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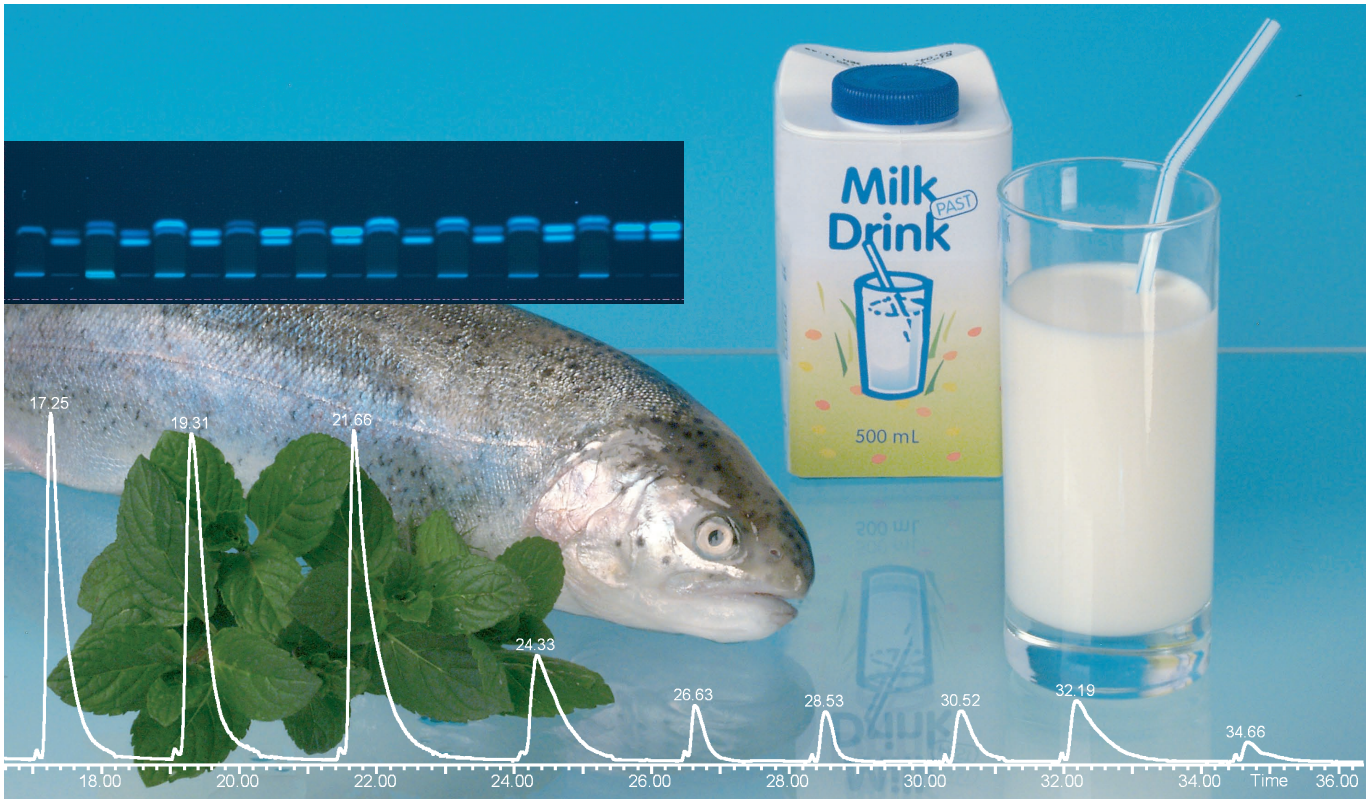
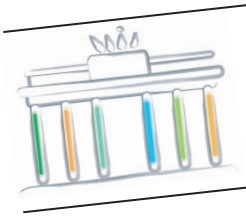


Effiziente Methoden zur Überwachung von Lebensmitteln



INTERNATIONAL SYMPOSIUM
ON HIGH-PERFORMANCE
THIN-LAYER CHROMATOGRAPHY

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Bestimmung von Amitrol in Wasser mittels AMD


1
▲ *Bayer Chemiepark (Foto: Bayer Industry Services)*


2
▲ *Herr Kinast bei der Durchführung der Amitrol Bestimmung*

Seit Mitte der 80er Jahre existiert das einst zum Zweck der Bearbeitung analytischer Fragestellungen aus den verschiedenen Forschungsbereichen gegründete AMD-Labor der Bayer AG. Heute liefert dieses Labor unter Leitung von Herrn Dr. Plaß* einen wichtigen Ergänzungsbeitrag zu den vorwiegend eingesetzten Trenntechniken GC und HPLC im analytischen Dienstleistungsspektrum der Bayer Industry Services GmbH & Co. OHG für Auftraggeber unterschiedlichster Herkunft. Spezialisiert hat sich das Labor auf die Analyse von Pflanzenschutzwirkstoffen und deren Metaboliten in diversen Matrizes, aber auch Probleme aus anderen Bereichen konnten oft erfolgreich bearbeitet werden. Durch den Mehrwellenlängen-Scan der CATS- bzw. winCATS-Software konnte im Laufe der Jahre eine umfangreiche Chromatographie-Datenbank aufgebaut werden. Diese ermöglicht beim Auftreten unbekannter Signale im Rahmen qualitativer Analysen aus einer Auswahl von über 1500 Substanzen eine Strukturzuordnung. Im Rahmen quantitativer Analysen kann umgekehrt auf An- bzw. Abwesenheit bestimmter Stoffe in einer Probe geprüft und darüber hinaus mit Hilfe eines umfangreichen Vorrates an Referenzsubstanzen eine Quantifizierung der gewünschten Signale durchgeführt werden. Zur zusätzlichen Charakterisierung der planar-chromatographisch getrennten Probenbestandteile steht dem Labor eine Reihe von biochemischen Sensoren zur Verfügung.

Einleitung

Amitrol (3-Amino-1H-1,2,4-triazol) ist ein systemisch wirkendes Herbizid aus der Klasse der Triazole, das zur Unkrautbekämpfung auf Bahngleisen, Wegen, Industrieflächen, sowie im Umfeld von Obstanbau und Ziergärten, des weiteren in Marschlandschaften und Bewässerungsgräben eingesetzt wird. Es wird von der Pflanze über Blätter und Wurzeln aufgenommen und besitzt eine sehr gute Wasserlöslichkeit. Die Adsorption und der biologische Abbau erfolgen bei huminstoffreichen Böden sehr rasch, während die Lebensdauer bei Abwesenheit organischer Substanz, z. B. in leichten, sandigen Böden, zunimmt. Obwohl Amitrol bei richtiger Anwendung keine

Gefahr für aquatische und terrestrische Lebewesen darstellt und auch nicht zur Bioakkumulation tendiert, besteht für Überwachungszwecke generell die Anforderung, Pflanzenschutzwirkstoffe mittels geeigneter spurenanalytischer Methoden in Wasserproben erfassen zu können.

Aufgrund der hohen Polarität von Amitrol, kombiniert mit einem niedrigen molaren UV-Absorptionskoeffizienten, ist die GC- und HPLC-Analytik erschwert und auch die für einige Wirkstoffgruppen beschriebene, auf der AMD-Technik basierende DIN 38407-11 für die Erfassung dieser Substanz in Wasser nicht geeignet. Zudem erschwert das geringe Molekulargewicht von Amitrol die massenselektive Detektion mittels HPLC-MS.

