CBS 99 • September 2007

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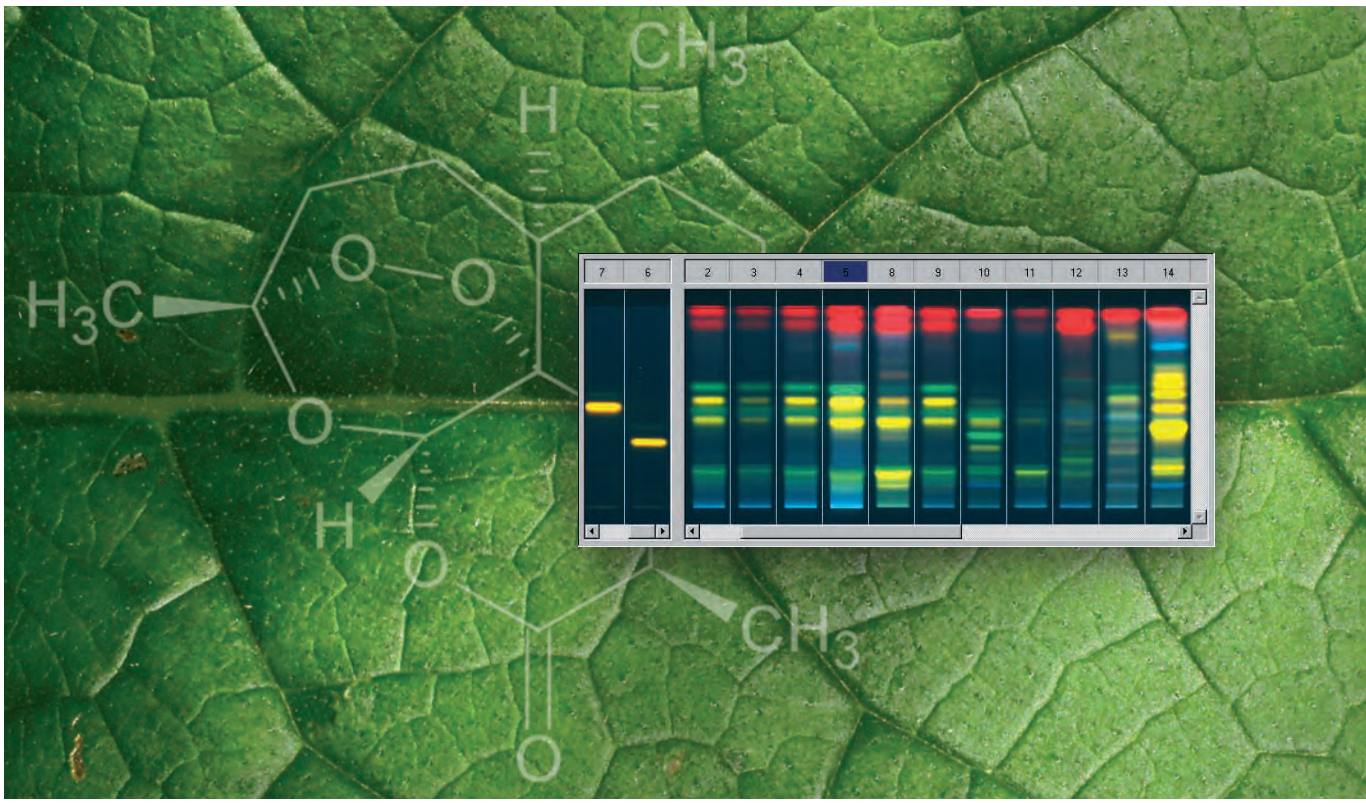

Analyse von Pflanzeninhaltsstoffen – Überlegenheit der Planar-Chromatographie dank digitaler Bilddokumentation

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99

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IN DIESER AUSGABE

Verfahren, Anwendungen

American Herbal Pharmacopoeia: HPTLC Analyse von arzneipflanzlichen Rohstoffen 2
Planar-Chromatographie – unverzichtbar in der Qualitätskontrolle von Arzneipflanzen-Produkten 3
Quantitative HPTLC-Analyse von Artemisinin in getrockneten Beifuss-Blättern (*Artemisia annua L.*) 4–5
Bestimmung von Sildenafil in Arzneimitteln und Aphrodisiaka auf pflanzlicher Basis..... 6–7
Eine universelle Detektion auf Basis der Fluoreszenzänderung 9–10
Bioluminex™: Ein effektiver und zugleich leicht einsetzbarer Schnelltest 11–13
Bestimmung von Acrylamid in Trinkwasser 14–15

In dieser Ausgabe hervorgehobene Produkte und Dienstleistungen

CAMAG BioLuminizer..... 11
Visueller Vergleich mehrerer Proben – ganz einfach! 16

Rubrik: Kennen Sie CAMAG?

Neue Mitarbeiter der CAMAG..... 8

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Aus der Praxis

American Herbal Pharmacopoeia: HPTLC-Analyse von arzneipflanzlichen Rohstoffen (Botanical Ingredients)


1

*Roy Upton** ▶
Präsident der American Herbal Pharmacopoeia

In den letzten Jahren wurden Hunderte bekannte Heilpflanzen von Aloe Vera bis Ginseng als Rohstoffe zur Herstellung einer breiten Palette von Produkten verwendet, die von Kaugummi über Toilettenpapier bis zu traditionellen und modernen Medikamenten reicht. Der steigende Einsatz derartiger Rohstoffe erfordert geeignete Methoden zur Bestimmung von Identität, Reinheit und Gehalt. Diese Methoden müssen nicht nur technisch leistungsfähig sein, sondern auch schnell und kostengünstig sowie kompatibel mit den Anforderungen der Guten Herstellungspraxis (GMP).

Für Medikamente sind Qualitätskontrollstandards seit Jahrzehnten im Europäischen und in Nationalen Arzneibüchern verankert. Jedoch ist in Nordamerika und vielen Entwicklungsländern die Schaffung von Standards für arzneipflanzliche Rohstoffe ein Novum. Das Fehlen von Qualitätsstandards führte weltweit zu vielen Fällen von leichten bis schwerwiegenden Nebenwirkungen, die von Lebertoxizität bis zum Tod reichen.

Viele dieser Nebenwirkungen hätten vermieden werden können, wenn der Industrie Identitätsstandards und effiziente analytische Methoden zur Verfügung gestanden hätten und diese eingesetzt worden wären. Die American Herbal Pharmacopoeia (AHP) wurde mit dem klaren Ziel gegründet, Standards für die Identifizierung und Qualitätskontrolle von Botanicals zu schaffen. Die AHP erkennt die Bedeutung von Heilpflanzen im Gesundheitswesen an und ebenso, dass Qualitätsstandards auf pflanzliche Arzneimittel angewendet werden müssen, um die Vorteile dieser Medikamente risikofrei nutzen zu können.

Für die AHP ist die Planar-Chromatographie unersetzlich. Mit HPTLC können viele Proben parallel untersucht werden und dies im Vergleich zu anderen analytischen Methoden mit einem Bruchteil an Zeit und Kosten. Mittels HPTLC können Proben nach weit mehr Gesichtspunkten geprüft werden als mit den meisten anderen analytischen Verfahren. Damit wird komplexe Information in einem hohen Mass zugänglich. Für die AHP ist die HPTLC die Methode der Wahl zur Qualitätssicherung pflanzlicher Rohstoffe und Endprodukte und für deren chemische Charakterisierung. Für die Industrie gehört die HPTLC zu den am breitesten einsetzbaren, praktischsten und kostengünstigsten Methoden für die Qualitätssicherung von Botanicals. Der Einsatz der HPTLC nimmt in Nordamerika zu, und das Wachstumspotential ist zu recht sehr hoch.

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

Planar-Chromatographie – unverzichtbar in der Qualitätskontrolle pflanzlicher Zubereitungen

◀ *Prof. Dr. Beat Meier**
Präsident der Expertengruppe Phytochemie der Schweizerischen Arzneibuch Organisation

2

Es ist wohl kein Zufall, dass Mentoren der Dünnschicht-Chromatographie, wie Egon Stahl oder Hellmut Jork, Wissenschaftler waren, die sich als Pharmakognost mit Arzneipflanzen beschäftigten. Vor der Einführung der DC beschränkte sich die Identitätsprüfung von Arzneipflanzen auf deren Morphologie und einige nasschemische Farbreaktionen. Die DC erlaubte dann auf relativ einfache Weise einen vertieften Einblick in die »Chemie« der Pflanzen. Die Metaboliten des Sekundärstoffwechsels zeigten von Art zu Art eine Variabilität, welche die Identifizierung von Pflanzen auf diesem Weg ermöglichte. Dies war insbesondere dort von Bedeutung, wo durch Extraktionsverfahren die Strukturelemente einer Pflanze wegfallen: In Extrakten aller Art und in Endprodukten, unabhängig davon, ob es sich um Arzneimittel oder eher als Lebensmittel verwendete Zubereitungen handelte. Leider hielt die Dünnschicht-Chromatographie vorübergehend technologisch nicht Schritt mit den säulenchromatographischen Verfahren und handelte sich dadurch einen schlechten Ruf infolge ungenügender Reproduzierbarkeit ein. Das lag aber nicht an der Methode, sondern viel eher bei deren Anwendern, die sich zu wenig um die Bedingungen kümmerten, die notwendig sind, um DC-Verfahren reproduzierbar durchzuführen. Die DC galt als einfaches und billiges Verfahren, was notwendige Investitionen in innovative Technik offensichtlich verhinderte.

