 0.9976. The limit of detection and quantification was 30 ng/band and 100 ng/band, respectively. The recovery was 100.1 %.

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

104 221 B. SUHAGIA*, T. RATHOD, S. SINDHU (*L. M. College of Pharmacy, Ahmedabad,Gujarat, India): Quantitative determination of Sapindus saponins in the pericarp of Sapindus mukorossi. Abstract No. F-287, 61st IPC (2009). HPTLC of saponins (a mixture of the seven different sapindosides A, B, C, D, E, F, and G) in the pericarp of Sapindus mukorossi on silica gel with chloroform - methanol - water 50:14:1. Detection by derivatization with sulfuric acid reagent and

evaluation under visible light. The hR_F values of sapindosides A to G are 19, 26, 41, 48, 59, 67, and 75. Quantitative determination by absorbance measurement at 550 nm after derivatization. The linearity ranges for sapinosides A to G were 0.9-4.3, 0.7-5.4, 3.3-15.2,1.2-9.7, 2.7-12.5, 0.2-1.3, 0.3-1.5 µg/band. The pericarp contained 15.2 % w/w of total sapindosides.

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

104 222 Divya SUKUMAR*, R. ARIMBOOR, C. ARUMUGHAN (*Agroprocessing & Natural Products Div., National Institute for interdisciplinary Science & Technology, Thiruvananthapuram, Kerala 695010, India, carumughan@yahoo.com): HPTLC fingerprinting and quantification of lignans as markers in sesame oil and its polyherbal formulations. J. Pharm. Biomed. Anal. 47, 795-801 (2008). HPTLC of sesamin and sesamoline (the major lignans in sesamum oil and its herbal formulations) on silica gel with benzene - methanol 50:1 with chamber saturation. Quantitative determination by absorbance measurement at 290 nm. The identity of sesamin and sesamoline was confirmed by UV-VIS spectra, NMR and MS of the compounds obtained by scraping off from the plate and elution. For fingerprint analysis derivatization with 5 % methanolic sulphuric acid was performed, followed by heating at 100 °C for 20 min and densitometry at 450 nm.

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

104 223 Katarzyna SZEWCZYK*, L. KOMSTA, A. SKALSKA.KAMINSKA (*Department of Pharmaceutical Botany, Faculty of Pharmacy, Medical University of Lublin, Chodzki 1, 20-093 Lublin, Poland; k.szewczyk@am.lublin.pl): Densitometric HPTLC method for analysis of triterpenoids in the leaves of Jovibarba sobolifera (Sims.) Opiz (Hen nad chickens houseleek). J. Planar Chromatogr. 22, 367-369 (2009). HPTLC of triterpenoids (alpha- and beta-amyrin, oleanolic acid) on silica gel prewashed with methanol and dichloromethane, with dichloromethane - ethyl acetate 37:3. in a horizontal chamber saturated for 15 min. Detection by spraying with 8 % sulfuric acid in ethanol and heating at 105 °C for 3 min. Evaluation in daylight and under UV 366 nm. Quantitative determination by absorbance measurement at 520 nm.

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

104 224 E. TOTH*, G. JANICSAK, I. MATHE, G. BLUNDEN (*Institute of Ecology and Botany of the Hungarian Academy of Sciences, Alkotmay u. 2, 2163 Vacratot, Hungary; totheniko@botanika.hu): Determination of phenylpropanoids in three Ballota species. J. Planar Chromatogr. 22, 293-296 (2009). TLC of verbascoside, forsythoside B, caffeoyl-malic acid and plant (Ballota nigra, B. hirsuta, and B. rupestris) extracts on silica gel with formic acid - acetic acid - water - ethyl acetate 15:15:36:134. Quantitative determination by fluorescence measurement at 395 nm. It was observed that amounts of phenylpropanoids in Ballota nigra leaves increase during the main and secondary flowering periods in June.

herbal, quality control, densitometry, quantitative analysis 32e

104 225 J. VADHAVANA*, B. PATEL, R. PATEL (*K. B. Institute of Pharmaceutical Education and Research, Gandhinagar, Gujarat, India): Simultaneous estimation of nebivolol hydrochloride and S-amlodipine besylate by high-performance thin-layer chromatography. Abstract No. F-247, 62st IPC (2009). HPTLC of nebivolol HCl and S-amlodipine besylate on silica gel (prewashed with methanol) with chloroform - toluene - methanol - acetic acid 50:20:1. The hR_F value was 33 and 48 for S-amlodipine and nebivolol, respectively. Quantitative determination by absorbance measurement at 271 nm. The method was linear in the range of 500-2500 ng/band for nebivolol and 250-1280 ng/band for S-amlodipine, respectively.

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

104 226 D. VASA*, N. VASA, P. GIDE, V. VAGHELA (*A. R. College of Pharmacy & G. H. Patel Institute of Pharmacy, Anand, Gujarat, India): HPTLC method development for estimation of rivastigmine hydrogen tartrate in pharmaceutical dosage form. Abstract No. F-365, 61st IPC (2009).

HPTLC of rivastigmine hydrogen tartrate on silica gel with methanol - 25 % ammonia - acetic acid 200:7:2. The hR_F value was 54. Quantitative determination by absorbance measurement at 215 nm. The method was linear in the range of 1-10 µg/band.

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

104 227 S. VASANTHARAJU*, A. KARTHIK, K. BHAT, C. PRASHANT, M. RAO, N. UDUPA (*Manipal College of Pharmaceutical Science, Manipal, Karnataka, India): HPTLC method development and validation of capsaicin in bulk drug. Abstract No. 9452, IHCB (2009). HPTLC of capsaicin on silica gel with toluene - ethyl acetate 3:2. The hR_F value of capsaicin was 38. Quantitative determination by absorbance measurement at 280 nm. The method was linear in the range of 100-1000 ng/spot. Capsaicin was subjected to different stress conditions (alkali, acid, oxidation, thermal). The method is suitable for separation of capsaicin from its degradation products and can be used to indicate stability.

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

104 228 R. VERMA*, H. MUKHTAR, R. SINGH, A. PASRIJA (*S.B.S College of Pharmacy, Patty, Punjab, India): Validated HPTLC method for the determination of 3H-4M-benzaldehyde in crude plant material, extracts and dosage form of Hemidesmus indicus. 60th Indian Pharmaceutical Congress PG-349 (2008). HPTLC of 3H-4M-benzaldehyde (in crude plant material, extracts and dosage form of Hemidesmus indicus) on silica gel with toluene - ethyl acetate - methanol - acetic acid 15:3:1:1. Quantitative determination by absorbance measurement at 230 nm.

herbal, HPTLC, densitometry, quantitative analysis 32e

104 229 S. WAKODE*, H. SINGH, V. SINGH (*Delhi Institute of Pharmaceutical Science & Research, New Delhi, India): Development and validation of HPTLC assay method for voriconazole in tablets. 60th Indian Pharmaceutical Congress PA-229 (2008). HPTLC of voriconazole on silica gel with toluene - methanol - glacial acetic acid 78:20:1. Quantitative determination by absorbance measurement at 254 nm. The method was linear in the range of 50-400 ng/spot, recovery was 99.9-100.7 %. The method was suitable for routine quality control of the dosage form.

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

104 230 P. WAVHAL*, J. SANGSHETTI, A. SARKATE, P. WAKTE, D. SHINDE (*Dept. of Chemical Technology, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, Maharashtra, India): Stability-indicating HPTLC determination of tadalafil in API and in its pharmaceutical dosage form. Abstract No. F-257, 61st IPC (2009). HPTLC of tadalafil on silica gel with *n*-hexane - ethyl acetate - acetonitrile 14:3:3. The hR_F value was 65. Quantitative determination by absorbance measurement at 215 nm. The method was linear in the range of 10-60 ng/band. The drug was subjected to different stress conditions (acid, alkali, oxidative, photodegradation, thermal) and showed degradation under all stress conditions. Degradation products and excipients of the formulation were well separated from the main component.