Im folgenden wird die Analytik von Amitrol in Wasserproben mittels AMD und selektiver post-chromatographischer Detektion mit dem Bratton-Marshall-Reagenz beschrieben, was für die Überwachung oder Überprüfung von Oberflächen- und Grundwasser ein interessantes Einsatzgebiet darstellt. Durch den hohen Automatisierungsgrad erweist sich die Methode als durchaus wettbewerbsfähig – die zu berechnende Analysenzeit pro Probe beträgt nur ca. 10 min.

Probenvorbereitung

Für die Abwasseranalytik im Bereich von 20 mg/L bis 0,01 mg/L ist keine weitere Probenvorbereitung zur Bestimmung des Amitrols notwendig. Die wässrige Probe kann direkt aufgetragen werden.

Wasserproben mit Konzentrationen unterhalb von 0,01 mg/L bis hin zum Grenzwert von 0,1 µg/L gemäss TWO lassen sich beispielsweise nach Ansäuern der Probe mit verd. Schwefelsäure, Eindampfen bis zur Trockene und anschliessendem Aufnehmen in ammoniakalischem Methanol nachweisen. Bei hohen Salzkonzentrationen empfiehlt sich die Anreicherung um den Faktor 1000 an einem Kationenaustauscher vom Typ Dowex 50 WX4-100 (Sigma-Aldrich).

Schicht

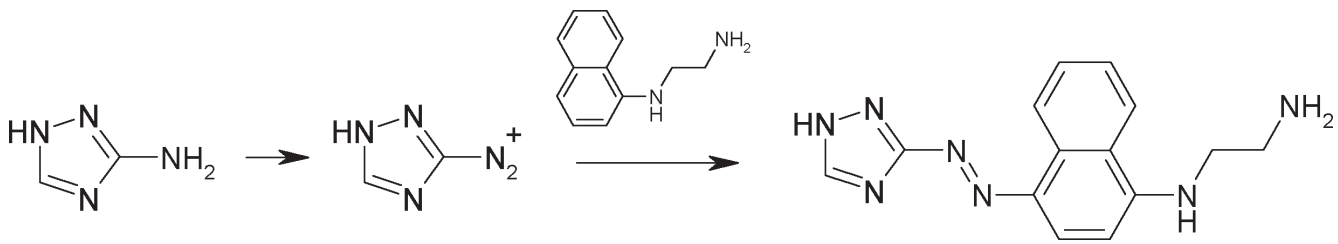
HPTLC-Platten LiChrospher Si 60 F_{254s} Merck, 20 x 10 cm, vorgewaschen mit 0,1 % Ameisensäure in Methanol (Eintauchen über 8 h) und über Nacht bei 1 hPa im Exsikkator getrocknet

Probenauftragung

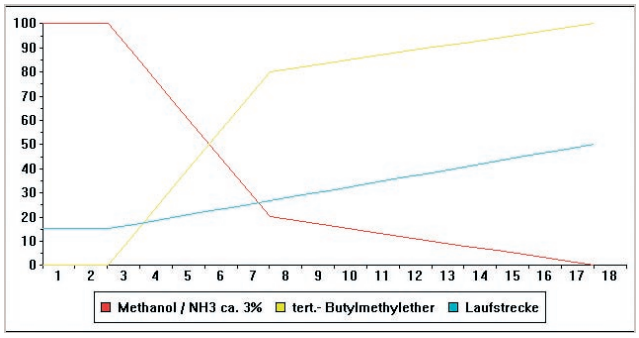
Bandförmig mit Linomat, Bandlänge 7 mm, seitlicher Randabstand 12 mm, unterer Randabstand 7 mm, Bahnabstand 10 mm, Auftragevolumina 2, 5, und 10 µL der 1-ppm-Kalibrierlösung (Auftragegeschwindigkeit 4 s/µL) und bis zu 100 µL der wässrigen Probelösungen (als 7 x 5 mm Fläche, Auftragegeschwindigkeit 10–15 s/µL).

Chromatographie

Im AMD 2-System mit einem 18-stufigen Gradienten von ammoniakalischem Methanol (hergestellt durch Einleiten von Ammoniak-Gas in Methanol unter Eiskühlung) übergehend zu tert.-Butylmethylether (max. Laufstrecke 50 mm). Hierbei dient der lipophile Gradiententeil lediglich zur Abtrennung von Störkomponenten (z. B. Phenylharnstoffe und Metaboliten). Der Lösungsmittelverbrauch liegt bei 140 mL pro Gradient. Die Gradientendauer beträgt insgesamt 128 min, davon sind 54 min Trocknungzeit.



▲ *Struktur bzw. Derivatisierung von Amitrol mit dem Bratton-Marshall-Reagenz*



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▲ *AMD2-Gradient (nach 3 Stufen zur Fokussierung) über 15 Stufen von Methanol/NH_3 nach tert.-Butylmethylether*

Lauf Nr.	Kond. J/N	Methanol %	Dichlormeth %	n-Hexan %	tert.- %	LM 5 %	Strecke mm	Trckzeit min	Laufzeit min	Enddrck mbar	Temp. °C
1	N	100					15.0	3.0	0.5	0	22.1
2	N	100					15.1	3.0	0.5	0	21.9
3	N	100					15.0	3.0	0.5	0	21.6
4	N	84			16		17.3	3.0	0.7	0	21.4
5	N	68			32		19.7	3.0	0.9	0	21.2
6	N	52			48		22.1	3.0	1.2	0	21.1
7	N	36			64		24.3	3.0	1.5	0	21.0
8	N	20			80		26.7	3.0	1.7	0	21.0
9	N	18			82		29.0	3.0	2.1	0	21.0
10	N	16			84		31.3	3.0	2.4	0	21.1
11	N	14			86		33.7	3.0	2.9	0	21.2
12	N	12			88		36.0	3.0	3.3	0	21.3
13	N	10			90		38.3	3.0	3.8	0	21.5
14	N	8			92		40.7	3.0	4.3	0	21.7
15	N	6			94		43.0	3.0	4.9	0	21.8
16	N	4			96		45.3	3.0	5.5	0	22.0
17	N	2			98		47.7	3.0	6.1	0	22.1
18	N				100		50.0	10.0	6.8	0	22.3

4

▲ *Protokoll der erfolgten 18 AMD2-Entwicklungsschritte*

Postchromatographische Derivatisierung

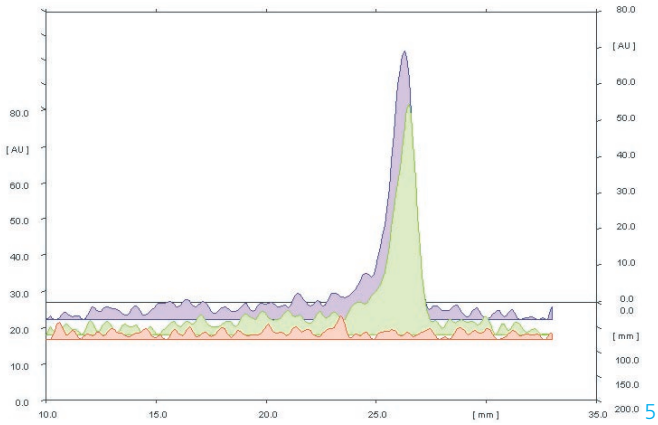
Die Platte wird zuerst salzsauer bedampft, anschliessend mit NOx begast. Die auf diese Weise vorbehandelte Platte wird mittels Tauchvorrichtung für 3 s in eine Lösung aus 0,2 g Bratton-Marshall Reagenz (N-[1-Naphthyl]ethylendiamindihydrochlorid) in 100 mL Methanol/Dichlormethan (1:4, v/v) getaucht. Hierbei entstehen aus primären aromatischen Aminen farbige Azoverbindungen, die als rotviolette Zonen auf farblosem Untergrund sichtbar sind. An dieser Stelle kann bereits eine visuelle Auswertung stattfinden. Für die densitometrische Auswertung empfiehlt sich zur Stabilisierung des Azofarbstoffes ein anschliessendes Bedampfen mit Ammoniak.