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Prof. Dr. Beat Meier ist auch Präsident des Permanenten Komitees der GA (Gesellschaft für Arzneimittelpflanzen-Forschung) zur Herstellung und Qualitätskontrolle von Arzneipflanzen-Produkten

In den letzten Jahren hat zum Glück ein Umdenken stattgefunden. Als Beispiel dafür kann das Europäische Arzneibuch betrachtet werden: Vor einigen Jahren wurde die allgemeine Monographie Dünnschicht-Chromatographie an die Erfordernisse der Moderne angepasst. Es ist nun möglich, analog zur HPLC mit High-Performance-Materialien zu arbeiten, was die Trennungen deutlich verbessert, Lösungsmittel spart und zu schnelleren Resultaten führt. Die Monographien im Europäischen Arzneibuch enthalten obligatorisch ein für die Pflanze möglichst spezifisches HPTLC-Chromatogramm zur Identitätsprüfung. Doch die HPTLC lässt sich nicht nur für Identitätsprüfungen einsetzen, sie leistet ihre Dienste auch beim Nachweis unerwünschter Substanzen, wie z.B. der Aristolochiasäuren, und ist ein wichtiges Instrument der Stabilitätsprüfung.

Kein anderes analytisches Verfahren vermag bis heute die Komplexizität eines Vielstoffgemisches, wie es ein Extrakt darstellt, in einem sogenannten Fingerprint visuell besser darzustellen als die HPTLC. Der Vorteil, zahlreiche Proben nebeneinander auftragen zu können, ermöglicht auf einfache Weise Vergleiche zwischen verschiedenen Ausgangsmaterialien, auf verschiedenen Stufen der Extraktzubereitung im Sinn einer in-process-Kontrolle, sowie zwischen verschiedenen Chargen und Produkten. Auch Veränderungen über die Zeit können so studiert werden. So hat sich die Planar-Chromatographie zum unverzichtbaren Bestandteil der Arzneipflanzenanalytik etabliert.

Quantitative HPTLC-Analyse von Artemisinin in getrockneten Beifuss-Blättern (*Artemisia annua L.*)

▲ *Das CAMAG Labor: Dr. Anita Ankli, Valeria Widmer, Dr. Eike Reich, Daniel Handloser, Mario Steiner (von links nach rechts)* 3

Das CAMAG Labor* ist ein Team von Wissenschaftlern und Applikations-Spezialisten mit grosser Erfahrung auf vielen Einsatzgebieten der Dünnschicht-Chromatographie. Sie arbeiten darauf hin, die weltweite Akzeptanz der HPTLC als standardisierte Analysenmethode zu erhöhen. Durch ihr umfangreiches Dienstleistungsangebot wollen sie Kunden dabei helfen, analytische Probleme mit HPTLC zu lösen.

Einleitung

Gegenwärtig sieht die Weltgesundheitsorganisation (WHO) Malaria als eine der grössten Bedrohungen der Menschheit an. Seit Mitte des zwanzigsten Jahrhunderts sind zwar viele Medikamente erfolgreich zur Prävention und Behandlung der Krankheit eingesetzt worden, aber eine zunehmende Resistenz der Erreger gegen zuvor wirksame Mittel fordern die Malariatherapie heraus. Der heutzutage effizienteste medizinische Ansatz ist die so genannte auf Artemisinin basierende Kombinations-Therapie (ACT) unter Verwendung von aus einjährigem Beifuss (*Artemisia annua*) isoliertem Artemisinin und davon synthetisch abgeleiteten Substanzen. Da die Totalsynthese von Artemisinin unrentabel ist, fokussiert die Pharmaindustrie den Grossanbau von Artemisinin-reichen Arten und die Bestimmung der günstigsten Wachstumsbedingungen, Erntezeitpunkte und Extraktionsverfahren.

Viele wissenschaftliche Artikel sind bereits dazu publiziert worden. Die meisten Methoden sind allerdings sehr komplex, teuer oder eher unpraktikabel. Zum Beispiel war die Trennung von Artemisinin und anderen Probenkomponenten und Detektion mit Schwefelsäure unzureichend. Daher wurde eine spezifische und zugleich einfache, kostengünstige Methode entwickelt [1], mit der Artemisinin quantitativ in Beifussblättern bestimmt werden kann. Die Abwesendheit eines jeglichen Chromophors erforderte die Derivatisierung mit Anisaldehyd-Reagenz. In einem weiten Konzentrationsbereich von 0.05 bis 3.25 % kann in nur einer Stunde der Artemisiningehalt von neun Proben in getrockneten Blättern gescreent werden und im linearen Bereich quantifiziert werden.

Probenvorbereitung

Getrocknete Blätter von *Artemisia annua* wurden fein gemahlen. 200 mg wurden mit 10 mL Toluen versetzt und im Ultraschall über 10 min bei RT (23 °C) extrahiert. Nach dem Zentrifugieren wurde der Überstand zum Screening eingesetzt. Zur Gehaltsbestimmung wurden die Lösungen entsprechend verdünnt.

Standardlösung

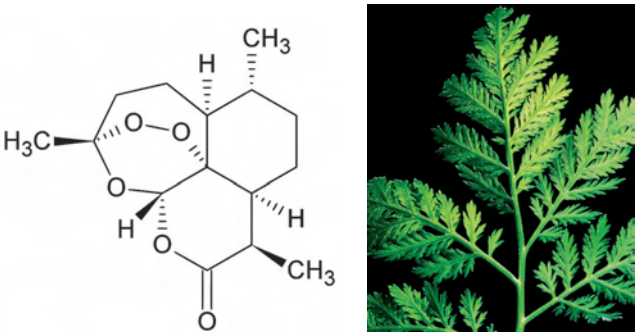
Zum Screening wurden 10 mg Artemisinin in 100 mL Toluen gelöst und für die Gehaltsbestimmung 1:10 mit Toluen verdünnt.

Schicht

HPTLC-Platten Kieselgel 60 F_{254} Merck, 20×10 cm

Probenauftragung

Bandförmig (8 mm Bandlänge) mit dem DC-Probenautomat 4, Auftragevolumen 2–10 µL Probe- und Standardlösung, Bahnabstand 10 mm, unterer Randabstand 8 mm, linker Randabstand 20 mm



▲ *Struktur von Artemisinin und ein Beifussblatt (Artemisia annua L.)* 4

Chromatographie

In der Doppeltrogkammer mit Cyclohexan – Ethylacetat – Essigsäure 20:10:1 nach 20 min Kammersättigung (10 mL Fliessmittel pro Trog), Laufstrecke 70 mm vom unteren Plattenrand.

Derivatisierung

Mit der Chromatogramm Tauchvorrichtung III für 1 s in Anisaldehyd-Reagenz (2 mL Anisaldehyd in 100 mL Ethanol und 80 mL Wasser plus 20 mL Essigsäure und 4 mL Schwefelsäure) tauchen. Nach 1 min wurde die Platte auf dem DC-Plattenheizer für 12 min bei 100 °C erhitzt.

Dokumentation

Mit dem DigiStore 2-System unter UV366 nm und unter Weisslichtbeleuchtung

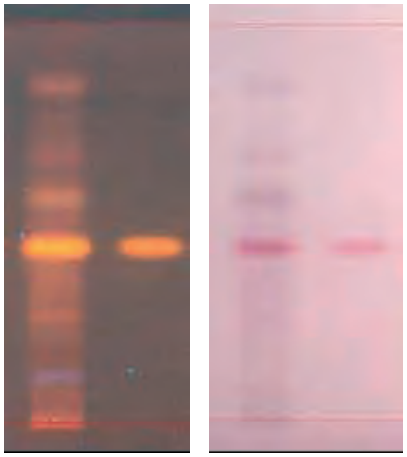
Auswertung

Mit dem TLC-Scanner 3 im Fluoreszenzmodus bei 520/>540 nm mit der Wolframlampe, Spaltgrösse 4 × 0.2 mm, zum Screening wahlweise Videodensitometrie mit der Software VideoScan.

Anmerkung: Der Unterschied zwischen Anregungs- und Emissionswellenlänge sollte mindestens 30 nm betragen. Dieser Fall ist ein Kompromiss auf Grund des vorhandenen Kantenfilters.

Ergebnisse und Diskussion

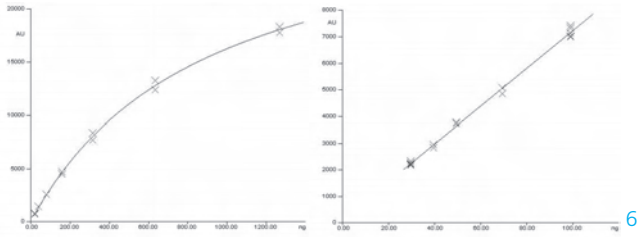
Artemisinin wird mit dem Anisaldehyd-Reagenz zu einem orangefarbenen Fluorophor derivatisiert, das bei 520/>540 nm selektiv detektiert werden kann.



5

▲ *Chromatogramm-Ausschnitt mit Artemisia annua Extrakt und Artemisinin-Standard*

Im Vergleich zur konventionellen Absorptionsmessung bei 535 nm ist das im Fluoreszenzmodus erhaltene Signal dreimal grösser. Mit der Michaelis–Menten 1 Regression ist es möglich, Artemisinin in einem weiten Konzentrationsbereich von 0.05 bis 3.25 % in getrockneten Blättern zu bestimmen. Für die Gehaltsbestimmung wird der lineare Arbeitsbereich zwischen 30 und 120 ng/Zone verwendet.