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

104 231 T. XONG (Xong Ting) (Pharm. Coll., Wuhan Univ., Wuhan, Hubei 430072, China): (Determination of astragaloside in Tianjian capsule) (Chinese). Chinese J. Hospit. Pharm. 29 (4), 336-338 (2009). TLC of astragaloside in Tianjian capsules on silica gel with chloroform - methanol - water 13:6:2. Detection by spraying with 5 % sulfuric acid in ethanol followed by heating at 105 ºC until coloration. Quantification by densitometry at 515 nm. The linearity was between 0.98 and 4.90 µg/spot ($r^2 = 0.998$), the *%RSD* was 2.4 % (n = 6) within plate and 2.3 % (n = 6) inter-plate, standard addition recovery was 99.2 % with RSD = 1.7 % (n = 6).

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

104 232 L. XU (Xu Li)*, Y. ZHAO (Zhao Yuan), Y. LUAN (Luan Yuquan), Y. YANG (Yang Yongshou), CH. WANG (Wang Chengjun) (*Basic Med. Coll., Dali Acad., Dali, Yunnan 671000, China): (Study of the content of puerarin in different parts of Radix Puerariae) (Chinese). Learned J. Dali Acad. (General Issue) 8 (10), 3-6 (2009). TLC of puerarin (in extracts obtained from different parts of Radix Puerariae) on silica gel with trichloromethane - methanol - water 14:5:1. Detection under UV 254 nm. The maximum amount of puerarin was found in the bine of the drug, followed by that in the root, whereas no puerarin was found in the flower and fruit.

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

104 233 A. YADAV, R.M. SINGH*, S.C. MAHTUR, P.K. SAINI, G.N. SINGH (*Indian Pharmacopoeia Commission, Govt. of India, Ministry of Health & Family Welfare, Sect-23, Rajnagar, Ghaziabad (U.P), India 201 002; raman19662002@yahoo.co.in; ipclab@vsnl.net): A simple and sensitive HPTLC method for simultaneous analysis of domperidone and paracetamol in tablet dosage form. J. Planar Chromatogr. 22, 421-424 (2009). TLC of domperidone and paracetamol on silica gel with acetone - toluene - methanol 2:2:1 in a twin trough chamber saturated for 30 min at room temperature. Quantitative determination by absorbance measurement at 285 nm for domperidone and at 248 nm for paracetamol.

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

104 234 A. YADAV*, N. TIWARI, P. SRIVASTAVA, S. SINGH, K. SHANKER, R. VERMA, M. GUPTA (*Analytical Chemistry Div. Central Institute of Medicinal and Aromatic Plants, Lucknow 226015, India, guptammg@rediffmail.com): Iridoid glycoside-based quantitative chromatographic fingerprint analysis: A rational approach for quality assessment of indian medicinal plant Gambhari (Gmelina arborea). J. Pharm. Biomed. Anal. 47, 841-846 (2008). HPTLC of iridoid glycosides in the aerial part of Gambhari (Gmelina arborea) with iridoid gycoside 6-0-(2“, 3“- dibenzoyl)-o-L-rhamnopyranosylcatalpol as a chemical marker for the standardization of G. arborea plant extracts on silica gel with chloroform - methanol 4:1. Quantitative determination by absorbance measurement at 240 nm and at 430 nm after derivatization with vanillin - sulfuric acid reagent. The linear working range was between 1000-5000 ng/spot with a good correlation coefficient of 0.994.

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

104 235 H. YAN (Yan Hua)*, Y. ZHANG (Zhang Yumei), J. SONG (Song Jincui), J. LU (Lu Jing) (*Nat. Inst. Cont. Pharm. & Biolog. Prod., Beijing 100050, China): (Analysis of adenosine and adenine in Lingzhi capsules by TLC and HPLC) (Chinese). Chinese J. Pharm. Anal. 28 (11), 1800-1803 (2008). TLC of adenosine and adenine in Lingzhi capsules on silica gel with chloroform - ethyl acetate - i-propanol - water 16:6:3:1. Detection under UV 254 nm. The method is simple, fast and accurate and can be used for the quality control of the medicine.

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

104 236 L. YANG (Yang Li)*, SH. ZHANG (Zhang Shengwan), W. DU (Du Wen), W WANG (Wang Wei), M. LI (Li Meiping) (*Coll. Life Sci. & Technol., Shanxi Univ., Taiyuan 030036, China): (Determination of hyperoside in the raw extract of Hypericum perforatum by thin-layer chromatography) (Chinese). Chinese J. Pharm. Anal. 28 (4), 608-610 (2008). TLC of hyperoside in the raw extract of Hypericum perforatum on silica gel with petroleum ether (60-90 °C) - ethyl acetate - methanol 1:4:2. Detection under daylight. Quantification by densitometry at 591 nm. Linearity was given between 0.1 and 14.4 µg/spot (r^2 = 0.9892), recovery was between 96.4 and 100.1 %.

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

104 237 H.E. ZAAZAA*, S.S. ABBAS, M. ABDELKAWY, M.M. ABDELRAHMAN (*Analytical Chemistry Department, Faculty of Pharmacy, Cairo University, Kasr El-Aini St., 11562 Cairo, Egypt): Spectrophotometric and spectrodensitometric determination of clopidogrel bisulfate with kinetic study of its alkaline degradation. Talanta 78 (3), 874-884 (2009). Presentation of a sensitive, selective and precise stability-indicating method for the determination of clopidogrel bisulfate in presence of its alkaline degradate and in pharmaceutical formulations. TLC on silica gel with hexane - methanol - ethyl acetate 87:10:3. Quantification by densitometry at 248 nm in the range of 0.6-3 µg/band. Recovery was 99.9 %. Clopidogrel could be determined in the presence of up to 90 % of its alkaline degradate. Method selectivity was evaluated using laboratory prepared mixtures. The analysis of clopidogrel in pharmaceutical dosage forms is possible without interference from additives.

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

104 238 CH. ZHENG (Zheng Cheng)*, T. YAO (Yao Tongwei), ZH. BAI (Bai Zhimin) *(Pharm. Coll., Zhejiang Univ., Hangzhou 310080, China): (Study of the quality standard for Jinlu pills) (Chinese). J. Chinese Trad. & Herb. Drugs 40 (6), 900-903 (2009). TLC of the TCM drug extracts on silica gel with 1) ethyl acetate - formic acid - glacial acetic acid - water 15:1:1:2; 2) petroleum ether (30-60 °C) - ethyl acetate - formic acid 15:5:1; 3) chloroform - ethyl acetate - methanol - water 31:81:25:2. Detection 1) by spraying with 10 % sulfuric acid in ethanol followed by heating at 105 °C until coloration; 2) under UV 254 nm.

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

104 239 Y. ZHOU (Zhou Ying)*, X. NIU (Niu Xiuhua) (*Nat.Inst. Cont. Pharm. & Biolog. Prod., Beijing 100050, China): (Determination of related impurity substances in nisoldipine by thin-layer chromatography) (Chinese). Drug Standards of China 9 (2), 144-146 (2008). TLC of nisoldipine silica gel with chloroform - acetone - triethylamine - water 90:5:1. Detection under UV 254 nm. Semiquantification of impurities by comparison of spots. The method was successfully used for the quality control of real life samples.