Densitometrische Auswertung

TLC-Scanner 3 mit winCATS Software, Absorptionsmessung im sichtbarem Bereich bei 490 nm, Michaelis-Menten 1-Regression über die Peakhöhen.

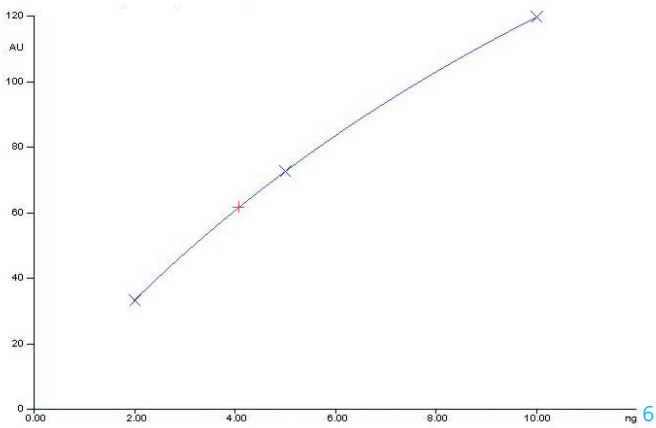
Ergebnisse und Diskussion

Nach dem oben beschriebenen Verfahren lässt sich Amitrol bis zu einer Absolutmenge von 1 ng auf der HPTLC-Platte nachweisen. Dieser Nachweis ist so selektiv, dass man bei einem Positivergebnis auf eine übliche Strukturabsicherung, z. B. mittels MS, verzichten kann. Die laborinterne Validierung belegte einen linearen Zusammenhang im Absolutmengenbereich zwischen 1 ng und 10 ng Amitrol. Die Auswertung der Präzision ergab eine relative Standardabweichung (VK) von ± 1,7 %. Bei Zugrundelegen einer nichtlinearen Kalibrierfunktion erweist sich der Arbeitsbereich nach oben als wesentlich grösser.



5

▲ *Übereinandergelegte Analogkurven (λ = 490 nm) einer Wasser-Blindprobe (rote Analogkurve), einer mit 4 ng Amitrol dotierten Wasserprobe (grüne Analogkurve) und dem 5 ng Amitrol-Standard (lila Analogkurve)*



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▲ *Michaelis-Menten 1-Regression ($y = 18{,}497\ x^2 + 340{,}896\ x$) von Amitrol über die Peakhöhen mittels Absorptionsmessung bei 490 nm, relative Standardabweichung von ± 0,01 %*

Für die Bearbeitung einer Einzelprobe erscheint die HPTLC-Methode aufgrund ihrer relativ grossen Bruttoanalysenzeit (AMD 2-Trennung incl. Probenauftragung) und den durch die Personalbindung erzeugten Kosten auf den ersten Blick wenig komfortabel. Jedoch durch die Möglichkeit, gleichzeitig bis zu 15 Wasserproben auf einer Platte analysieren zu können, beträgt die zu berechnende Analysenzeit pro Probe nur noch ca. 10 min. Daran hat die Automatisierung der Probenauftragung (Linomat oder ATS4) und der Chromatographie (AMD 2) einen massgeblichen Anteil. Ein weiteres wichtiges Instrument zur Minimierung der Analysekosten ist die automatische Auswertung der Signale mittels der winCATS Software. Die oben beschriebene Derivatisierung ist standardisiert und robust, so dass sie auch von wenig geübtem Personal durchgeführt werden kann.

Die Betrachtung der oben beschriebenen Amitrol-Analytik per HPTLC zeigt, dass sich diese Technik insbesondere bei hohem Automatisierungsgrad als durchaus wettbewerbsfähig erweist, und auch in Zukunft durch das Erarbeiten innovativer, kundenspezifischer Lösungsansätze für weitere analytische Fragestellungen Standardleistungen zu etablieren vermag.

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

* Dr. Ernst Plaß, Bayer Industry Services GmbH & Co. OHG, Bayer Chemiepark, Gebäude C 601, 41538 Dormagen, ernst.plass.ep@bayerindustry.de

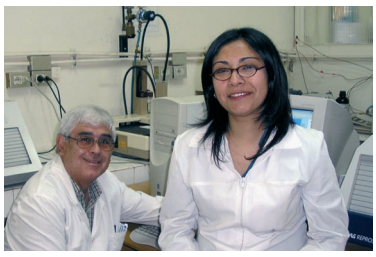
7

CAMAG AMD 2 System
(Automatisierte Mehrfachentwicklung)

Das AMD-Labor der Bayer Industry Services setzt 6 AMD-Systeme parallel ein, um in Verbindung mit dem Mehrwellenlängen-Scan der winCATS-Software im analytischen Dienstleistungsspektrum nach 1500 Substanzen screenen zu können. Durch den hohen Automatisierungsgrad erweist sich die planarchromatographische Methode als durchaus wettbewerbsfähig.

AMD wird eingesetzt, um die gewünschte Trennleistung auf der zur Verfügung stehenden Trennstrecke zu erhöhen. Bei großem Polaritätsbereich der zu trennenden Komponenten, bei hoher und unterschiedlicher Matrixbelastung sowie generell bei Vielkomponenten-Gemischen ist dies eine bevorzugt eingesetzte Trenntechnik. Durch die Mehrfach- und Gradientenentwicklung wird eine Fokussierung der Zonen erreicht und die Peakschärfe verbessert. Dies führt oft zu einer besseren Nachweisempfindlichkeit.

Überwachung der Oxytetracyclin-Dosis in mit Arzneistoffen versetztem Lachsfutter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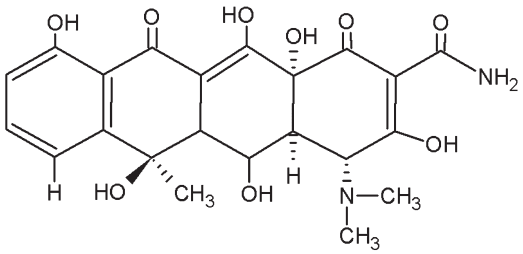
9

▲ *Prof. Dr. Mario Vega, Maritza Alvarado und Prof. Mario Aranda (rechts)*

Prof. Dr. Mario Vega*, Universität Concepcion, Chile, und seine Arbeitsgruppe unterstützten die Planar-Chromatographie in Südamerika aktiv durch Schulung und Consulting. So fand der zuletzt durchgeführte Servicekurs 2004 mit Verstärkung durch Jan Masthoff, CAMAG, Muttenz statt (siehe CBS 94). Dieses Jahr wurde das Team eingeladen, einen Workshop zur Planar-Chromatographie auf dem führenden Lateinamerikanischen Kongress für Chromatographie, COLACRO XI in Merida, Mexico, zu halten.