6

▲ *Kalibrierungskurven für Artemisinin-Screening (links) und Gehaltsbestimmung (rechts)*

Tabelle mit einigen Validierungsdaten:

Validierungsparameter	Ergebnisse für Artemisinin
Screening	20–1300 ng/Zone (0.05–3.25 %) y = 30722.958 x / (853.168 + x) *RSD* = 3.67 %
Linearität	30–120 ng/Zone (0.075–0.3 %) y = 122.6 + 71.04 x *RSD* = 2.92 %, r = 0.9983
Präzision	*RSD* = 0.77 % (*n* = 5)
Wiederholbarkeit	*RSD* = 1.9 % (*n* = 3)[1]
Laborpräzision	*RSD* = 1.2 % (*n* = 3)[2]
Präzision der Extraktion	*RSD* = 5.2 % (*n* = 5)[3]

[1] Ein Aliquot als 3-fach Bestimmung auf 3 Platten am selben Tag

[2] Eine Probe auf drei Platten an veschiedenen Tagen

[3] 5 Aufarbeitungen einer Probe auf einer Platte

Weitere Informationen erhalten Sie auf Anfrage von den Autoren.

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

*www.camag-laboratory.com

Bestimmung von Sildenafil in Arzneimitteln und Aphrodisiaka auf pflanzlicher Basis

▲ *Dr. Ehab A. Abourashed*

Dr. Abourashed* ist Direktor der Qualitätssicherung bei ElSohly Laboratories, Inc. (ELI) und Ausserordentlicher Professor in der Forschung am National Center for Natural Products Research (NCNPR), Universität von Mississippi, USA. Er beschäftigt sich mit der Qualitätsbeurteilung bzw. dem Fingerprint von pflanzlichen Produkten sowie der Entdeckung neuer Arzneimittel natürlichen Ursprungs. Er verwendet die HPTLC auch für das Screening und die Auswertung der antioxidativen Aktivität von verschiedenen Extrakten und einzelnen Komponenten anhand des DPPH-Tests auf freie Radikale [1]. Das gut aufgelöste HPTLC-Fingerprint in Kombination mit der Densitometrie ist eine einzigartige Charakterisierungsmöglichkeit.

Die Qualität des Fingerprints wird von Naturstoff-Chemikern, die komplexe Extrakte visuell auswerten, hoch geschätzt. Die HPTLC ist aufgrund ihrer Einfachheit, Genauigkeit/Präzision, hohen Geschwindigkeit und Kosteneffektivität von Vorteil und erfüllt alle Anforderungen, die eine zuverlässige, pharmazeutische Analytik stellt.

Einleitung

Seit seiner Einführung in den 90er Jahren ist Sildenafil zu einem der bedeutendsten Medikamente zur Behandlung der erektilen Dysfunktion geworden. Aufgrund seiner Relevanz wurden verschiedene analytische Methoden zur Quantifizierung von Sildenafil in pharmazeutischen Formulierungen und anderen Matrices erarbeitet. Über 60 % dieser Methoden basieren auf der HPLC, während TLC [2] nur für qualitative Zwecke eingesetzt wird. Wegen der Vorteile der HPTLC wurde eine neue Hochdurchsatz-Methode entwickelt [3], um Sildenafil in pharmazeutischen Produkten mittels HPTLC/UV zu quantifizieren.

Probenvorbereitung

Pharmazeutische Produkte wurden mit 10 mL dest. Wasser für 5 min im Ultraschallbad extrahiert. 85 mL Methanol wurden hinzugefügt, und die Lösung wurde für weitere 10 min im Ultraschall extrahiert, auf 100 mL mit Methanol aufgefüllt und 1:5 verdünnt.Bei pflanzlichen Produkten wurde der Inhalt von 3 Kapseln mit Methanol für 15 min im Ultraschallbad extrahiert. 1 mL des klaren Überstandes wurde mit Methanol auf 25 mL verdünnt.

Standardlösung

Sildenafilcitrat wurde in Methanol (1.052 mg/mL) gelöst, entsprechend 0.752 mg/mL Sildenafil (Stammlösung), und 1:5 verdünnt, um eine 150 µg/mL Sildenafil-Lösung zu erhalten.

Schicht

HPTLC-Platte Kieselgel 60 F_{254} (Merck) 20 × 10 cm

Probenauftragung

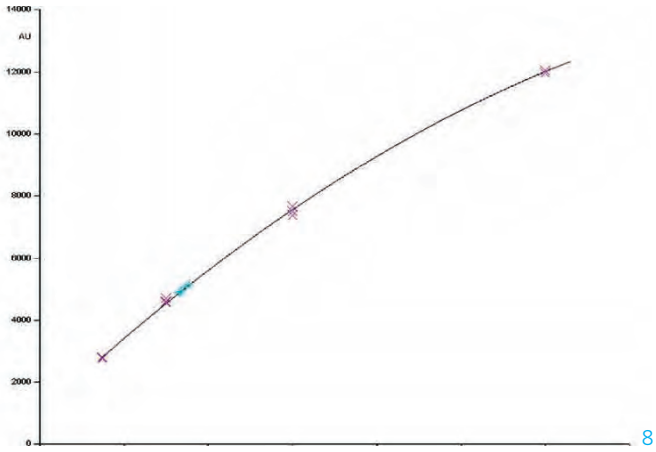
Bandweise mit dem Linomat, Auftragevolumen 4 µL für Proben und 1 bis 8 µL für Standardlösungen (150 bis 1200 ng/Band), Bandlänge 5 mm, Bahnabstand 9 mm, Abstand vom unteren Plattenrand 10 mm, Abstand vom seitlichen Plattenrand 12 mm

Chromatographie

In der Doppeltrogkammer mit Chloroform – Methanol – Diethylamin 9:1:0.1 nach 30 min Kammersättigung. Die Laufstrecke betrug 80 mm vom unteren Plattenrand. Danach wurden die Platten für 1 min im warmen Luftstrom getrocknet.

Densitometrie

TLC-Scanner 3 mit winCATS-Software, Absorptionsmessung bei 305 nm, polynome Kalibration über die Peakfläche



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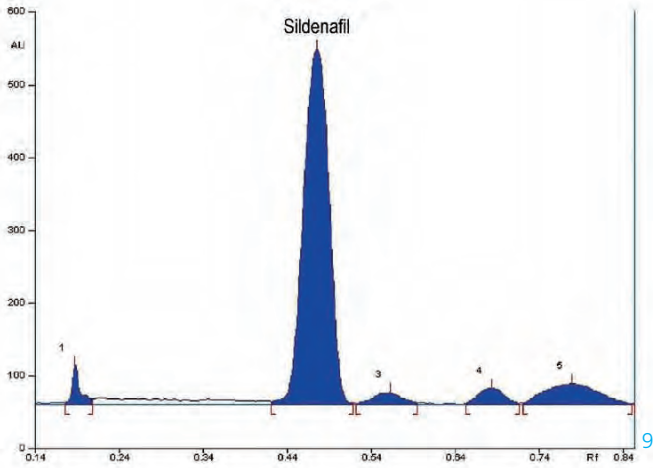
▲ *Sildenafil-Kalibrierkurve von 150 bis 1200 ng/Band*



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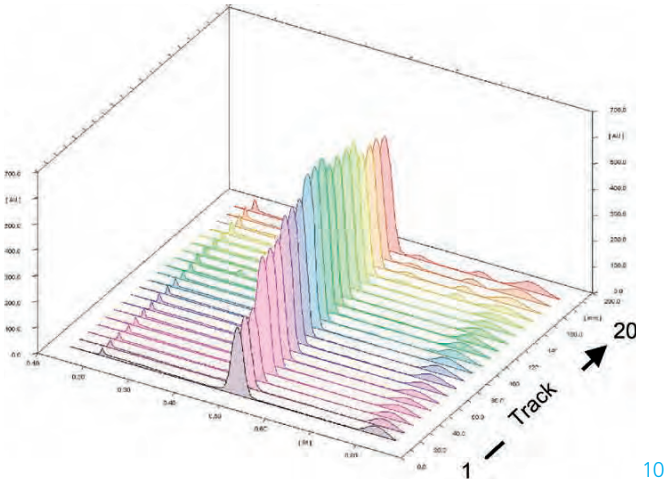
▲ *Chromatogramm, gemessen bei 305 nm, eines pflanzlichen Produktes, das einen gut abgegrenzten Sildenafil-Peak (272 ng/Band) zeigt*



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▲ *3-D Graphik von Kalibrierstandards (Bahn 1–12; 4 Konzentrationen je dreifach) sowie pharmazeutischen (Bahn 13–16) und pflanzlichen Produkten (Bahnen 17–20)*

Anmerkung: Diese Bahnanordnung erleichtert die Zuordnung, es ist aber empfehlenswert, Standard- und Probeauftragungen abwechselnd auf der Platte anzuordnen.

Ergebnisse und Diskussion

Die optimale Wellenlänge von 305 nm wurde durch die Aufnahme des Sildenafil-Spektrums bei hR_F 48 ± 1 bestimmt. Die 4-Punkte-Kalibration (je n = 3) von 150 bis 1200 ng/Band zeigte eine polynome Regression ($y = -0.003x^2 + 12.726x + 978.663$) mit $R > 0.9997$. Die Wiederholbarkeit wurde bestimmt, indem die gleiche Probe (n = 6) auf der gleichen Platte mehrmals aufgetragen wurde. Die relative Standardabweichung (*RSD*) war 0.7 bis 3.1 %, ermittelt an 4 verschiedenen Platten. Die Präzision über mehrere Tage (*RSD* <1 %) wurde berechnet, indem die gleiche Probe (n = 6) auf 4 verschiedenen Platten während 10 Tagen analysiert wurde. Die Methodengenauigkeit wurde durch die Wiederfindung von dotierten Proben ermittelt, wobei die mittlere Wiederfindung bei 98.2 % ± 3.3 % (*RSD* 3.4 %) für 3 verschiedene Konzentrationsniveaus lag. Der Sildenafilgehalt in vier pharmazeutischen Produkten lag zwischen 49.7 und 50.5 mg/Tablette und entspricht 99.4 bis 100.9 % des angegebenen Gehaltes. Das pflanzliche Produkt, das nur natürliche Inhaltsstoffe aufgelistet hatte, zeigte einen Sildenafilgehalt von 85 mg/Tablette, was nicht auf dem Etikett vermerkt war. Die Ergebnisse zeigen, dass die Planar-Chromatographie zur Qualitätskontrolle von Sildenafil-Arzneimitteln geeignet ist und auch dessen illegale Beimischung zu pflanzlichen Produkten erkennen kann.