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

33. Inorganic substances

104 240 A. RADOICIC, H. MAJSTOROVIC, T. SABO, Z. TESIC, Dusanka MILOJKOVIC-OPSENICA* (*Faculty of Chemistry, University of Belgrade, P. O. Box 51, 11158 Belgrade, Serbia; dusankam@chem.bg.ac.yu): Hydrophilic-interaction planar chromatography of some water-soluble Co(III) complexes on different adsorbents. J. Planar Chromatogr. 22, 249-253 (2009). Investigation of the chromatographic behavor of twelve neutral, mixed cobalt(III)complexes of the uns-cis-edda-type in six planar chromatographic systems. Four different stationary phases - silica gel, cyano phase, cellulose, and alumina - were combined with water-organic solvent (methanol or acetone) binary mobile phases. Hydrophilic-interaction chromatography was assumed to be the mechanism determining separation under normal-phase conditions (use of mobile phases with small amounts of water), whereas the use of mobile phases with high water content lead to reversed-phase chromatography.

qualitative identification 33a

35. Other technical products and complex mixtures

104 241 Dorina CASONI*, C. SARBU (*Department of Analytical Chemistry, Faculty of Chemistry and Chemical Engineering, Babes-Bolyai University, Arany Janos Str. No 11, 400028 Cluj-Napoca, Romania): Lipophilicity of some preservatives estimated by RP-TLC using stationary phases with different polarity. Chromatographia 70 (7-8), 1277-1282 (2009). HPTLC of preservatives on three stationary phases of different polarity: RP-18, RP-18W and cyano phase, with methanol - water mixtures in different volume proportions. The resulting RM values showed a linear decrease with increasing methanol concentration of the mobile phase (determination coefficients for all stationary phases were >0.98). The retention behavior of the preservatives on RP phase is in

good agreement with their polarity. Principal component analysis showed that for all three stationary phases the same lipophilic interactions take place.

HPTLC, autoradiography 35b

38. Chiral separation

104 242 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. Bio. Chromatogr. 21(10), 1064-1068 (2008). Indirect chiral TLC 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 on silica gel and RP-18 with phenol - water 3:1 and solvent combinations of acetonitrile and triethylamine phosphate buffer. The methods were applied for determination of the enantiomeric impurity of l-penicillamine, d-penicillamine, and pharmaceutical formulations of d-penicillamine.

pharmaceutical research, quality control, quantitative analysis, qualitative identification, comparison of methods 38, 28a

104 243 R. BHUSHAN*, S. TANWAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee - 247667, India): Different approaches of impregnation for resolution of enantiomers of atenolol, propranolol and salbutamol using Cu(II)-l-amino acid complexes for ligand exchange on commercial thin-layer chromatographic plates. J. Chromatogr. A 1217(8), 1396-1398 (2010). Sparation of enantiomers of atenolol, propranolol, and salbutamol using different loading/impregnation techniques for the Cu(II) complexes of l-proline, l-phenylalanine, l-histidine, N,N-dimethyl-l-phenylalanine, and l-tryptophan. TLC on silica gel with acetonitrile - methanol - 2 mM aqueous solution of Cu(II) 3:4:5. The different techniques were: A) using the Cu(II)-l-amino acid complex as chiral mobile phase additive, B) development of plates in solutions of Cu-complex, and C) with a solution of Cu(II)acetate as mobile phase additive for plates impregnated with the amino acids. Detection of zones by exposure to iodine vapor.

pharmaceutical research, quality control, quantitative analysis, qualitative identification, comparison of methods 38

104 244 R. BHUSHAN*, S. TANWAR (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee, 247667, India): Direct TLC resolution of the enantiomers of three beta-blockers by ligand exchange with Cu(II)-L-amino acid complex, using four different approaches. Chromatographia 70 (5-6), 1001-1006 (2009). Chiral TLC of the enantiomers of atenolol, propranolol, and salbutamol by complexation with Cu(II) cation and five L-amino acids using different techniques: 1) using the Cu(II)-L-amino acid complex as chiral mobile phase additive with untreated silica gel plates, 2) by mixing the Cu-complex with silica gel before preparing the TLC plates, 3) by development with solutions of the Cu-complex on untreated silica gel plates, and 4) by using a solution of Cu(II) acetate as mobile phase additive for plates prepared by mixing the L-amino acid with silica gel. Detection of zones by exposure to iodine vapor.

pharmaceutical research, qualitative identification 38

International Symposium for High-Performance Thin-Layer Chromatography BASEL, 06th–08th July 2011

We are enthusiastic to learn how many analysts are now confronted with situations where HPTLC is a suitable solution to their problems and is favored over better known and more widely used analytical methods. At the same time it is rather difficult nowadays to find a place for this "old technique" in the minds of opinion leaders, even if the need exists in analytical laboratories. This has arisen through inadequate information and training.

How to select the method; how to use HPTLC when it is described as a method of choice; avoidance of usual mistakes; which other samples may be well covered by this technique;… To address these issues an exchange of knowledge is foremost, from which sprang our motivation to hold again an international event with the Interlaken series spirit, last held in 1997. After Lyon 2003, Berlin 2006, and Helsinki 2008, we are pleased to announce that the 4th International Symposium for High-Performance Thin-Layer Chromatography will be organized on 06th–08th July 2011, in the Congress Centre of Basel, Switzerland. This new major issue will include a workshop, a symposium, and a manufacturers session.

The scientific program will feature invited keynote speakers, selected submitted lectures and poster presentations. Contributions are invited from all areas of high-performance thin-layer chromatography, but especially from colleagues working in the pharmaceutical, food, environmental and medical fields. Papers on theory, method development, validation, instrumental methods, hyphenated techniques, and quantitative applications in all areas of chemistry would be most welcome.

Deadlines for
Abstract submission (oral and poster): **March 1st 2011**
Final registration: **May 30th 2011**

The **participation fee** includes the full scientific program, and added to that, lunches, coffee breaks, the symposium dinner, and the social events.
Industrial **500** €
Academic **400** €
Students **200** €
Reduction of **100** € with ISPS or CCCM membership.

Special accommodation rates are available for hotels close to the symposium venue.

info@hptlc.com for general information
committee@hptlc.com for scientific matters
registration@hptlc.com for registration concerns

International Scientific Committee
Prof. Dr. Colin Poole, USA (chairman)
PD Dr. Gertrud Morlock, Germany (co-chairwoman)
Prof. Dr. Mario Vega, Chile
Prof. Dr. Wang Zheng Tao, China
Dr. Jacques Pothier, France
Prof. Dr. Ilkka Ojanperä, Finland
Prof. Dr. Lothar Kroh, Germany
Prof. Dr. Imre Klebovich, Hungary
Dr. J. Madhusudana Rao, India
Prof. Dr. Teresa Kowalska, Poland
Dr. Wanchai De-Eknamkul, Thailand
Dr. Irena Vovk, Slovenia
Dr. Vicente Cebolla, Spain
Dr. Eike Reich, Switzerland
Prof. Dr. Joseph Sherma, USA

www.hptlc.com

Fortsetzung von Seite 7

digkeit 3,5 cm/s) in das Anisaldehyd-Schwefelsäure-Reagenz (16 mL Schwefelsäure und 1 mL *p*-Methoxybenzaldehyd wurden unter Eiswasser-Kühlung zur Mischung aus 20 mL Essigsäure und 170 mL Methanol gegeben) getaucht, im warmen Luftstrom getrocknet und 2 min (Kieselgel-Platte) bzw. 30 s (RP18-Platte) auf dem DC-Plattenheizer bei 110 °C erhitzt.