Bereits 1995 (CBS 75) präsentierte die Arbeitsgruppe eine Veröffentlichung zur Bestimmung von Chinolon-Antibiotika in Fisch und Fischfutter. In der Zwischenzeit wurden weitere Antibiotika und antibakterielle Mittel mittels Planar-Chromatographie untersucht [1–4]. Die folgende Arbeit setzt die Untersuchungen in mit Arzneistoffen versetztem Fischfutter fort. Im Vergleich zu früheren Veröffentlichungen weist die nachfolgende Methode Verbesserungen hinsichtlich der Probenvorbereitung und Chromatographie auf und zeigt auch Validierungsdaten.



▲ *Strukturformel von Oxytetracyclin*

Einleitung

Die Aquakultur in Chile hat sich zu einer der wichtigsten Aquakulturen weltweit entwickelt mit einer Exporthöhe von US$ 1150 Millionen im Jahre 2005. Leider nimmt die Verbreitung von Krankheiten aufgrund der Massen-Fischhaltung zu. Der Einsatz von Antibiotika und antibakteriellen Mitteln ist deshalb Routine: Das Breitbandantibiotikum Oxytetracyclin wird standardmäßig zur Behandlung und Vorbeugung einiger Fischkrankheiten eingesetzt.

Das Antibiotikum kann durch Zugabe zum Wasser, Injektion oder durch das Fischfutter verabreicht werden; letzteres wird aufgrund der geringen Kosten am häufigsten in der Aquakultur eingesetzt. Eine sehr wichtige Aufgabe ist die Sicherstellung einer homogenen Verteilung des Antibiotikums in den Futter-Pellets, eine Voraussetzung für eine korrekte antibakterielle Dosierung.

Einen grundlegenden Vorteil der Planar-Chromatographie nutzend, ermöglicht die Methode einen einfachen Extraktionsprozess ohne Festphasenextraktion oder Entfettung. Das Verfahren wurde in erster Linie entwickelt, um die Lachsfutter-Industrie mit einer notwendigen, zuverlässigen und kostengünstigen Hochdurchsatz-Methode auszustatten zur standardmäßigen Oxytetracyclin-Erfassung in mit Arzneimitteln versehenem Fischfutter, welches generell als eine komplexe Matrix wegen des hohen Fett- (28–34 %) und Proteingehaltes (41–48 %) anzusehen ist.

Probenvorbereitung

Kommerziell mit Arzneimitteln versehenes Fischfutter wurde von EWOS Chile erhalten. Zu 5 g gemahlener Fischfutterprobe wurden 50 mL Methanol und Salzsäure (0.15 mol/L) 9:1 (v/v) hinzugefügt. Oxytetracyclin wurde 30 min in einer mechanischen Schüttelvorrichtung extrahiert und weitere 20 min in einem Ultraschallbad. Nach dem Absetzen wurde 1 mL des klaren Überstandes für die Chromatographie benutzt.

Standardlösung

Oxytetracyclin-Hydrochlorid wurde in Methanol – Salzsäure (0.15 mol/L) 9:1 (1 mg/mL) gelöst.

Schicht

HPTLC Platten Kieselgel 60 F_{254} (Merck) 20 x 10 cm wurden mit Methanol vorgewaschen und bei 120 °C 30 min getrocknet. Zusätzlich wurden die Platten in eine 5 %ige EDTA-Lösung getaucht (pH 7.0) und bei 120 °C im Ofen für 1 Stunde getrocknet.

Probenauftragung

Bandförmig mit DC-Probenautomat, 20 Bahnen, Auftragevolumina 0.2–10 µL der Probelösung und 0.1–0.5 µL der Standardlösung, Bandlänge 6 mm (Bahnabstand 8.9 mm), seitlicher Randabstand 15 mm, unterer Randabstand 8 mm, Auftragegeschwindigkeit 100 nL/s

Chromatographie

In einer Doppeltrogkammer mit der organischen Phase von Chloroform – Methanol – EDTA 5 % 12:4:1 (v/v/v) nach 30 min Kammersättigung; Laufstrecke vom unteren Plattenrand 50 mm. Nach der Chromatographie wurde die Platte im Ofen bei 120 °C 10 min getrocknet.

Densitometrie

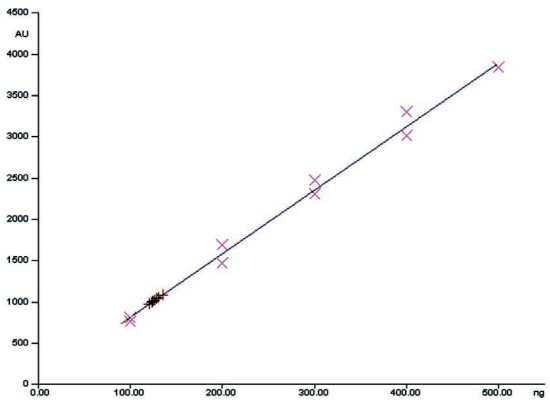
TLC-Scanner 3 mit winCATS Software; Fluoreszenzmessung bei UV 366/>400 nm; lineare Kalibration über die Peakfläche

Dokumentation

Mit VideoStore-Dokumentationssystem Aufnahme (Reflektion) bei UV 366/>400 nm

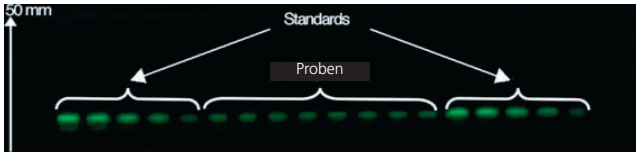
Ergebnisse und Diskussion

Die Kalibration (100–500 ng/Zone) wurde mittels linearer Regression durchgeführt und zeigte ein Bestimmtheitsmaß (r^2) von 0.9925.



10

▲ *Kalibrierfunktion von Oxytetracyclin (y = 7.687x + 42.975; sdv = 4.5 %; r = 0.9963)*

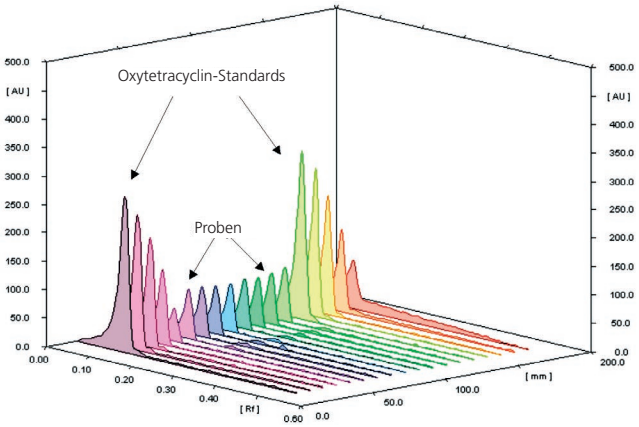


11

▲ *Chromatogramm von Oxytetracyclin-Standards und Fischfutterprobe-Extrakten; Aufnahme bei UV 366/>400 nm*

Die Wiederfindungsraten wurden anhand von Blindproben, die mit 3 verschiedenen Oxytetracyclin-Konzentrationen dotiert wurden, berechnet. Über 3 Tage wurde jedes Spike-Niveau täglich bestimmt (Doppelbestimmung). Die Wiederfindungsraten von Oxytetracyclin bei 500, 2500 und 5000 mg/kg betrugen 73 % ± 4.2 %, 101 % ± 2.6 % und 101 % ± 4.0 %. Die interne Laborpräzision zeigte eine relative Standardabweichung von 5.7 %, 2.6 % und 4.0 % bei 500, 2500 und 5000 mg/kg.