Weitere Informationen erhalten Sie auf Anfrage vom Autor.

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

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

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

Dr. Dieter Jänchen zum 80. Geburtstag


11

Als am 17. Dezember 1958 Dr. Jänchen eine Firma unter dem Namen »CAMAG Chemie-Erzeugnisse und Adsorptionstechnik (Muttenz) AG« in das Handelsregister eintragen liess, ahnte wohl niemand, dass sich daraus einmal ein Unternehmen auf dem Gebiet der Planar-Chromatographie von Weltgeltung entwickeln würde.

Doch zunächst begann es mit Aluminumoxid für die Chromatographie, wofür sich bald industrielle Grossabnehmer interessierten. Der feinkörnige Anteil war der Einstieg in die Dünnschicht-Chromatographie, die in diesen Jahren ihren Siegeszug um die Welt anzutreten begann. Aus dieser Situation heraus erkannte der Jubilar, dass sich hier ein mikroanalytisches Trennverfahren entwickelte, das für einen Unternehmer eine Herausforderung darstellte und nach und nach ein erhebliches Entwicklungspotential offenbarte. Es ist sein besonderes Verdienst, aus dem geradezu simplen DC-Handling der ersten Jahre Schritt für Schritt ein Instrumentarium entwickelt zu haben, das heute die Bezeichnung »high tech« verdient. Dabei fand er in Deutschland wertvolle Unterstützung durch kreative Mitstreiter, so z. B. die Professoren Kaiser, Ebel und Jork sowie Dr. Burger. Im eigenen Hause baute er ein Team auf, das in Forschung/Entwicklung, Produktion und Vertrieb neue Wege ging.

Im Vertrieb war Dr. Jänchen am Anfang sein eigener erfolgreichster Mitarbeiter. Über Deutschland und die Schweiz hinaus erkannte er schnell die Bedeutung internationaler Märkte für die Entwicklung des Unternehmens. Als die instrumentelle Entwicklung der DC in ihre entscheidende Phase trat, war er mit besonderem Engagement weltweit erfolgreich. 1962 hatte er bereits in 18 Ländern Vertretungen selbst akquiriert, ein Jahr später waren es bereits doppelt so viele. Die Entwicklung von Tochterfirmen in Deutschland und USA hat er stets mit besonderer Fürsorge und Zuneigung begleitet, und das ist auch heute noch so.

Der von ihm ins Leben gerufene CAMAG Bibliography Service (CBS), der zweimal jährlich erscheint, ist weit mehr als eine Firmenzeitschrift im üblichen Sinne. Die Sammlung der Referate bedeutender DC/HPTLC-Arbeiten (CCBS) hat sich zu einer DC-Datenbank von internationalem Rang entwickelt.

Sein hundertprozentiges Engagement für die instrumentelle DC/HPTLC auf höchstem Qualitätsniveau machen ihn für die Mitarbeiter zu einem fortwährend richtungsweisenden Chef. Diejenigen, die das Glück haben, ihn näher zu kennen, wissen um seinen Humor und seine menschliche Wärme. Er stellt auch heute noch seine reichen Erfahrungen in den Dienst der Firma, wo er noch regelmässig anzutreffen ist. Seine Geradlinigkeit und Effizienz im Denken und Entscheiden sind nach wie vor geschätzt. Bis heute ist es für seine Mitarbeiter eine Selbstverständlichkeit, ihn mit »Chef« anzusprechen.

Ebenso wie in der täglichen Arbeit strebte Dr. Jänchen auch im Hobby-Bereich hohes Niveau an. 26 Jahre lang hat er als engagierter Fluglehrer mit Kunstflug- und Wolkenflug-Lehrberechtigung seine reichen Erfahrungen an Flugschüler weitergegeben. Er hat im Segelflug alle 3 Diamanten zur Gold-C erflogen und die Instrumenten-Flugberechtigung erlangt. Sein spektakulärstes Motorflugerlebnis war ein Transatlantik-Flug mit einer kleinen Einmotorigen. Freunde und Mitarbeiter denken heute noch gern an die Flüge mit ihm zurück.

Wir wünschen unserem Chef zum 80. Geburtstag am 06.06.2007 alles Gute, vor allem Gesundheit und auch weiterhin Schaffenskraft sowie noch viele schöne gemeinsame Jahre mit seiner Frau Brigitte!


12

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PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

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

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

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

Herzlichst Ihre

Gerda Morlock

Gerda Morlock
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Dear friends

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

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

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

Sincerely,

Gerda Morlock

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CAMAG

SEPTEMBER 2007 99

THE CBS CLASSIFICATION SYSTEM

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

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

1. Reviews and books

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

preparative TLC, review 1a

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

review 1b

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

comparison of methods, review 1a

2. Fundamentals, theory and general

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

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

relative retention 2b

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

HPTLC, qualitative identification 2d

3. General techniques

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

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

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

3d

4. Special techniques

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

HPTLC, TLC-MS online coupling 4e

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

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

review 4e

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

quality control, food analysis, comparison of methods 4d

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

qualitative identification HPTLC, TLC MS online coupling 4e

5. Hydrocarbons and halogen derivatives

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

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

comparison of methods 5b

7. Phenols

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

pharmaceutical research, qualitative identification, quantitative analysis densitometry 7