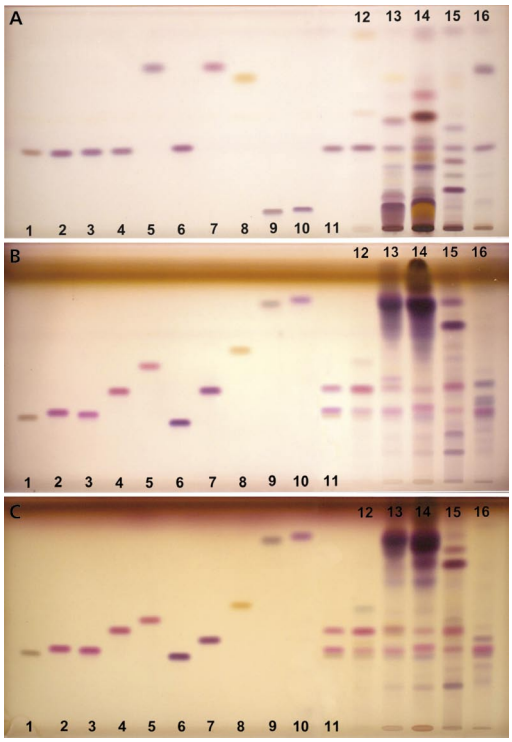
Anmerkung (Editor): Im Falle einer quantitativen Auswertung sollte die Platte auf den kalten Plattenheizer gelegt und mit diesem auf 110 °C erhitzt werden. Nur dies gewährleistet eine gleichmässige Erhitzung über die gesamte Plattenfläche und somit eine gute Präzision.

Dokumentation

Mit DigiStore 2 unter UV 366 nm und Weisslicht

Ergebnisse und Diskussion

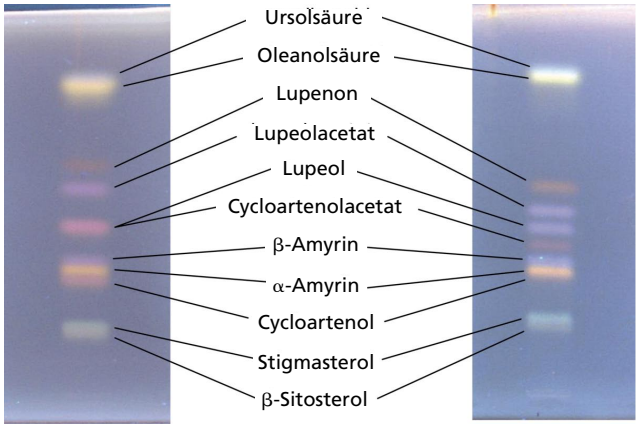
Die Trennung von Triterpenoiden mit unterschiedlichen funktionellen Gruppen (Alkohole, Säuren, Ketone und Ester) wurde auf HPTLC-Platten Kieselgel 60 erreicht, die Verwendung von HPTLC-Platten RP18 war jedoch entscheidend für die Trennung von vier isomeren Triterpenolen und zwei isomeren Triterpenolestern.



9

Trennung von Triterpenoiden auf (A) Kieselgel- und RP18-Phase mit (B) Ethylacetat – Acetonitril 3:2 und (C) Aceton – Acetonitril 5:1. Tracks: 1 α-Amyrin; 2 β-Amyrin; 3 δ-Amyrin; 4 Lupeol; 5 Lupeolacetat; 6 Cycloartenol; 7 Cycloartenolacetat; 8 Lupenon; 9 Ursolsäure; 10 Oleanolsäure; 11 α-Amyrin, β-Amyrin and Lupeol; 12 Kohl; 13 Rosmarin; 14 Salbei; 15 Eichenrinde; 16 Tomate

Die Trennung von isomeren Triterpenolen (α-Amyrin, β-Amyrin, Lupeol und Cycloartenol) wurde mittels Ethylacetat – Acetonitril 3:2 erreicht, allerdings gelang mit diesem Fliessmittelsystem die Trennung der isomeren Ester nicht (verminderte Auflösung zwischen Cycloartenolacetat und Lupeol). Andererseits wurden diese Ester mit Aceton – Acetonitril 5:1 aufgetrennt, jedoch wurde dabei Cycloartenol nicht ganz von α-Amyrin getrennt.



10

Charakteristische Fluoreszenz der Banden im UV 366 nm nach Derivatisierung mit Anisaldehyd-Schwefelsäure-Reagenz; Entwicklung auf RP18-Platten mit Ethylacetat – Acetonitril 3:2 (links) und Aceton – Acetonitril 5:1 (rechts)

Weiterhin ermöglichten die beiden Methoden auf der Umkehrphase bis zu einem gewissen Grad auch die Trennung von Triterpenoiden mit verschiedenen funktionellen Gruppen ohne Störung durch die strukturell verwandten Sterole.

Zur Identifizierung war die charakteristische Anfärbung und Fluoreszenz der Banden nach Derivatisierung mit dem Anisaldehyd-Schwefelsäure-Reagenz hilfreich. Durch diese selektive, postchromatographische Derivatisierung parallel für alle Proben ermöglicht die HPTLC ein einfaches, schnelles und kostengünstiges Screening auf Triterpenoide.

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

[1] M. Martelanc, I. Vovk, B. Simonovska, J. Chromatogr. A 1164 (2007) 145 und [2] dito 1216 (2009) 6662

* Dr. Irena Vovk, Nationales Institut für Chemie, Labor für Lebensmittelchemie, Hajdrihova 19, 1000 Ljubljana, Slovenien, irena.vovk@ki.si

Validierte HPTLC-Methode zur Bestimmung von Secoisolariciresinol-Diglucosid in Leinsamen

11

Prof. Silvia Coran

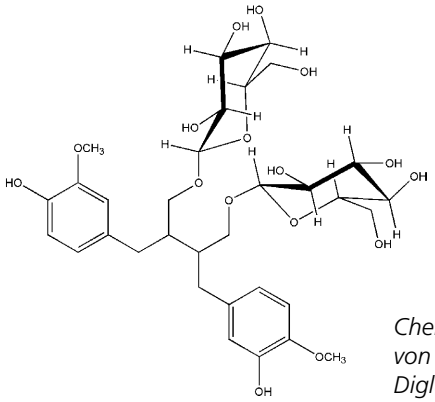
Den vollen Nutzen aus einem analytischen System zu ziehen erfordert zuverlässige Kenntnisse über die Geräte, ihre Funktionen und die verwendete Software. Gerade für die HPTLC ist dies besonders wichtig, da nur durch Koordination der einzelnen Analysenschritte das grosse Potential der Methode ausgeschöpft werden kann. Dieser Bereich bildet einen Schwerpunkt in der Arbeit von Silvia A. Coran*, ausserordentliche Professorin an der Universität Florenz, Pharmazeutische Chemie, Abteilung Pharmazeutische Wissenschaften. Eine sorgfältige Optimierung der verschiedenen HPTLC-Parameter ermöglicht die Entwicklung neuer analytischer Methoden, die eine leistungsstarke Alternative zur HPLC bilden. Die Validierung wurde nach den strengen Richtlinien für HPLC-Methoden erfolgreich durchgeführt.

Einleitung

Secoisolariciresinol-Diglucosid (SDG) ist ein Pflanzenlignan, das insbesondere in Leinsamen vorkommt. SDG ist von grossem Interesse, seit festgestellt wurde, dass es die wichtigste Vorstufe der Säugetier-Lignane ist, die möglicherweise vor hormonell bedingtem Krebs schützen können. Da SDG aus Pflanzen gewonnen wird und eine leichte Estrogenwirkung aufweist, wird es als Phyto-Estrogen bezeichnet.

Nachdem vor einigen Jahren eine neuartige Methode zur quantitativen Bestimmung von SDG in Leinsamen auf einer Kieselgel-Schicht [1] vorgeschlagen worden war, konnte jetzt eine signifikante Verbesserung der Methode erreicht werden. Dies wurde durch den Wechsel von der Kieselgel- auf die RP18-Schicht erreicht, welche die nötige Reproduzierbarkeit von Platte zu Platte gewährleistet, ohne dass die Schichtaktivität kontrolliert werden muss [2].

Diese selektive und robuste Methode wurde daraufhin komplett nach dem Validierungsprotokoll der SFSTP-Kommission (Société Française des Sciences et Techniques Pharmaceutiques) validiert [3]. Diese strengen Richtlinien für die Validierung quantitativer HPLC-Methoden beinhalten die Berechnung des Genauigkeitsprofils, des Gesamtfehlers sowie der Messunsicherheit der Methode. Die validierte HPTLC-Methode ist bezüglich Benutzerfreundlichkeit und Routinedurchsatz mit der gängigen HPLC-Methode konkurrenzfähig. Vor allem aber kann mit HPTLC die Probenvorbereitung beschleunigt werden, da das zeitaufwändige Entfetten der Leinsamen entfällt.