Unter Berücksichtigung des Auftragevolumens von 10 µL wurden als LOD (S/N 3) und LOQ (S/N 10) 14.8 mg/kg bzw. 49.2 mg/kg erhalten. Das Auftragen unterschiedlicher Probevolumina ermöglichte aufgrund der selektiven Fluoreszenzmessung die Quantifizierung von Oxytetracyclin in Lachsfutter zwischen 100 und 10.000 mg/kg ohne Matrixstörungen.



12

▲ *3D-Grafik von Oxytetracyclin-Standardbahnen (hR_F 12) und Fischfutterprobe-Bahnen*

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich

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4. S. Dhanesar, J. Planar Chromatogr. 12, 280-287, 1999.

Wie entsteht eine CBS-Ausgabe?

Den CBS gibt es seit 1963. Diese CAMAG Firmenzeitschrift ist neben dem seit 1987 erscheinenden Journal of Planar Chromatography das einzige Forum, das sich ausschliesslich mit der zeitgemässen Dünnschicht-Chromatographie befasst und somit ihre Anhänger auf dem Laufenden hält. An seiner Philosophie hat sich nichts geändert, wohl aber an seiner Erscheinung, an der Qualität seiner Beiträge, an seiner Auflage – sie beträgt zur Zeit 15 000 – und an der Art der Kommunikation.

Wie entsteht der CBS-Teil »Gelbe Seiten« (Literatur-Berichtsteil)?

Eine Anzahl international verteilter CBS-Referenten, die beruflich mit der modernen Dünnschicht-Chromatographie zu tun haben, senden uns Kurzreferate von DC/HPTLC-Publikationen in Zeitschriften, die ihnen zugeteilt sind, nach von uns erstellten Vorgaben (mit Fokus auf planar-chromatographische Parameter). Damit ist nicht beabsichtigt, die DC/HPTLC-Literatur lückenlos zu erfassen, sondern vorwiegend Arbeiten zu berücksichtigen, die aus der Sicht des bisherigen Wissensstandes Innovationen darstellen.


13

Einer der hervorragendsten und produktivsten CBS-Referenten ist *Professor Lin Leming* in Dalian. Er berichtet für den CBS seit 1984 und referiert alle relevanten chinesischen Zeitschriften. Das erklärt, warum ein verhältnismässig grosser Teil der im CBS erwähnten Publikationen chinesischer Provenienz ist, der aufgrund der Sprache westlichen Lesern sonst gar nicht zugänglich wäre. Professor Lin referiert aber auch eine Reihe westlicher Zeitschriften, u.a. das Journal of Chromatography.


14

Die Referate übermitteln die CBS-Referenten als Datenbank-File an *Frau Valeria Widmer*, die daraus das CBS-Referat editiert und das Layout des CBS-Berichtsteils erstellt. Frau Dr. Morlock lektoriert anschliessend die Referate und schickt das Ganze an unseren Grafiker, ergänzt mit ihrem »Dear Friends« Editorial.

Wie entsteht der CBS-Teil »Weisse Seiten«?

Dieser Teil des CBS erfuhr im Laufe der Jahre die grössten Wandlungen. War er seit seiner Einführung im Jahre 1967 in erster Linie der Bewerbung von CAMAG Produkten gewidmet, dient er seit einigen Jahren überwiegend dem »Öffentlichmachen« guter Anwendungen der Planar-Chromatographie, der Bekanntmachung von Schulungsmöglichkeiten und nur noch im untergeordneten Masse der Information über CAMAG Produkt-Neuheiten. Die Anwendungsbeiträge werden von namhaften Wissenschaftlern verfasst, von Frau Dr. Morlock ausgewählt und redigiert, d.h. bearbeitet und in ein weitgehend einheitliches Informationsformat gebracht. Die Beiträge werden im Endstadium von mehreren Fachleuten begutachtet.

Bei der Akquirierung der Beiträge wird Frau Dr. Morlock effizient von unserer deutschen Vertriebsmannschaft, gelegentlich auch von unseren internationalen Vertretungen unterstützt. Natürlich wird auch ein gewisser Anteil vom CAMAG Labor beigesteuert. Gerne können Sie eine interessante Anwendung auch direkt an cbs@camag.com mailen.


15

◀ *Dr. habil. Klaus Zieloff, bis 2003 Vertriebsleiter von CAMAG Berlin, jetzt im Teilruhestand, jedoch noch immer aktiv für wissenschaftliche Fragestellungen und für Kundenschulung:*

»Den grössten Teil der deutschsprachigen Auflage des CBS verschicken wir von CAMAG Berlin an Empfänger in Deutschland (>3500 Exemplare). Wir betrachten den CBS als unverzichtbares Bindeglied zu unseren Kunden. Die applikativen Beiträge haben bei den CBS-Empfängern nicht selten Geräteanschaffungen ausgelöst, in manchen Fällen sogar die Wiedereinführung der modernen DC/HPTLC als analytische Methode. Der Empfängerkreis dieser Hauszeitschrift wird von CAMAG Berlin laufend aktualisiert. Umfragen haben ergeben, dass die meisten CBS-Leser noch immer die gedruckte Form bevorzugen, wenngleich der Inhalt auch über Internet zugänglich ist.«

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PLANAR CHROMATOGRAPHY

CBS

Liebe Freunde

Ob Brot, Milch, Fleisch oder Fertiggerichte: Immer wieder findet man Stoffe in Lebensmitteln, die da nicht hineingehören – so in jüngster Zeit die Kontaminante ITX (Isopropylthioxanthon) in Milch, Joghurt und Fetten (S. 11–13). Die Kopplung der bewährten, wirtschaftlichen Planar-Chromatographie mit der Massenspektrometrie bietet hierbei zusätzliche Sicherheit und wird gezielt und zeitsparend immer dann eingesetzt, wenn die chromatographische Suche erfolgreich war. Dies erweist sich als sinnvolle Routine-Analytik für einen hohen Probendurchsatz, wenn dringender Handlungsbedarf binnen kürzester Zeit gefragt ist. Damit hat man auch bei etwaigen künftigen akuten Qualitätsproblemen eine effektive Methode zur Verfügung.

Wie intelligent und einfach man auch problematische Pestizide bestimmen kann, sehen Sie auf S. 2–5. Mittels einer Derivatisierungsreaktion wird das Pestizid Amitrol hochselektiv umgesetzt und damit detektierbar. Die Flexibilität hinsichtlich der Detektion zeigt sich immer wieder als Stärke der Planar-Chromatographie.

Einladen möchte ich Sie auch, sich mal Seite 8 »Wie entsteht eine CBS-Ausgabe?« anzuschauen. Vielleicht haben auch Sie eine interessante Anwendung, die Sie der DC/HPTLC-Gemeinschaft vermitteln möchten. Dazu steht Ihnen der CBS als Forum zur Verfügung, doch können Sie Ihre Methode auch auf dem Internationalen Symposium für HPTLC in Berlin, 9.–11. Oktober 2006, vorstellen. Workshops zur Weiterbildung, Austausch untereinander, neueste Entwicklungen, Beflügelung durch interessante Ideen, Lösungen für ihre Fragestellungen … viele Argumente, sich dafür Zeit zu nehmen.