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

pharmaceutical research, quality control, quantitative analysis, densitometry 7

8. Substances containing heterocyclic oxygen

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

herbal, qualitative identification 8a

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

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

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

11. Organic acids and lipids

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

HPTLC, qualitative identification 11a

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

HPTLC, quantitative analysis 11c

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

qualitative identification 11c

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

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

densitometry, quantitative analysis, biological research 11c

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

clinical chemistry research, qualitative identification 11c

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

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

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

HPTLC, quantitative analysis, biological research 11c

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

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

pharmaceutical research, qualitative identification 11a

13. Steroids

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

qualitative identification, biological research 13d

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

13c

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

herbal, qualitative identification, preparative TLC 13e

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

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

pharmaceutical research, qualitative identification HPTLC densitometry 13a

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

qualitative identification 13a

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

qualitative identification 13d

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

qualitative identification 13d

14. Steroid glycosides, saponins and other terpenoid glycosides

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

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

herbal, quality control, quantitative analysis, densitometry 14

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

herbal, qualitative identification 14

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

food analysis, qualitative identification 14

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

herbal, HPTLC, quantitative analysis 14

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

herbal, preparative TLC qualitative identification 14

15. Terpenes and other volatile plant ingredients

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

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

pharmaceutical research, qualitative identification 15a

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

pharmaceutical research, herbal, preparative TLC 15a

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

herbal, qualitative identification 15a

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

traditional medicine, herbal, qualitative identification 15a

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

cosmetics, qualitative identification 15b

17. Amines, amides and related nitrogen compounds

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

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

pharmaceutical research, preparative TLC 17c

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

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

qualitative identification, HPTLC, biological research 17a

18. Amino acids and peptides, chemical structure of proteins

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

review, biological research 18

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

pharmaceutical research, qualitative identification 18

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

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

qualitative identification 18a

22. Alkaloids

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

herbal, qualitative identification, preparative TLC 22

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

herbal, quantitative analysis, densitometry 22

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

herbal, pharmaceutical research, qualitative identification 22

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

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

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

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

herbal, qualitative identification 22

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

pharmaceutical research, herbal, preparative TLC 22

23. Other substances containing heterocyclic nitrogen

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

HPTLC, quantitative analysis 23d

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

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

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

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

qualitative identification 23e, 3d

24. Organic sulfur compounds

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

pharmaceutical research, preparative TLC 24

27. Vitamins and various growth regulators

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

qualitative identification 27

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

HPTLC, quantitative analysis, densitometry, biological research 27

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

food analysis, quality control, qualitative identification, review 27

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

food analysis, quality control, qualitative identification 27

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

agricultural, qualitative identification, HPTLC 27

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

qualitative identification 27

28. Antibiotics, Mycotoxins

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

food analysis, qualitative identification 28a

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

qualitative identification, bioassay 28a

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

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

food analysis, quality control, qualitative identification 28a

29. Pesticides and other agrochemicals

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

agricultural, qualitative identification 29d

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

agricultural, quantitative analysis 29e

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

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

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

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

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

agricultural, environmental, HPTLC, qualitative identification 29d

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

agricultural, environmental, HPTLC, qualitative identification 29

30. Synthetic and natural dyes

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

pharmaceutical research, quantitative analysis 30a

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

food analysis, densitometry, qualitative identification 30

32. Pharmaceutical and biomedical applications

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

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

quality control, densitometry, quantitative analysis 32a

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

traditional medicine, HPTLC, quantitative analysis, densitometry 32a

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

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

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

HPTLC 32a

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

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

herbal, qualitative identification 32e

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

quality control, densitometry, quantitative analysis 32a

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

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

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

environmental, densitometry, quantitative analysis 32c

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

pharmaceutical research, qualitative identification 32a

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

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

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

quality control, quantitative analysis, densitometry 32a

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

quality control, qualitative identification 32e

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

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

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

pharmaceutical research 32a

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

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

traditional medicine, herbal, preparative TLC 32e

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

herbal, qualitative identification 32d

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

herbal, qualitative identification 32e

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

traditional medicine, herbal, qualitative identification 32e

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

herbal, preparative TLC 32e

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

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

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

qualitative identification 32e

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

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

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

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

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

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

pharmaceutical research, qualitative identification 32a

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

traditional medicine, densitometry 32e

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

herbal, qualitative identification 32e

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

herbal, qualitative identification 32e

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

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

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

quality control, densitometry quantitative analysis 32a

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

quality control, densitometry, quantitative analysis 32a

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

quality control, HPTLC, densitometry 32a

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

pharmaceutical research, qualitative identification 32c

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

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

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

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

quality control, qualitative identification, AMD 32 a

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

qualitative identification 32e

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

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

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

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

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

herbal, qualitative identification 32e

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

qualitative identification 32e

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

pharmaceutical research, HPTLC 32a

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

toxicology, qualitative identification 32c

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

toxicology, qualitative identification 32c

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

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

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

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

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

quality control, quantitative analysis, HPTLC 32a

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

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

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

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

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

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

traditional medicine, HPTLC 32a

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

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

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

traditional medicine herbal, qualitative identification, preparative TLC 32e

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

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

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

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

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

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

pharmaceutical research, organic synthesis 32a

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

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

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

quality control, qualitative identification 32a

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

herbal, qualitative identification 32e

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

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

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

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

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

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

HPTLC, quantitative analysis 32e

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

pharmaceutical research, qualitative identification 32a

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

herbal, food analysis, quantitative analysis, densitometry 32e

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

pharmaceutical research, qualitative identification 32a

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

herbal, traditional medicine, qualitative identification 32e

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

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

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

qualitative identification 32e

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

quality control, quantitative analysis, densitometry, HPTLC 32a

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

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

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

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

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

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

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

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

pharmaceutical research, densitometry, quantitative analysis 32a

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

herbal, traditional medicine, qualitative identification 32e

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

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

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

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

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

pharmaceutical research, HPTLC, densitometry, AMD 32b

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

pharmaceutical research, qualitative identification 32a

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

densitometry 32a

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

densitometry 32a

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

herbal, qualitative identification 32e

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

herbal, food analysis, qualitative identification 32e

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

toxicology, qualitative identification 32c

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

traditional medicine, herbal, toxicology, qualitative identification 32e

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

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

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

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

quality control, densitometry, quantitative analysis, HPTLC 32a

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

traditional medicine, herbal, qualitative identification 32e

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

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

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

pharmaceutical research, qualitative identification 32a

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

pharmaceutical research, qualitative identification, HPTLC 32a

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

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

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

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

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

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

preparative TLC, qualitative identification 32e

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

traditional medicine, herbal, preparative TLC 32e

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

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

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

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

33. Inorganic substances

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

qualitative identification 33a

37. Environmental analysis

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

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

38. Chiral separation

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

qualitative identification 38

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

City of Helsinki, Picture Bank/Photo Niko Soveri

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

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

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

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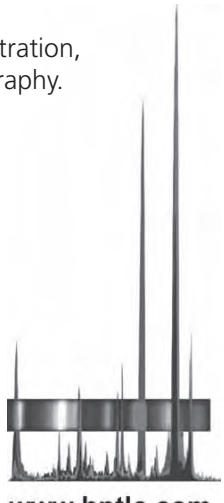

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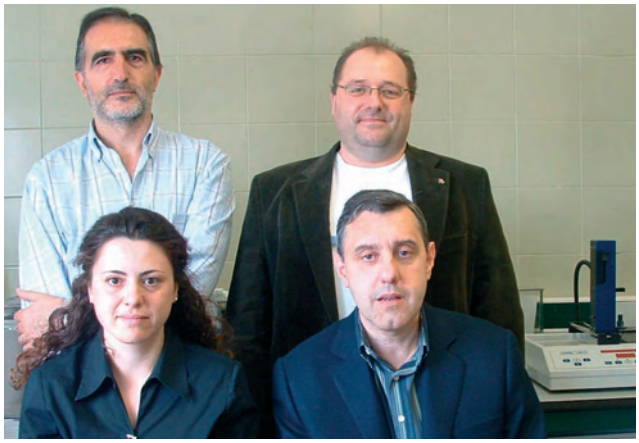
Eine universelle Detektion auf Basis der Fluoreszenzänderung



13

▲ *Dr. Elena Mateos, Dr. Vicente L. Cebolla*, Dr. Luis Membrado, Dr. Jesús Vela (von links vorne gegen den Uhrzeigersinn)*

Die Arbeitsgruppe »Trenn- und Detektionstechnologie« des Instituts für Kohlenstoff-Chemie (CSIC) in Zaragoza, Spanien, entwickelt seit vielen Jahren analytische Techniken, um sehr komplexe Mischungen zu charakterisieren, die meist aus Stufen der fossilen Brennstoffumstellung stammen. Diese bestehen aus strukturell unterschiedlichen hochmolekularen und/oder hochsiedenden Verbindungen.

Diese Mischungen beinhalten auch gesättigte Kohlenwasserstoffe, die weder UV-Licht absorbieren noch unter den üblichen analytischen Bedingungen fluoreszieren. Die Bestimmung der Kohlenwasserstoffart gehört in der Ölindustrie zur klassischen Analytik, die erhebliche technische Schwierigkeiten mit sich bringt. Deshalb beschäftigt sich die Arbeitsgruppe u.a. mit der Entwicklung von universellen Detektionssystemen, um Verbindungen ohne Chromophor wie gesättigte Kohlenwasserstoffe oder Lipide zu detektieren und zu quantifizieren.

Einleitung

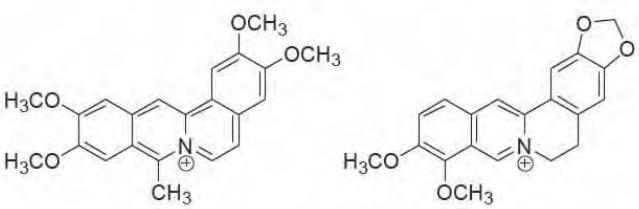
Die HPTLC wurde gewählt, da es eine gut geeignete Methode ist, um alle Bestandteile (auch die, die nicht eluieren) einer komplexen, stark kontaminierten Probe zu analysieren. Dies ist gegenüber den Säulentechniken vorteilhaft, bei denen hochmolekulare und/oder polare Kohlenwasserstoffe von der stationären Phase irreversibel adsorbiert werden können. Für die HPTLC wurde eine universelle Detektion entwickelt. Diese nutzt die Emissionsänderung von bestimmten, fluoreszierenden Substanzen, die durch nicht kovalente Wechselwirkung zum Analyten induziert wird [1, 2]. Deshalb ist es weder eine Derivatisierung, noch eine destruktive Methode und somit von besonderem Interesse für die Detektion von nicht fluoreszierenden Verbindungen.

Tatsächlich induziert jeder Analyt eine Änderung in den Fluoreszenzspektren von bestimmten Fluorophoren wie Berberin- und Coralynkationen. Ihre Emissionsintensitäten sind ausnahmslos von der Fluoreszenzsteigerung (positive Peaks) oder Fluoreszenzhinderung (negative Peaks) abhängig. Die HPTLC ermöglicht als lösungsmittelfreies Medium das Studium der intermolekularen, photophysikalischen Prozesse. Die Intensität und Lage der Emission ist, neben der Dielektrizitätskonstante des Mediums, vom Gleichgewicht zwischen unspezifischen und spezifischen Wechselwirkungen abhängig. So entsteht eine Mikroumgebung, die das Fluorophor isoliert und ein Abklingen der Fluoreszenz verhindert [3, 4]. Man unterscheidet zwischen unspezifischen Wechselwirkungen, die elektrostatischer Natur sind, i.e. Ionen-Dipol induzierte Interaktionen im Falle der untersuchten kationischen Fluorophore, und spezifischen Interaktionen gerichteter Art, wie z.B. Elektronendonator oder H-Brückenbildung.

Es wurde gezeigt, dass polare Verbindungen, z.B. Antibiotika und Aminosäuren, negative Peaks ergeben, die einer Fluoreszenzminderung entsprechen [1, 2]. Diese Fluoreszenzminderung kann rein spezifischen Interaktionen zugeschrieben werden, die die Geschwindigkeitskonstante des strahlungslosen Abklingens vergrössern und dabei eine Verringerung der Quantenausbeute bewirken.

Schicht

HPTLC-Platte Kieselgel 60 (Merck) 10 x 10 cm



▲ *Strukturformeln von Coralyn- (links) und Berberinkationen (rechts)*

Imprägnierung der Schicht

Kieselgelplatten werden je nach ihrer Verträglichkeit mit dem Fliessmittel prä- oder post-chromatographisch mit Berberin- oder Coralynlösungen imprägniert. Zum Beispiel erfolgt das Imprägnieren vor der Chromatographie durch Tauchen in eine methanolische Coralyn- (6 oder 12 mg/L) oder Berberinlösung (60 mg/L) mit anschliessender Trocknung bei 40°C über Nacht.

Auftragen

Bandweise mit dem DC-Probenautomat 4, 7 Bahnen pro Platte, Bandlänge 2 mm, Bahnabstand 10 mm, Abstand vom unteren Plattenrand 10 mm, Auftragevolumen 1–10 µL je nach Probenkonzentration, 1 Leerbahn pro Platte.

Chromatographie

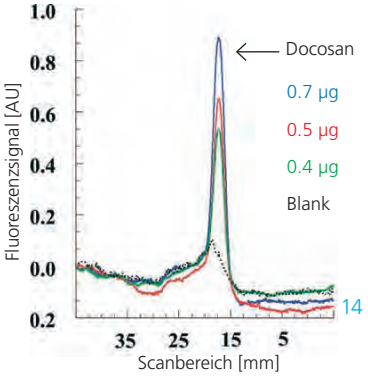
In der Horizontal-Entwicklungskammer: Gesättigte Kohlenwasserstoffe mit Dichlormethan, schwere Gasöl-Proben mit n-Hexan und Cholesterol mit Petrolether – Diethylether – Essigsäure 80:20:1. Die Laufzeit beträgt 5 min.