Chemische Struktur von Secoisolariciresinol-Diglucosid (SDG)

Probenvorbereitung

Die Leinsamen wurden mit flüssigem N_2 gekühlt und fein gemahlen. 100 mg dieses noch nicht entfetteten Pulvers wurden mit 2 mL wässriger NaOH 0.1 M vermischt und während 60 min bei 40 °C im Ultraschallbad extrahiert. Nach Abkühlen auf Raumtemperatur wurde die Probe mit HCl neutralisiert, mit 50 µL HCOOH auf pH 3 eingestellt und mit Methanol auf 10 mL verdünnt. Für die Quantifizierung wurden 5 µL dieser Lösung aufgetragen.

Standardlösung

4.3 mg SDG wurden in 5 mL MeOH (0.86 mg/mL)

gelöst. Diese Lösung wurde mit HCOOH 0.1 % auf eine Konzentration von 214 ng/µL verdünnt.

Schicht

HPTLC-Platten Kieselgel RP18 W $F_{254\,s}$, 10 x 10 cm (Merck), vorgewaschen durch Eintauchen in MeOH über Nacht, danach unter N_2 im Vakuum getrocknet.

Anmerkung (Editor): W bezeichnet mit Wasser benetzbare Schichten, die für die Auftragung wässriger Proben geeignet sind.

Probenauftragung

Bandförmig mit Linomat 5, 18 Bahnen auf den beiden gegenüberliegenden Seiten der Platte (pro Seite 9), Bandlänge 7 mm, Bahnabstand 8.7 mm, seitlicher Randabstand 15 mm, Auftragevolumen 1–5 µL (Standardlösung) und 5 µL (Proben), Auftragegeschwindigkeit 60 nL/s.

Chromatographie

In der Horizontal-Entwicklungskammer 10 x 10 cm mit 8 mL MeOH - HCOOH 0.1 % 2:2 (4 mL pro Seite), Laufstrecke 50 mm (Entwicklungszeit 12 min).

Densitometrie

TLC-Scanner 3 mit winCATS Software, Absorptionsmessung bei 282 nm, Spaltgrösse 5 x 0.45 mm, Messgeschwindigkeit 20 mm/s, Datenauflösung 50 µm/Schritt, polynome Regression über die Peakfläche.

Ergebnisse und Diskussion

Die Validierung wurde basierend auf dem Genauigkeitsprofil in zwei strategischen Schritten durchgeführt. Das Akzeptanzkriterium wurde gemäss FDA-Richtlinien für die Bioanalytik auf 15 % gesetzt. Für die Vorvalidierung wurde eine Fünfpunkt-Kalibration im Bereich von 214–1071 ng/Band in Vierfachbestimmung durchgeführt und an drei verschiedenen Tagen wiederholt. Für die Berechnung der Regressionsfunktion wurden vier Modelle beigezogen: linear, gewichtet linear (1/x), quadratisch und gewichtet quadratisch (1/x). Der mittlere Bias (Abweichung vom »wahren« Wert), die Wiederholbarkeit und die interne Vergleichspräzision wurden mithilfe der vier Modelle bei jeder Standardkonzentration berechnet. Die im Vertrauensbereich erhaltenen Genauigkeitsprofile zeigten, dass das quadratische Modell mit einem Arbeitsbereichs-Index von 1.00 und der kleinsten Abweichung am geeignetsten war.

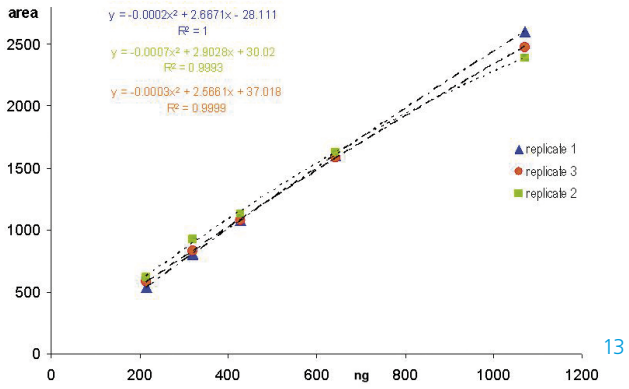
12

Horizontal-Entwicklungskammer

Die Horizontal-Entwicklungskammer (HDC, Horizontal Developing Chamber) erlaubt es, die Platte von beiden gegenüberliegenden Seiten zur Mitte zu entwickeln und damit die Probenzahl pro Platte gegenüber herkömmlichen Entwicklungstechniken zu verdoppeln. Hier wird die HDC zur Trennung von Secoisolariciresinol-Diglucosid in Leinsamen eingesetzt und erweist sich als sehr zeitsparend. Es werden in dieser Anwendung mit der HDC 10 x 10 cm 18 Proben zeitgleich in 12 min entwickelt. Bei Einsatz der HDC 20 x 10 cm wären es 36 Proben in 12 min! Das sind 40 bzw. 20 Sekunden pro Probe.

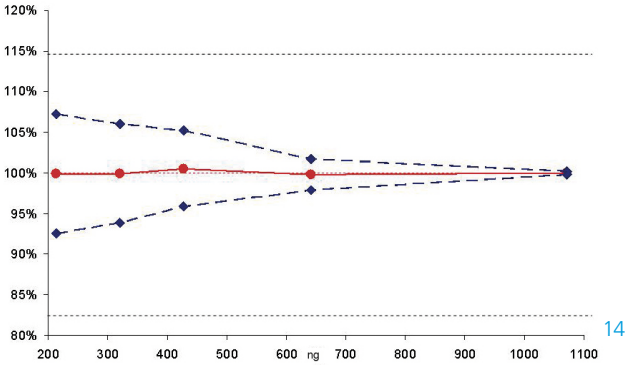
Für die Entwicklung werden bei dieser Anwendung 4 mL Fliessmittel pro Seite eingesetzt, es reichen jedoch schon 2,5 mL aus. Somit ist die HDC auch hier im Vergleich zu anderen Kammertypen am sparsamsten, z. B. benötigt die HDC im Vergleich zu einer Flachbodenkammer 75 % weniger Fliessmittel und zwar nur 0,14 mL pro Probe. Auch die Entsorgungkosten für das Fliessmittel sind weit unter 0,01 Cent pro Probe.

Ihre einfache Handhabung beim Konditionieren in der Tank-Konfiguration oder bei der Entwicklung in der Sandwich-Konfiguration machen sie sehr flexibel. Die HDC ist unübertroffen ökonomisch, vielseitig und gut reproduzierbar im Routinebetrieb. Testen Sie sie!