Herzlichst Ihre

Gerda Morlock

Gerda Morlock
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Dear friends

Whether bread, milk, meat or convenience food: Again and again foreign, possibly harmful substances are detected in food! The most recent example was the contaminant ITX (isopropylthioxanthone) that was found in milk, yoghurt and fats (p. 11–13). In this context the coupling of planar chromatography, an established and cost-effective method, with mass spectrometry offered additional confirmation, confirmation that was targeted, fast with high sample throughput, and totally reliable. This seems to be a reasonable routine analysis that ensures quality when there is a sense of urgency to the task at hand.

Please see pages 2–5 for an intelligent and straightforward approach to determining problematic pesticides. By means of a derivatization reaction the pesticide Amitrol is detected with good selectivity. Flexibility regarding detection is once again a powerful feature of planar chromatography.

May I invite you to seriously ponder page 8 "The process of publishing CBS!" You also might have a challenging application to be shared with the TLC/HPTLC community. Use CBS as a forum or present your method at the International Symposium for HPTLC in Berlin, 9th–11th October 2006. Workshops for further education, fruitful discussions, latest research, refreshing new ideas, answers to your questions… all good reasons to come to Berlin this autumn.

Sincerely,

Gerda Morlock

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CAMAG

MÄRZ
MARCH
2006

96

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**

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

1. Reviews and books

96 001 C.F. POOLE (Department of Chemistry, Wayne State University, Detroit, MI 48202, USA): Thin-layer chromatography: challenges and opportunities. J. Chromatogr. A 1000 (1-2), 963-984 (2003). Identification of core technologies with the potential to influence the development of TLC over the next decade. Core technologies are identified as: (i) methods to provide a constant and optimum mobile phase velocity (forced flow and electroosmotically-driven flow), (ii) video densitometry for recording multidimensional chromatograms, (iii) in situ scanning mass spectrometry, and (iv) bioactivity monitoring for selective detection. In combination with two-dimensional, multiple development and coupled column-layer separation techniques these core technologies could dramatically increase the use of TLC for the characterization of complex mixtures. It is also demonstrated that TLC has strong potential as a surrogate chromatographic model for estimating biopartitioning properties. To convert these opportunities into practice the current state-of-the-art of the core technologies is described and the principle obstacles to progress identified.

review, HPTLC 1

96 002 P.G. RIGHETTI, Cecilia GELFI, R. SEBASTIANO, A. CITTERIO* (*Department of Chemistry, Materials and Engineering Chemistry, Politecnico di Milano, via Mancinelli 7, Milano 20131, Italy): Surfing silica surfaces superciliously. J. Chromatogr. A 1053 (1-2), 15-26 (2004). The mini-review summarizes the development of different classes of novel quaternarized heterocyclic compounds able to modulate and reverse the electroendoosmotic flow (EOF) in a most peculiar manner. The first class comprises mono-salt compounds, with the determinant omega-iodoalkyl chains of different lengths (typically C4-C8), able to be adsorbed by silicas at alkaline pH and spontaneously alkylate ionized silanols, thus becoming covalently affixed to it. The second class is constituted by di-salt compounds, attached at the termini of an alkyl chain of variable lengths (typically C4-C8). This second class is unable to bind covalently silica surfaces although in thin-layer chromatography it exhibits an extraordinary affinity for silica beads, contrary to the first one. On the basis of the strikingly different behavior structural rules are derived for the minimum requirements for general classes of amines to bind to silica walls and modify EOF. For compounds unable to bind covalently to the wall, the most important structural motif is two quaternary nitrogens spaced apart by a C4 chain: this seems to be the average distance (i.e. 0.8 nm) between two adjacent, ionized silanols for a snug fit. The other structural binding motif is the "hydrophobic decoration", i.e. the ratio of charged groups to alkyl residue in the various amines; amines with high levels of such alkane groups (i.e. with higher hydrophobicity), seem to bind more tenaciously to the wall, probably due to hydrophobic interaction not to the wall but among the amine derivatives themselves when carpeting the silica.

review, electroendoosmotic flow 1, 2a

96 167 A.M. SIOUFFI et al., see section 38

2. Fundamentals, theory and general

96 007 R. E. KAISER (Institute for Chromatography, P. O. Box 1141, 67085 Bad Dürkheim, Germany): Methods of detecting and/or reducing systematic errors in quantitative planer chromatography. Part 2. Systematic errors caused by separation systems. J. Planar Chromatogr. 18, 118-126 (2005). Discussion of systematic errors in analytical PLC data. Second part of a series covering fundamentals of systematic quantitative errors caused by separation systems, evaluation and calibration errors, nonlinear separation and quantitation techniques, the sf4-procedure for finding total systematic errors, systematic errors caused by regulations, and conclusions and proposals for quantitative planar liquid chromatography. 1) Great number of different separation systems. 2) Separation system and mobile phase. 3) Multiple and forward-backward runs - circular and linear systems. 4) Fast and small is better and reduces systematic errors. 5) Type of separation

system and quantitation mode. 6) Clean calibration substances available through a special mode of separation. 7) The gas phase in PLC. 8) Fundamentals of different separations systems (1. Important factors 2. Summary). 9) Possibilities and trends and the state of the art today; proneness to errors number (PEN).

quantitative analysis, systematic errors 2a

96 008 R. E. KAISER (Institute for Chromatography, P. O. Box 1141, 67085 Bad Dürkheim, Germany): Methods of detecting and/or reducing systematic errors in quantitative planar chromatography. Part 3. Evaluation and calibration errors. J. Planar Chromatogr. 18, 256-263 (2005). Third part of a series discussing fundamentals of systematic quantitative errors; systematic errors caused in separation systems; evaluation and calibration errors; nonlinear separation and quantitation techniques; the sf4-procedure for finding summarized systematic errors; systematic errors caused by regulation; conclusions and proposals for quantitative PLC. A correlation function is needed to obtain correct quantitative results from the raw data of a chromatogram - i. e. maximum peak height, peak area of part or all of a PLC spot, a line or a circle (for circular chromatography): $Yi = Ai + Bi \times Xi + Ci \times (Xi)^2 + Di \times (Xi)^3$. After 1) Introduction (and example), 2) Evaluation, 3) Calibration errors (3.1 Calibration function found by polynomial interpolation, 3.2 Calibration data analysis, 3.3 Data details for polynomial interpolation, 3.4 Analysis of the overall data quality, the data Goodness, 3.5 Effect of mathematical accuracy, 3.6 Positioning of the calibration sample ‚i' and the number of different concentrations/amounts to use) follows 4) A possible future of sampling and flexible precise positioning not only of the calibration substances.

systematic errors 2a

96 009 R. E. KAISER (Institute for Chromatography, P. O. Box 1141, 67085 Bad Dürkheim, Germany): Methods for detecting and reducing systematic errors in quantitative planar chromatography. - Part 1. Fundamentals of systematic quantitative errors. J. Planar Chromatogr. 18, 51-56 (2005). First part of a series covering systematic errors arising in separation systems, evaluation and calibration errors, nonlinear separation and quantitation techniques, the sf4-procedure for finding total systematic errors, systematic errors caused by regulations, and conclusions and proposals for quantitative planar liquid chromatography (PLC). 1) Nonsystematic errors: 1.1 Finding systematic errors hidden in nonsystematic errors, 1.2 Are random errors unstable? 1.3 Errors caused by (false) statements. 2) The main sources of systematic errors in quantitative PLC: 2.1 Chromatography, 2.2 Physics (baseline - baseplane; practical example). 2.3 Mathematics (detectability limit of the systematic error and the standard certainty STC). 3) Sampling chromatography.