Densitometrie

Fluoreszenzmessung von Berberin und Coralyn bei der Anregungswellenlänge 365 bzw. 410 nm und Emissionswellenlänge > 450 nm.

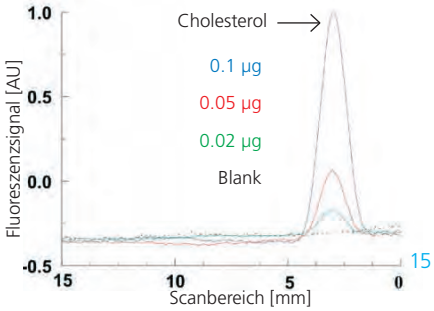
Ergebnisse und Diskussion

Auf mit Berberin- oder Coralynlösungen imprägnierten Kieselgelschichten zeigen Paraffinverbindungen wie das nicht-fluoreszierende Docosan (n-C22H46) eine erhöhte Fluoreszenzemission. Das Signal hängt im gegebenen System von der Alkan-Konzentration und -Kettenlänge ab [3–5].



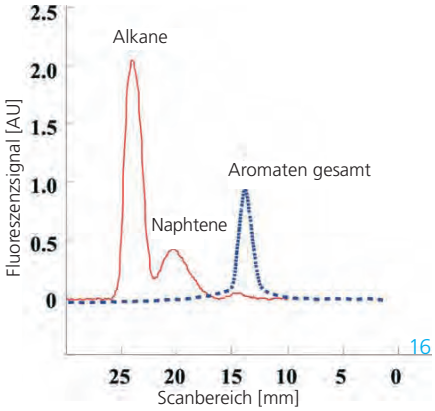
▲ *Fluoreszenzsignal von nicht fluoreszierendem Docosan auf einer mit Coralynlösung imprägnierten Schicht (6 mg/L)*

Dieses Phenomen ist nicht auf gesättigte Kohlenwasserstoffe beschränkt. Weitere, apolare Verbindungen wie Cholesterol geben je nach Konzentraton ebenso positive Peaks. In allen Fällen kann die Detektionsempfindlichkeit durch die Konzentration der Imprägnierlösung beeinflusst werden [5].



▲ *Fluoreszenzsignal von Cholesterol auf einer mit Coralynlösung imprägnierten Platte (6 mg/L)*

Diese Detektion wurde für petrochemische Proben eingesetzt. Der lineare Bereich für Alkane und Naphthene lag zwischen 0.05–1.5 µg bzw. 0.6–2.4 µg. Die erhaltenen Ergebnisse sind im Einklang mit Ergebnissen anderer Techniken der petrochemischen Industrie [5, 6].



▲ *Analyse einer schweren Gasöl-Probe von einer Raffinerie: Überlagerung der Absorptionsmessung bei UV 254 nm (blau) der gesamten Aromaten mit Aceton auf Kieselgel, und Fluoreszenzmessung (rot) von Alkanen und Naphthenen, mit n-Hexan auf einer mit Berberin imprägnierten Kieselgelschicht (60 mg/L)*

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

Dank an das Spanische Ministerium für Erziehung und Wissenschaft (Projekt CTQ2005-00227/PPQ) und die Universität Zaragoza (Projekt UZ2006-CIE-15) für die finanzielle Unterstützung.

Weitere Informationen sind vom Autor auf Anfrage erhältlich.

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Bioluminex™: Ein effektiver und zugleich leicht einsetzbarer Schnelltest

17

▲ *Das Team für neue Entwicklungen: Ms. Larissa Ikenouye, Ms Sarah Hickey, Dr. Sheryl Verbitski, Mr. Gerald Gourdin (von links nach rechts)*

Dr. Verbitski*, Leiterin für Neue Entwicklungen bei ChromaDex (www.chromadex.com) in Boulder, Colorado, und ihr Team setzen chromatographische Trenntechniken ein, um Nahrungsergänzungsmittel, pflanzliche Biomasse, Arzneimittel, Lebensmittel und Getränke zu analysieren. Ein Schwerpunkt ist die Weiterentwicklung des Bioluminex-Schnelltests (www.bioluminex.com), der kürzlich in den internationalen Markt eingeführt wurde.

Einleitung

ChromaDex hat einen Schnelltest zur Identifizierung von einzelnen Verbindungen mit biologischer Aktivität in komplexen Mischungen entwickelt. Zusätzlich kann diese Technologie zur Bioassay-basierten Fraktionierung eingesetzt werden. Für komplexe Mischungen wie Lebensmittel, Zusatzstoffe und Nahrungsergänzungsmittel ermitteln bisherige Bioaktivitätstests nur die Gesamtaktivität. In solchen Proben erfordert die Identifizierung aktiver Verbindungen eine mühsame Isolierung einzelner Verbindungen und deren anschliessende Bestimmung der biologischen Aktivität.

Alternativ ermöglicht der Bioluminex™-Schnelltest die direkte Kopplung von Biolumineszenz und HPTLC, wodurch einzigartig, schnell und effektiv die Toxizität der biologischen Aktivität in komplexen Gemischen überwacht werden kann. Nach der Chromatographie wird die HPTLC-Platte mit biolumineszenten Bakterien durch ein einfaches Tauchverfahren benetzt. Einzelne Verbindungen, die die Bakterienaktivität vermindern, werden selektiv als dunkle Zonen auf einem lumineszenten Hintergrund identifiziert. Ergebnisse werden innerhalb von Sekunden erhalten und mittels Digitalaufnahme dokumentiert. Dieser Schnelltest ist besonders geeignet, um Lebensmittel, Nahrungsergänzungsmittel und ähnliche komplexe Mischungen auf unübliche chemische und toxische Verfälschungen zu untersuchen. Er ist zudem zur Identifizierung von Verbindungen mit biologischer Aktivität sehr hilfreich. Diese Technologie ist als komplettes Testkit verfügbar und erlaubt eine kostengünstige Analyse vieler Proben.

Das biolumineszente, marine Bakterium *Vibrio fischeri* wurde für diese Anwendungen ausgewählt. Erreichen *V. fischeri*-Zellen eine kritische Zelldichte, sondern die atmungsaktiven Zellen die überschüssige freie Energie als detektierbares, blau-grünes Licht aus. Die beobachtete Biolumineszenz spiegelt die Stoffwechselaktivität der Zelle wieder und wird durch toxische Substanzen vermindert. Somit ist die Reduzierung der Lichtemission ein Mass für die Toxizität und kann in HPTLC-Chromatogrammen selektiv detektiert und quantifiziert werden.

Schicht

Merck oder Bioluminex™ HPTLC-Platten Kieselgel 60 F_{254} (10 × 10 cm), vorgewaschen mit Methanol (Chromatographie) und getrocknet bei 100 °C für 15 min.

Probenauftragung

Bandförmig mit dem DC-Probenautomat 4, Bandlänge 6 mm, Auftragevolumen 1–10 µL je nach Probe und Konzentration

Chromatographie

In der Doppeltrogkammer (10 × 10 cm, vorkonditioniert mit Filterpapier für 30 min) mit den unten genannten Fliessmitteln. Die Laufstrecke beträgt 60 mm vom unteren Plattenrand. Die entwickelte Platte wurde vor der Detektion für 2 h bei 40 °C getrocknet.

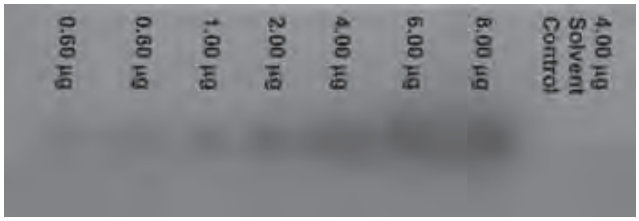
Bioluminex™-Detektion

Bioluminex-Bakterien werden 30 h in 200 mL komplexem Bioluminex™ Kulturmedium bei 120 Be-

wegungen pro min und 28 °C in einem Inkubator unter atmosphärischen Bedingungen vermehrt. Direkt vor der Detektion wird der Bioluminex™-Puffer zu der lumineszenten Bakterienlösung gegeben. Die entwickelte HPTLC-Platte wird dann in die gepufferte, lumineszente Bakteriensuspension mittels der Chromatogramm-Tauchvorrichtung III getaucht. Die Bildqualität wird durch das Abstreifen der überschüssigen Bakterienlösung mit einem handelsüblichen Scheibenwischer verbessert, und die Bilder werden innerhalb von 10 min aufgenommen.

Ergebnisse und Diskussion

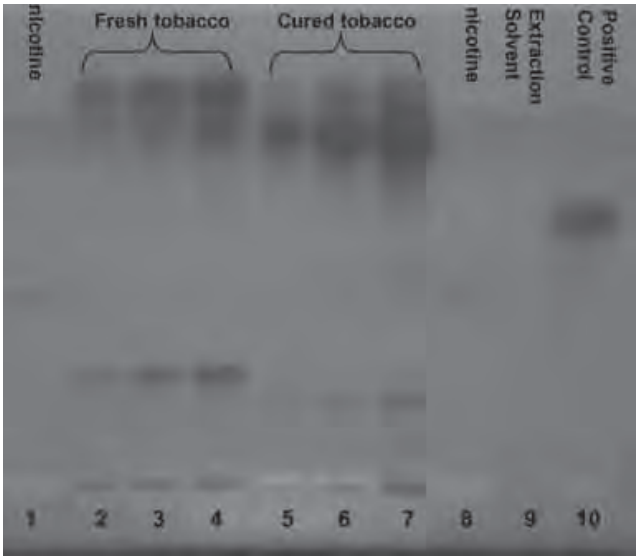
Der Bioluminex-Schnelltest kann leicht eingesetzt werden, um die Aktivität einer Substanz zu untersuchen. Nachfolgend wurden zum Beispiel unterschiedliche Mengen an Melamin auf die HPTLC-Platte aufgetragen. In den USA führte Melamin als unerlaubter Tierfutter-Zusatz zum Tod von Katzen und Hunden. Mit *Vibrio fischeri* wird die bio-aktive Wirkung von Melamin durch Biolumineszenzminderung angezeigt.