13

Polynome Kalibrierfunktionen der 3 Wiederholmessungen

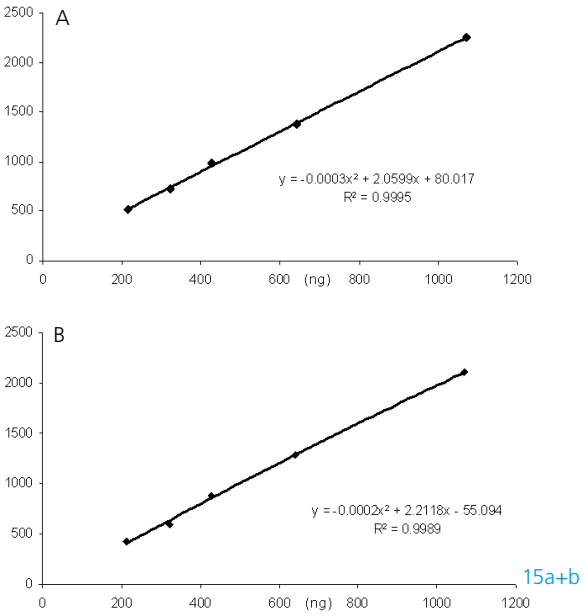


14

Genauigkeitsprofil von SDG mittels der quadratischen Regressionsanalyse: ◆ Vertrauensbereich, ● relative Richtigkeit

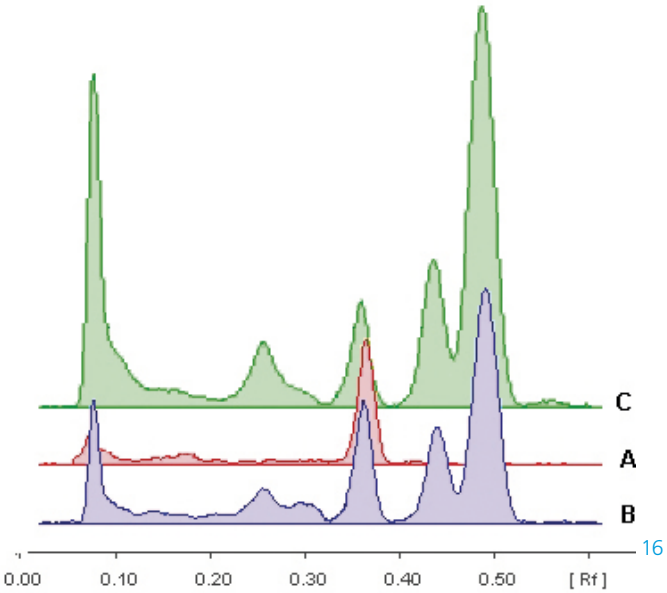
Da keine Matrix-Blindprobe vorhanden war, wurde das Standardadditions-Verfahren mit Interpolation gewählt. Die durch quadratische Regression erhaltene Standardadditionskurve stimmte mit der Kalibrierkurve der reinen Substanz ohne Matrix überein, womit die Eignung der Kalibrierung bestätigt werden konnte. Beide Korrelationskoeffizienten lagen über 0.999. Nach erfolgreicher Validierung wurde für die Routinebestimmung auf 10 x 10 cm-Platten eine Dreipunkt-Kalibration (321, 642 und 1071 ng/Band) gewählt.

Für die Bestimmung der Richtigkeit wurde eine schon untersuchte hydrolysierte Probe mit verschiedenen Standardkonzentrationen aufgestockt. Die Wiederfindungsrate lag zwischen 100.4 und 103.2 %. Die Methode erfüllte die Anforderungen mit einem Fehler innerhalb der Akzeptanzkriterien (95 %) und einer Standardabweichung von 2.0 bzw. 3.6 % für die Wiederholbarkeit und interne Vergleichspräzision. Zur Überprüfung der Methode wurde der SDG-Gehalt von fünf verschiedenen Leinsamen-Sorten bestimmt. Die gemessenen Werte lagen zwischen 0.9 und 1.3 % bei einer relativen Standardabweichung (*%RSD*) von ≤ 2.3 % (n = 3).



15a+b

Polynome Kalibrierfunktion von SDG: (A) ohne Matrix, (B) mit Matrix



16

HPTLC-Profil von SDG-Standard (A) und Leinsamenproben (B und C) bei 282 nm

Weitere Informationen sind bei der Autorin auf Anfrage erhältlich.

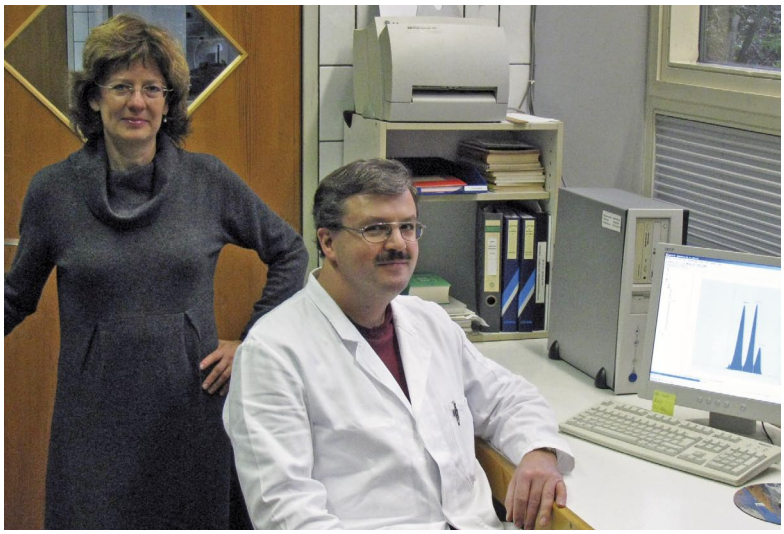
*Prof. Silvia A. Coran, Universität Florenz, Pharmazeutische Fakultät, Abteilung Pharmazeutische Wissenschaften, Via Ugo Schiff 6, 50019 Sesto Fiorentino (Florenz), Italien, coran©unifi.it

[1] S.A. Coran, V. Giannellini, M. Bambagiotti-Alberti, J Chromatogr A 1045 (2004) 217.

[2] S.A. Coran, G. Bartolucci, M. Bambagiotti-Alberti, J Chromatogr A 1207 (2008) 155.

[3] P. Hubert et al., J Pharm Biomed Anal 45 (2007) 82.

Bestimmung von Aloe Vera-Gel in Kosmetika



17

Evamaria Kratz, Jürgen Geisser

Das Kosmetikteam des CVUA Karlsruhe* prüft amtlich erhobene Stichproben des Einzelhandels oder von baden-württembergischen Herstellern bzw. Importeuren von kosmetischen Mitteln (Marktüberwachung) auf Einhaltung der kosmetikrechtlichen Vorschriften.

Einleitung

Das Gel aus dem Blattinneren der Aloe Vera-Pflanze wird traditionell zur Hautpflege eingesetzt. In den letzten Jahren wurden zahlreiche kosmetische Produkte auf den Markt gebracht, bei denen der Aloe Vera-Extrakt als wirksamer Inhaltsstoff deklariert wird. Da Aloe Vera ein teurer Rohstoff ist, sind Streckungen des Gels mit Wasser und Geliermittel oder eine extrem niedrige Dosierung im Endprodukt trotz werblicher Hervorhebung nicht auszuschliessen. Das Aloe Vera-Gel wird auch als aufkonzentrierter Rohstoff (z.B. 10-fach oder 200-fach) angeboten.

Nachfolgend beschrieben ist die Untersuchung solcher Aloe Vera-Produkte, bei denen Aloe Vera besonders werbewirksam hervorgehoben ist und in Gehalten von mindestens 5 % zu erwarten wäre. Ab dieser Konzentration ist auch eine Wirksamkeit anzunehmen, wie z.B. Steigerung der Hautfeuchtigkeit oder besondere Pflegeeffekte. Als Markerverbindung für den Aloe Vera-Zusatz wurde Aloverose gewählt. Diese acetylierte Polymannose (Molmasse 50–500 kDa, ca. 1,1 Acetylgruppen pro Mannose-Molekül) liegt zu ca. 20 % im ca. 0,5 % Feststoffgehalt des frischen Aloe Vera-Gels vor und ist somit besser zur analytischen Überwachung geeignet als weitere Inhaltsstoffe wie Äpfelsäure und Glucose.