densitometry, quantitative analysis, systematic errors 2a

96 013 A. PIENIAK, M. SAJEWICZ, K. KACZMARSKI, Teresa KOWALSKA* (*Institute of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland): The initial stage of the development of planar chromatograms. J. Planar Chromatogr. 18, 13-18 (2005). Re-investigation of the initial stage of the retention process in TLC, namely the moment of the first contact between the analyte deposited at the origin and the mobile phase employed. Presentation of the results obtained in two different ways, i. e. as the dependence of densitometrically measured concentration profiles and of retardation factors (RF) on the amount of analyte and on the mode of application (dry or wet) to the stationary phase layer.

chromatogram development, retention process 2a

96 002 P.G. RIGHETTI et al., see section 1

96 014 J. SHERMA (Lafayette College, Department of Chemistry, Easton, PA 18042, USA): Thin-layer chromatography-densitometry. J. Assoc. Off. Anal. Chem. 88, 1516 (2005). Short overview

on the use of densitometry in thin-layer chromatography as one of the most active areas in TLC research. See also the biennial reviews of Planar Chromatography by J. Sherma in Anal. Chem 74, 2653-2662 (2002) and 76, 3251-3261 (2004).

quantitative analysis, densitometry 2a

96 015 C. SULLIVAN, J. SHERMA* (*Department of Chemistry, Lafayette College, Easton, PA 18042, USA): Development and validation of a method for determination of caffeine in diuretic tablets and capsules by high-performance thin-layer chromatography on silica gel plates with a concentration zone using manual spotting and ultraviolet absorption densitometry. J. Assoc. Off. Anal. Chem. 88, 1537-1543 (2005). HPTLC of caffeine, acetaminophen and acetylsalicylic acid on silica gel with concentration zones using methanol - ethyl acetate 3:17. UV absorption densitometry is used for the quantitative determination of caffeine. Precision, accuracy, linearity, limits of detection and quantitation, and selectivity were validated. - A comparative study using a caffeine standard solution and a multicomponent analgesic tablet solution containing caffeine, acetaminophen, and acetylsalicylic acid showed that manual application on the concentration zone and instrumental application on the silica gel gave quite similar results in terms of number of theoretical plates, resolution, limit of detection, and linearity.

quality control, densitometry, quantitative analysis, HPTLC 2a, 32a

96 012 Barbara OSCIK-MENDYK (Faculty of Chemistry, M. Curie-Sklodowska University, M. Curie-Sklodowska Sq. 3, 20-031 Lublin, Poland): Comparison of adsorption in liquid-solid chromatography on the basis of different models of retention. J. Planar Chromatogr. 18, 199-202 (2005). Evaluation of two models of retention in liquid adsorption chromatography. Terms related to adsorption from two equations based on different retention models were compared. The terms were calculated for selected substances in four chromatographic systems with a different polar modifier. Analysis of the data has proved the comparability of the terms and that they can be used interchangeably to describe adsorption phenomena. TLC of e.g. nitroanilines, nitrotoluidines, nitrophenols on silica gel in horizontal chambers with heptane - ethyl acetate, heptane - 1,4-dioxane, heptane - tetrahydrofuran and heptane - ethylene chloride. Visualization with iodine vapor, detection by scanning.

adsorption, retention model 2b

96 004 Elzbieta BRZEZINSKA*, Grazyna KOSKA, Alicja KLIMCZAK (*Department of Analytical Chemistry, Medical University of Lódz, Muszy skiego 1, 90-151, Lódz, Poland): Application of thin-layer chromatographic data in quantitative structure-activity relationship assay of thiazole and benzothiazole derivatives with H1-antihistamine activity. Part II. J. Chromatogr. A 1007 (1-2), 157-164 (2003). Quantitative structure-activity relationship (QSAR) analysis of H1-antihistamine activity was carried out and chromatographic data of 2-[2-(phenylamino)thiazol-4-yl]ethanamine, 2-(2-benzyl-4-thiazolyl)ethanamine, 2-(2-benzhydrylthiazol-4-yl)ethylamine derivative, and 2-(1-piperazinyl- and 2-(hexahydro-1H-1,4-diazepin-1-yl)benzothiazole derivatives were obtained. Silica gel impregnated with solutions of selected amino acid mixtures (-Asp, Asn, -Thr and -Lys) were used in two developing solvents as human histamine H1-receptor (hH1R) antagonistic interaction models. Lipophilicity data of the examined compounds were obtained and used in the QSAR assay. Using regression analysis, relationships between chromatographic and biological activity data were found. The correlations obtained in the present experiment with NP-TLC are more significant that those obtained in the experiment with RP2-TLC because of the optimal fitting of the chromatographic system conditions to the lipophilicity of solutes. All proposed chromatographic models should facilitate pre-selection of the new drug candidates. The correlations of calculated pA2(H1) values of the tested compounds predicted by the use of the best equations versus their pA2(H1) obtained from the biological tests were significant (R2= 0.91-0.94).

pharmaceutical research, densitometry, quantitative analysis, qualitative identification, thiazoles, benzothiazoles 2c

96 005 Elzbieta BRZEZINSKA*, Grazyna KOSKA, K. WALCZYNSKI (*Department of Analytical Chemistry, Medical University of Lódz, Muszy skiego 1, 90-151, Lódz, Poland) : Application of thin-layer chromatographic data in quantitative structure-activity relationship assay of thiazole and benzothiazole derivatives with H1-antihistamine activity. Part I. J. Chromatogr. A 1007 (1-2), 145-155 (2003). A quantitative structure-activity relationship analysis of H1-antihistamine activity and chromatographic data of 2-[2-(phenylamino)thiazol-4-yl]ethanamine; 2-(2-benzyl-4-thiazolyl)ethanamine; 2-(2-benzhydrylthiazol-4-yl)ethylamine derivative; 2-(1-piperazinyl- and 2-(hexahydro-1H-1,4-diazepin-1-yl)benzothiazole derivatives were collected. Silica gel RP2, impregnated with solutions of selected amino acid mixtures (-Asp, -Asn, -Thr and -Lys), were used in two developing solvents as hH1R antagonistic interaction models. Using regression analysis, the relationships between chromatographic and biological activity data were found. The correlations obtained in regression analysis for the examined thiazole and benzothiazole derivatives with H1-antihistamine activity [pA2(H1)] represent their interaction with all the proposed biochromatographic models (S1-S7). Some of the calculated equations can be applied to predict the pharmacological activity of new drug candidates. The best multivariate relationships useful in predicting the pharmacological activity of thiazole and benzothiazole derivatives were obtained on RP2 phase with acetonitrile - methanol - buffer 2:2:1. The log P values of particular compounds are extremely important for this kind of activity.