18

▲ *Detektion von Melamin*

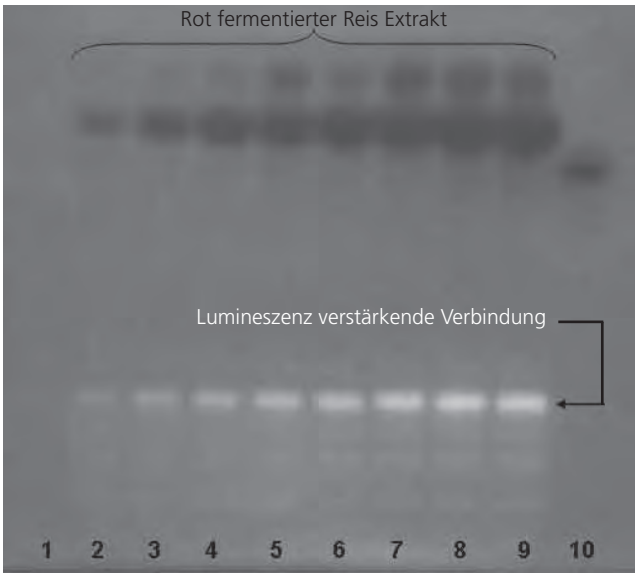
Als weiteres Beispiel dient die Untersuchung von frischem und getrocknetem Tabak. Nach der Chromatographie mit Chloroform – Methanol – Ammoniumhydroxid 9:1:0.05 werden die Proben mit dem Bioluminex-Schnelltest detektiert. Die zwei verschiedenen Extrakte zeigen einen einzigartigen chemischen und biologischen Fingerprint. Der charakteristische Fingerprint der Bioluminex-Detektion kann zur Unterscheidung von frischem und behandeltem Tabak genutzt werden.



19

▲ *Detektion von frischen gegenüber getrockneten Tabakextrakten*

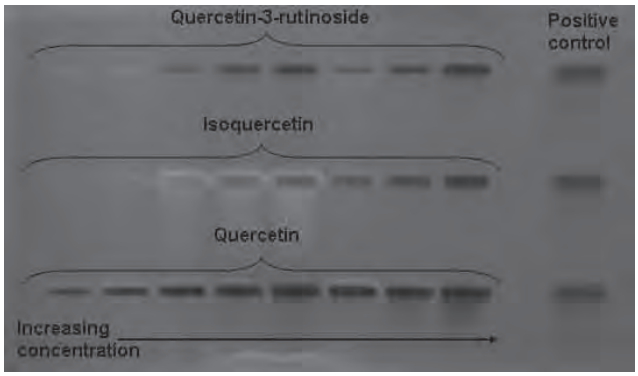
Komplexe Gemische, wie rot fermentierter Reisextrakt, dessen Extrakt Cholesterol-senkend und bakteriostatisch wirkt und in Asien auch als Heilmittel bei Infektionen eingesetzt wird, können nach Substanzen mit interessanter biologischer Aktivität untersucht werden. Nachfolgend wurde rot fermentierter Reis mit Methanol extrahiert, mit Chloroform – Ethylformiat – Ameisensäure – Methanol 5:5:2:2 chromatographiert und mit dem Bioluminex-Schnelltest detektiert. Zwei Verbindungen zeigen bei hR_f 82 und 91 eine Verminderung der *V. fischeri*-Biolumineszenz, während dagegen eine Verbindung bei hR_f 25 die Biolumineszenz verstärkt.



20

▲ *Detektion von rot fermentiertem Reisextrakt*

Der Bioluminex-Schnelltest ist ein einfaches Mittel, um auch Struktur-Wirkungsbeziehungen (SAR) zu untersuchen. Zum Beispiel wird nachfolgend die SAR-Analyse der drei strukturell ähnlichen Verbindungen Quercetin, Isoquercitin und Quercetin-3-rutinosid aufgezeigt (Auftragen unterschiedlicher Volumina, ohne Chromatographie).



21

▲ *SAR-Analyse von strukturell ähnlichen Verbindungen*

Die Anwendbarkeit dieser neuartigen Bioaktivitäts-basierten Detektion wurde bisher für Proben im Lebensmittelbereich, in der forensischen Analytik und Umweltanalytik gezeigt. Zum Beispiel detektiert *V. fischeri* Ochratoxin in Dosenmais, Aflatoxin B1 in Honig, Digoxin in Milch, Benzopyren in Selleriesamen, Capsaicin in Cayenne-Pfeffer, Strychnin oder Monofluoressigsäure in verschiedenen Getränken, Domoinsäure in Limonade und Patulin in Apfelsaft.

Dabei ist die Entdeckung und Strukturaufklärung von neuen bioaktiven Verbindungen, die so in einigen Proben detektierbar sind, höchst interessant. Ein weiterer hilfreicher Schritt ist daher der Einsatz der Massenspektrometrie, um weitere Informationen über diese bioaktiven Verbindungen zu bekommen.

Weitere Informationen sind vom Autor auf Anfrage erhältlich.

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

22

CAMAG BioLuminizer

Die Trennleistung der Planar-Chromatographie verbunden mit der Biolumineszenz-Detektion ermöglicht die Identifizierung von biologisch aktiven Substanzen. Alle Verbindungen, die einen entsprechenden Effekt zeigen, werden in komplexen Gemischen im Pikomol-Bereich angezeigt, so in Abwasser oder forensischen Proben. Die umfassende Wirkungsdetektion von bioaktiven Substanzen erfasst z.B. auch unbekannte Metaboliten und unterscheidet sich damit von der herkömmlichen gezielten Suche mittels Standardsubstanzen.

BioLuminizer ist ein kompaktes, benutzerfreundliches Detektionssystem, das aussagekräftige Bilder von hoher Qualität und Auflösung bei kurzen Belichtungszeiten liefert.

Unter der Bezeichnung BioLuminex Standardkit (Nr. 022.9765) liefert CAMAG einen kompletten Satz der benötigten Verbrauchsmaterialien.

Bestimmung von Acrylamid in Trinkwasser

23

▲ *Alexander Alpmann, Doktorand im Arbeitskreis, betreut von Dr. G. Morlock*

Der Arbeitskreis von Professor Dr. Wolfgang Schwack*, Institut für Lebensmittelchemie, Universität Hohenheim, Stuttgart (siehe CBS 93) forscht u.a. auf dem Gebiet der Planar-Chromatographie. Gerade die Flexibilität der Methode beeindruckt immer wieder, wenn es gilt, schwierige Fragestellungen auf einfache Art zu lösen.

Einleitung

Polyacrylamid wird sowohl in der Papier-, Kosmetik-, Textil- und Bauindustrie eingesetzt als auch als Flockungshilfsmittel in der Trinkwasseraufbereitung. Dabei kann das stark wasserlösliche Monomer Acrylamid (AA) in das Grund- bzw. Trinkwasser übergehen. Aufgrund seiner Kanzerogenität wurde in der EU-Trinkwasser-Richtlinie 98/83/EC die maximal zulässige AA-Konzentration auf 0.1 µg/L festgesetzt.

Die Verwendung der HPLC-MS/MS nach DIN 38413-6 ist jedoch durch den hohen apparativen Aufwand für kleinere Labors unattraktiv. Ausserdem kann – aufgrund des geringen Molekulargewichtes – das protonierte Molekül von AA (72 Da) durch Matrixfragmente überlagert werden und in Ultraspuren (ng/L) im UV-Bereich nicht detektiert werden. Eine kostengünstige und selektive Alternative für routinemässige Untersuchungen ist die Derivatisierung von AA mit einem Fluorophor. Diese erfolgt prä-chromatographisch in der Auftragezone der HPTLC-Platte.

Probenvorbereitung

Wasserproben (500 mL) werden mit 250 µL N,N-Dimethylacrylamid (1 ng/µL in Methanol) als internem Standard (IS) dotiert, mittels Festphasenextraktion an sphärischer Aktivkohle extrahiert und 5 mal mit je 2 mL Methanol – Acetonitril 1:1 eluiert. Das Eluat wird am Rotationsverdampfer unter Stickstoffstrom auf ca. 1 mL eingeengt.

Standard- und Derivatisierungslösung

Entionisiertes Wasser (500 mL) wird mit 50–200 µL AA-Lösung (1 ng/µL in Methanol) und je 250 µL IS dotiert und analog der Wasserproben extrahiert. Als Blindprobe dient undotiertes Leitungswasser.

Das Derivatisierungsreagenz Dansulfinsäure wird – da es nicht kommerziell erhältlich ist – nach einer einfachen Methode von Scully et al. [1] synthetisiert und eingesetzt (3.2 µg/µL in Methanol).

Schicht

HPTLC-Platte Kieselgel 60 (Merck) 10×10 cm

Probenauftragung

Mit dem DC-Probenautomat 4 mit beheizbarer Sprühdüse (40 °C) als 6×3 mm Fläche, 8 Bahnen, Auftragevolumen 100 µL für Proben und Standards, Bahnabstand 10 mm, seitlicher und unterer Randabstand 12 bzw. 8 mm, Auftragegeschwindigkeit 350 nL/s, Übersprühen der Startzonen mit je 20 µL Derivatisierungslösung

Derivatisierung

Auf dem DC-Plattenheizer für 1 h bei 120 °C

Chromatographie

Die Startzonen werden mit Methanol fokussiert und in der Doppeltrogkammer mit Ethylacetat entwickelt (Laufstrecke 70 mm vom unteren Plattenrand, Laufzeit 15 min). Nach 2 min Trocknen im Warmluftstrom wird die Platte zur Fluoreszenzverstärkung in Polypropylenglykol-Lösung (25% in n-Hexan) mit der Chromatogramm-Tauchvorrichtung III getaucht (Tauchgeschwindigkeit 5 cm/s, Eintauchzeit 1 s).