Bisher war lediglich für den Aloe Vera-Rohstoff eine ^{1}H-NMR-Methode etabliert, bei der Aloverose über die Signale der Acetylgruppen quantifiziert wird. Mit der neu entwickelten HPTLC-Methode kann erstmals geprüft werden, ob kosmetische Fertigerzeugnisse mit Aloe Vera der Verbrauchererwartung bzw. den Wirksamkeitsaussagen entsprechen oder eine Irreführung zugrunde liegt. Die Quantifizierung des Aloe Vera-Zusatzes erfolgt über Mannose nach Hydrolyse von Aloverose. Die im Lebensmittelbereich etablierten HPLC-Methoden zur Zuckerbestimmung erwiesen sich aufgrund unzureichender Nachweisgrenzen als nicht geeignet.

Standardlösung

Als Stammlösungen werden 20 mg Aloverose mit Wasser zu 20 mL gelöst; bei Glucose und Galactose sind es 50 mg auf 50 mL (tiefgefroren 1 Jahr haltbar). 0,8 mL Aloverose-Stammlösung wird entsprechend der Probelösung hydrolysiert und neutralisiert. Als Standardgemischlösung werden der hydrolysierten Aloverose-Stammlösung je 1 mL Glucose- und Galactose-Stammlösung zugegeben (0,8 bzw. 1 mg/100 mL).

Probenvorbereitung

Je nach Produkt werden 1 bis 5 g Probe mit Wasser homogenisiert. Lipophile Probenbestandteile werden aus der angesäuerten Probelösung durch Ausschütteln mit Diethylether abgetrennt. Aloverose wird durch Hydrolyse mit Schwefelsäure (3,35 M, 3 h, 85 °C) zu Mannose abgebaut und anschliessend neutralisiert. Wasserstoffperoxid, das in manchen Produkten enthalten ist, muss vor der Hydrolyse durch Kaliumjodid zerstört werden.

Schicht

HPTLC-Platten Kieselgel 60 (Merck), 20 × 10 cm, imprägniert durch Tauchen in 0,5 M NaH_2PO_4-Lösung (Eintauchzeit 1 s, Eintauchgeschwindigkeit 4 cm/s), vortrocknen bei Raumtemperatur und anschliessendes Trocknen auf dem DC-Plattenheizer (5 min, 60 °C).

Probenauftragung

Bandförmig mit DC Probenautomat 4, 18 Bahnen, Bandlänge 6 mm, Auftragevolumen 1–10 µL Standardlösung und je nach Produkt 1–15 µL Probelösung, unterer Randabstand 10 mm, seitlicher Randabstand 12 mm, Bahnabstand 10 mm.

Anmerkung (Editor): Bei der hier vorliegenden starken Matrixbelastung empfiehlt es sich, die Probelösung in Rechteckform aufzutragen, eine Möglichkeit, die der ATS4 bietet (siehe Seite 15), und vor der eigentlichen Chromatographie zu fokussieren.

Chromatographie

In der Flachbodenkammer 20 × 10 cm mit 20 mL Aceton – *i*-Propanol – Ameisensäure (0,1 M) 2:2:1, Laufstrecke 80 mm (Laufzeit ca. 75 min, Zweifachentwicklung bei Produkten mit hohem Glucosegehalt empfehlenswert), Trocknen der Platte auf dem DC-Plattenheizer (5 min, 60 °C).

Anmerkung (Editor): Laufstrecken über 60 mm führen zwar zu grösseren Laufstreckendifferenzen, aber meist auch zu einer Peakverbreiterung. In der Regel verbessert sich die Auflösung dadurch nicht.

Postchromatographische Derivatisierung

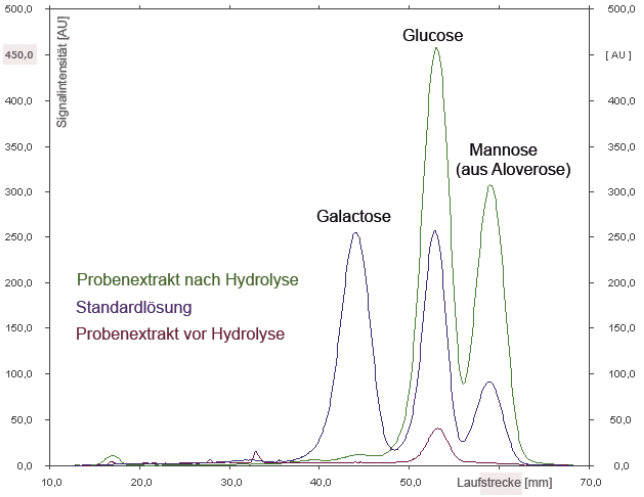
Durch Tauchen der Platte (Eintauchzeit 1 s, Eintauchgeschwindigkeit 4 cm/s) in 4-Aminobenzoesäure-Reagenz (1 g 4-Aminobenzoesäure in 36 mL Eisessig lösen, 40 mL Wasser, dann 2 mL 85 %ige *o*-Phosphorsäure und 120 mL Aceton zugeben) und Erhitzen auf dem DC-Plattenheizer (10 min, 110 °C). Dieses Reagenz ist gekühlt unter Stickstoff 2 Wochen haltbar.

Densitometrie

TLC-Scanner 3 mit winCATS Software, Fluoreszenzmessung mit Hg-Lampe bei 366 nm, Auswertung über die Peakfläche mit winCATS Planar Chromatography Manager

Ergebnisse und Diskussion

Nach Hydrolyse von Aloverose wird Mannose mittels HPTLC gut von anderen Kohlenhydraten (ausser Fructose) in der Probe abgetrennt. Die Derivatisierung der Mannose mit 4-Aminobenzoesäure zu einem fluoreszierenden Produkt ermöglicht den empfindlichen Nachweis. Die vergleichende Analyse der Probe vor und nach der Hydrolyse lässt erkennen, ob Mannose vorab enthalten ist. Bei Anwesenheit von Aloe Vera ist Glucose als natürlicher Bestandteil des Aloe Vera-Gels schon vor der Hydrolyse nachweisbar.

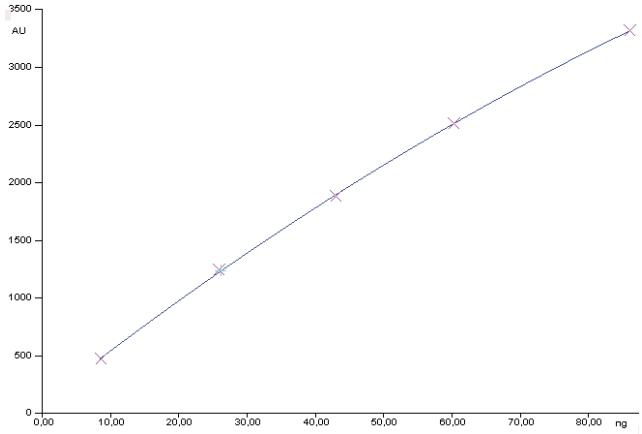


Vergleichende Analyse des Probenextraktes vor und nach der Hydrolyse

Nach Hydrolyse von in kosmetischen Formulierungen auch vorkommendem Maltodextrin steigt die Glucose-Konzentration an. Mannosehaltige Dickungsmittel werden nach der Hydrolyse über charakteristische Bausteine wie Glucuronsäure bei Xanthan (organische Säuren werden mit einer separaten HPTLC-Methode bestimmt) und Galactose bei Galactomannanen (Guarmehl oder Johannisbrotkernmehl) bestimmt und deren Verhältnis zu Mannose anteilig ermittelt.

Die Bestimmungsgrenze von Aloe Vera-Gel in Produkten liegt bei ca. 3 %, was für die Problemstellung ausreichend ist. Die Reststandardabweichung

der polynomen Kalibrierfunktion für Mannose liegt im Arbeitsbereich von 8–80 ng/Band bei 2,1 % und bei einer Matrixkalibration bei 4,0 %.



19

Polynome Auswertung (Peakfläche) im Bereich 8–80 ng/Band Mannose aus Aloverose

Die neue Methode ermöglicht es dem Kosmetikteam des CVUA Karlsruhe, bei Kosmetikproben, deren Werbung auf einen besonders hohen Aloe Vera-Gehalt hinweist, diese Aussagen auf ihren Wahrheitsgehalt zu überprüfen. Es ergaben sich in den letzten beiden Jahren mehrere Beanstandungen, die die betroffenen Kosmetikhersteller dazu veranlassten, in Zukunft mehr Sorgfalt auf die Qualität und Quantität der Aloe Vera-Rohstoffe zu legen.

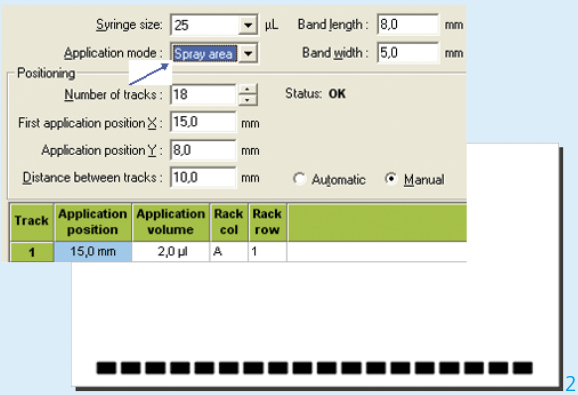
Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

*J. Geisser und E. Kratz, Chemisches und Veterinäruntersuchungsamt Karlsruhe, Weissenburger Str. 3, 76187 Karlsruhe, Juergen.Geisser@cvuaka.bwl.de; Fachgebiet Kosmetika unter www.ua-bw.de

20

Flächenauftragung mit dem DC-Probenautomat (ATS 4)

Bei der Bestimmung von Aloverose in kosmetischen Produkten limitieren die durch die Probenvorbereitung generierte hohe Salzfracht und weitere Bestandteile wie Tenside das Probevolumen zur bandförmigen Auftragung. Für solche Anwendungen ist die flächenförmige Auftragung von Proben von Vorteil: Trotz hoher Matrixbelastung kann das Auftragevolumen erhöht und somit die Bestimmungsgrenze verbessert werden. Je nach Fliessmittel ist eine nachfolgende Fokussierung der Analyten relevant.

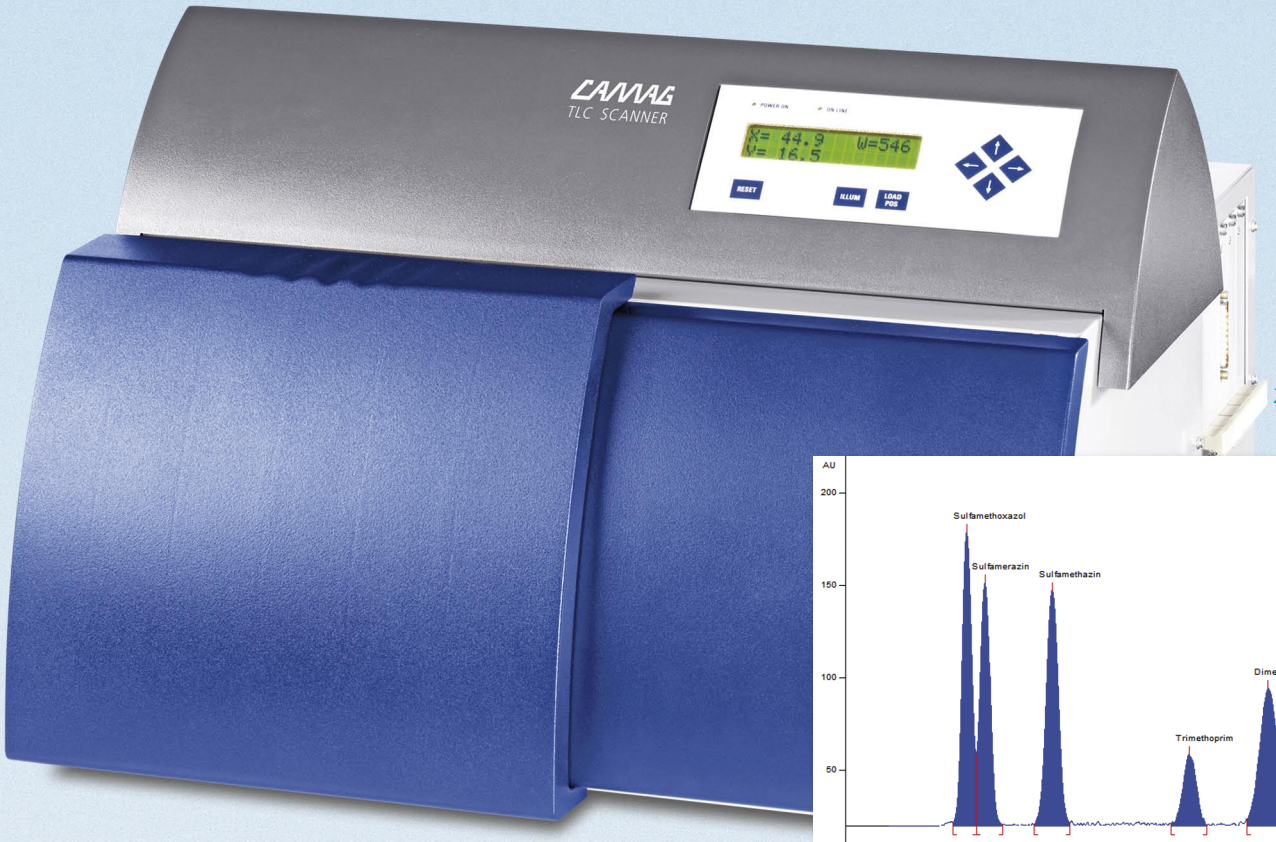


21

Für grössere, wässrige Probenvolumina empfiehlt sich zudem die ATS4-Option mit beheizbarer Sprühdüse (bis 60 °C), denn diese ermöglicht eine deutliche Verkürzung der Auftragezeit.

CAMAG TLC-Scanner

Der CAMAG TLC-Scanner in Verbindung mit winCATS Software ist der fortschrittlichste Messplatz zur densitometrischen Auswertung von Dünnschicht-Chromatogrammen.



22

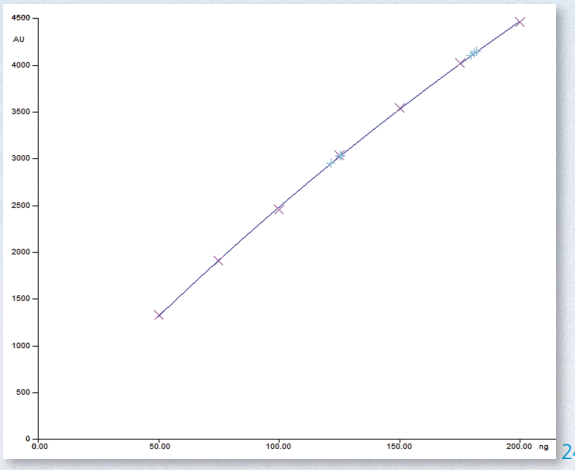
23

Densitogramm von Antibiotika nach der Chromatographie

Die wichtigsten Eigenschaften in Kürze:

- Messung in Remission, wahlweise Absorption oder Fluoreszenz
- Objektformat bis 200 x 200 mm
- Spektralbereich 190–900 nm
- Automatische Schaltung aller Lampen: Deuterium-, Halogen-Wolfram- und Hg-Hochdrucklampe
- Messchrittauflösung 25–200 µm
- Messgeschwindigkeit 1–100 mm/s
- Spektrenaufnahme mit bis zu 100 mm/s
- Automatische Einstellung der Verstärkung
- Schnelle Datenübertragung

Näheres unter: **www.camag.com/tlc-scanner**



24

Kalibrierkurve von Sulfamethazin

CAMAG

Weltweit führend in der Planar-Chromatographie

CBS 104 D 996.1310-7