Dokumentation

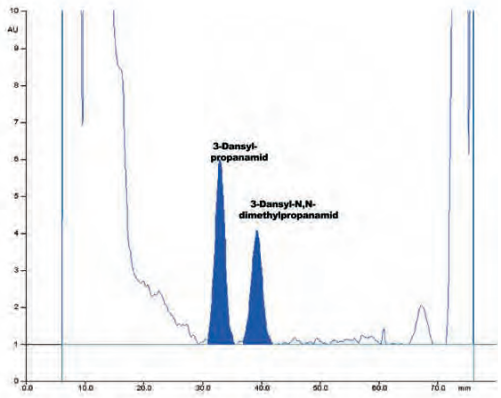
Mit DigiStore 2-System bei 366/>400 nm

Densitometrische Auswertung

TLC-Scanner 3 mit winCATS-Software, Fluoreszenzmessung bei UV 366/>400, lineare Kalibration über die Peakfläche

Ergebnisse und Diskussion

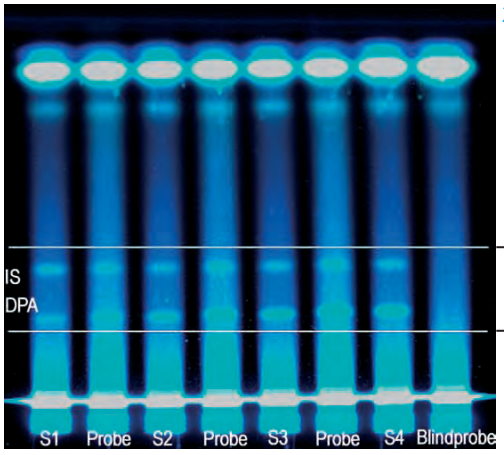
AA wird nach Derivatisierung mit Dansulfinsäure zu 3-Dansylpropanamid (DPA) ohne störende Matrix selektiv detektiert. Die Güte der Probenvorbereitung wird durch die Korrektur mit dem IS (derivatisiert zu 3-Dansyl-N,N-Dimethylpropanamid) sichergestellt.



24

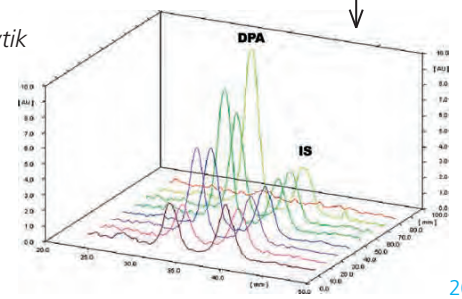
▲ *Fluoreszenzscan einer dotierten Wasserprobe (0.2 µg/L)*

Die mittlere Präzision unter Wiederholbedingungen betrug 4.8 % (*RSD*, *n* = 3, über 3 Konzentrationsstufen) und die mittlere Wiederfindung über 3 Konzentrationsbereiche 96 % (korrigiert über den IS).



25

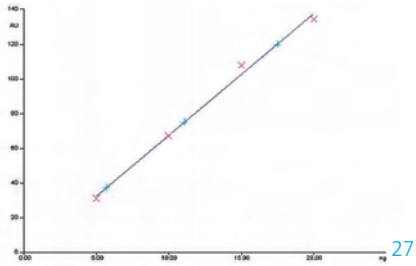
▲ *Plattenfoto der Ultra-Spurenanalytik von AA (als DPA) in Trinkwasser*



26

Fluoreszenzscan im markierten Bereich von Standards (0.1–0.4 µg/L, S1-S4), dotierten Blindproben (0.1–0.3 µg/L) und Blindprobe ▲

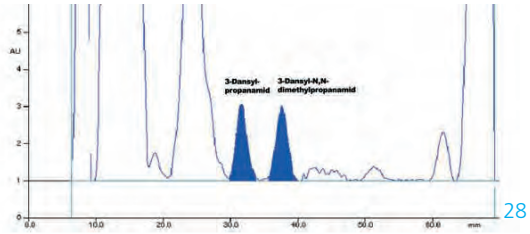
Die Regression ist im Arbeitsbereich von 0.1 bis 0.4 µg/L linear mit einer relativen Standardabweichung von ± 5.2 % (r = 0.9957).



27

▲ *Lineare Kalibration von DPA (5–20 ng/Zone bzw. 0.1–0.4 µg/L)*

Die berechnete Bestimmungsgrenze der Methode liegt bei 0.08 µg/L AA in Trinkwasser und ermöglicht die verlässliche Überwachung des festgelegten Grenzwertes von 0.1 µg/L.



28

▲ *Densitogramm einer Blindprobe dotiert unterhalb der Bestimmungsgrenze bei 0.05 µg/L*

Ein Methodenvergleich mit der HPLC-MS/MS zeigte vergleichbare Ergebnisse für die Ultra-Spurenanalyse von AA in Grundwasser und belegt die Leistungsfähigkeit des neuen Verfahrens:

Methodenvergleich	HPLC-MS/MS AA [µg/L]	HPTLC-FLD AA [µg/L]
Grundwasserprobe 1	<0.05	<0.05
Grundwasserprobe 2	0.07	0.09
Grundwasserprobe 3	0.18	0.24
Grundwasserprobe 4	0.59	0.60

Bei Relevanz können zudem Massenspektren durch online Extraktion (ca. 1 min/Zone) aufgenommen werden. Das durch die Derivatisierung protonierte Molekül höherer Masse von *m/z* 307 ist dabei von grossem Vorteil, da es störungsfreier als *m/z* 72 und mit einem einfachen MS (anstelle der MS/MS) detektiert werden kann.

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

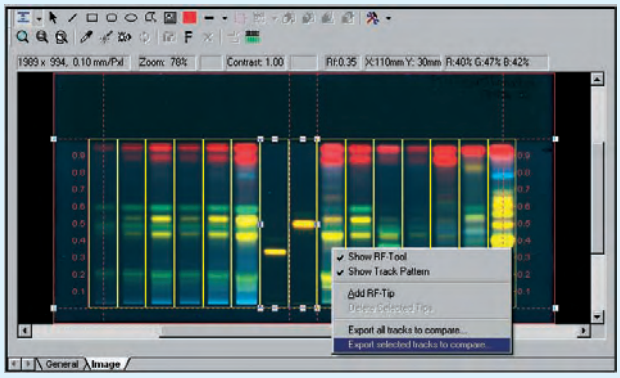
Dank der Landesstiftung Baden-Württemberg (Projekt-Nr. P-LS-E2/25) und dem Betriebs- und Forschungslaboratorium des Zweckverbandes Landeswasserversorgung, Standort Langenau, für die HPLC-MS/MS-Messungen.

*www.ilc.uni-hohenheim.de

Visueller Vergleich mehrerer Proben – ganz einfach!

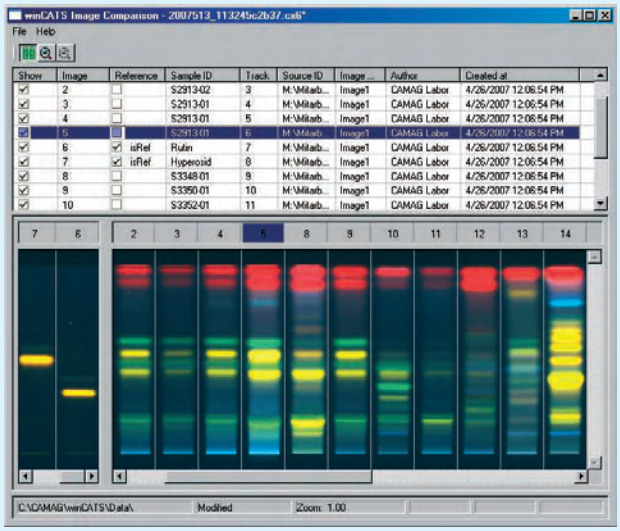
Die neue Software-Option »Bildvergleich« nutzt einen der grossen Vorteile und Stärken der Planar-Chromatographie: die Möglichkeit sich von dem Chromatgramm ein »Bild« zu machen.

Mit der Option »Bildvergleich« – sie läuft ab winCATS 1.4.3 – lassen sich Bahnen von verschiedenen DC/HPTLC-Platten direkt auf dem Bildschirm miteinander vergleichen:



29

▲ *Alle verfügbaren Bahnen der Platte werden automatisch markiert und können für den Transfer zur Option »Bildvergleich« ausgewählt werden.*



30

▲ *Bildvergleich von Passiflora incarnata (Passionsblume) mit weiteren Passiflora Spezies: Standard Rutin (Bahn 7) und Hyperosid (8), Passiflora incarnata S2913 mit unterschiedlichen Auftragevolumina (0–5), Passiflora incarnata S3350 (9), Passiflora incarnata S3352 (10).*

- Gleichzeitige Darstellung von Bahnen individueller Proben oder Probengruppen sowie direkter Vergleich von Referenzbahnen unterschiedlicher Platten
- Automatische Übertragung von Bahn-Informationen, wie Position, Breite, Länge, ID-Daten etc., zur Softwareoption »Bildvergleich«
- Unterscheidung zwischen Referenz- und Probebahnen
- Ausdruck des zusammengestellten Vergleichsbildes inklusive zusätzlicher Informationen
- Speicherung in Archiven mit Vergleichsdaten verschiedener Chargen oder Abfüllungen etc.
- Rückverfolgung aller generierten Daten bis zur ursprünglichen Analyse.

CAMAG

Weltweit führend in der Planar-Chromatographie

CBS 99 D 996.1310-2