pharmaceutical research, quantitative analysis, thiazoles, benzothiazoles 2c

96 114 T. LJ. DJAKOVIC-SEKULIC et al., see section 32a

96 010 M. KOSTECKA*, A. NIEWIADOMY, R. CZECZKO (*Department of Chemistry, University of Agriculture, Akademicka 15, 20031 Lublin, Poland): Evaluation of N-substituted 2,4-dihydroxyphenylthioamide fungicide lipophilicity using the chromatographic techniques HPLC and HPTLC. Chromatographia 62 (3-4), 121-126 (2005). 2,4-Dihydroxyphenylthioamide derivatives modified on the N-aryl ring have substantial fungicidal activity. To determine their quantitative structure-activity relationships their lipophilicity was determined by use of column liquid chromatography and thin-layer chromatography. Methanol - water systems were used as mobile phases and the linear dependences of retention (RM and log k) on volume fraction of organic modifier, phi, were determined. This enabled precise determination of lipophilicity (RMw and log kw) by extrapolation. Correlations were found between quantities characterizing the lipophilicity of the compounds. Deviations enabled discovery of compound structural features which increase or reduce lipophilicity. When these data were correlated with biological activity against the phytopathogenic fungi Alternaria alternata and Botrytis cinerea parabolic dependences were obtained.

qualitative identification, HPTLC, 2,4-dihydroxyphenylthioamide, fungicide 2c

96 011 M. M. NATIC*, R. M. BAOSIC, D. M. MILOJKOVIC-OPSENICA, Z. LJ. TESIC* (*Faculty of Chemistry, University of Belgrade, P. O. Box 158, 11001 Belgrade, Serbia and Montenegro): Estimation of the hydrophobicity of tris-beta-diketonato complexes from reversed-phase thin-layer chromatographic data. J. Planar Chromatogr. 18, 344-348 (2005). TLC of beta-diketonato complexes of the type M(acac) 3-n(phacphac)n and M(acac)3-n(phSacphSac)n, where M represents cobalt(III) or chromium(III), acac is the pentanedionato ion, phacphac the 1,3-diphenyl-1,3-propanedionato ion, and phSacphSac the 3-mercato-1,3-diphenylprop-2-en-1-thione ion (n= 0-3), on RP-18 with mixtures of tetrahydrofuran, acetonitrile, or acetone (as organic modifier) with water.

pharmaceutical research, qualitative identification 2c

96 126 Jolanta OBNISKA et al., see section 32a

96 127 Jolanta OBNISKA et al., see section 32a

96 019 Nada U. PERISIC-JANJIC*, T. LJ. DJAKOVIC-SEKULIC, L. R. JEVRIC, B. Z. JOVANOVIC (*Department of Chemistry, Faculty of Sciences, University of Novi Sad, Trg D. Obradovica 3, 21000 Novi Sad, Serbia and Montenegro): Study of quantitative structure-retention relationships for s-triazine derivatives in different RP HPTLC systems. J. Planar Chromatogr. 18, 212-216 (2005). Retention factors on RP-18 layers corresponding to zero percent organic modifier in the aqueous mobile phase were determined for five mobile phase mixtures: methanol - water, acetone - water, acetonitrile - water, 2-propanol - water, and tetrahydrofuran - water and relationships between the retention factors obtained with different organic mobile phase modifiers were examined. A variety of partition coefficients were calculated by use of different software products and the correlation between these partition coefficients and chromatographically obtained lipophilicity was analyzed. On the basis of correlations between retention factors and the partition coefficient log P, RP-18 with methanol - water as mobile phase was selected as the best RP-HPTLC system for determination of the octanol/water partition coefficient and thus the lipophilicity of the molecules. Visualization under UV light at 254 nm.

agricultural, toxicology, qualitative identification, HPTLC 2c

96 016 T. TUZIMSKI*, A. BARTOSIEWICZ (*Department of Inorganic and Analytical Chemistry, Medical University, Staszica 6, 20-081 Lublin, Poland, ttuzim@panaceum.am.lublin.pl): Correlation of retention parameters of pesticides in normal and RP systems and their utilization for the separation of a mixture of ten urea herbicides and fungicides by two-dimensional TLC on cyanopropyl-bonded polar stationary phase and two-adsorbent-layer multi-K plate. Chromatographia 58 (11-12), 781-788 (2003). Ten urea herbicides and fungicides have been separated by use of two-dimensional thin-layer chromatography. The largest differences are obtained by combination of normal-phase (NP) system and reversed-phase (RP) system on cyanopropyl-bonded polar adsorbents and on two-adsorbent layer containing a narrow zone of octadecyl silica and adjacent a wide zone of silica (or vice versa). The greatest spread of points was obtained for combination of nonaqueous NP systems with ethyl acetate on silica and RP systems comprising a polar solvent (methanol) in water on octadecyl silica adsorbent wettable with water (RP-18W). A good spread of points was also obtained for pairs of normal-phase systems with heptane-ethyl acetate mobile phases and reversed-phase systems with water-dioxane mobile phases, both on cyanopropyl-bonded polar adsorbents. The correlations of Rf values in NP/RP systems were utilized in practical separation of a mixture of ten urea pesticides using 2D-TLC on these adsorbents. The plates were scanned and videoscanned showing the real pictures of TLC chromatograms.

HPTLC, retention, normal and reversed phase, pesticide, herbicide, videoscanning 2c, 29

96 017 T. TUZIMSKI*, E. SOCZEWINSKI (*Department of Inorganic and Analytical Chemistry, Medical University, Staszica 6, 20-081 Lublin, Poland, ttuzim@panaceum.am.lublin.pl): Use of database of plots of pesticide retention (Rf) against mobile-phase compositions for fractionation of a mixture of pesticides by micropreparative Thin-Layer Chromatography. Chromatographia 59 (1-2), 121-128 (2004). Relationships between Rf values and mobile phase composition have been determined for urea herbicides and fungicides in normal-phase systems (NP) of the type silica-nonpolar or weakly polar diluent (heptane, toluene, diisopropyl ether) - polar modifier (ethyl acetate, tetrahydrofuran, dioxane, ethyl-methyl ketone and 2-propanol). These relationships constitute a retention database which has enabled to choose optimum systems for preliminary fractionation of a multicomponent mixture of pesticides by zonal micropreparative TLC. The mixture was applied from the edge of the layer in the “frontal + elution” mode which incre-

ased the separation efficiency because of displacement effects. The separated simpler fractions were applied to a silica plate and rechromatographed. The plate was videoscanned, furnishing a real picture of the plate showing preliminary separation of the simpler pesticide fractions. Complete separation of the fractions was carried out by two-dimensional thin-layer chromatography on plates with chemically bonded-cyanopropyl silica stationary phase using non-aqueous eluent in the first direction and aqueous reversed-phase eluent in the second direction.

agricultural, HPTLC, Retention, pesticide, herbicide, fungicide, videoscanning 2c, 29

96 006 J. D. VELICKOVIC, Z. LJ. TESIC, Dusanka M. MILOJKOVIC-OPSENICA* (*Faculty of Chemistry, University of Belgrade, P. O. Box 158, 11001 Belgrade, Serbia and Montenegro): Evaluation of the lipophilicity of some 1-arylpiperazines by planar chromatography. J. Planar Chromatogr. 18, 358-363 (2005). TLC of ten 1-arylpiperazines and five so-called ‚standards' (e.g. thiourea, anthracene, o-phenylenediamine) on RP-18 W in a horizontal chamber with 80-100 % methanol in water in steps of 5 % and 60-80 % dioxane in water in steps of 5 %. Detection under UV light at 254 nm.

pharmaceutical research, qualitative identification 2c

96 081 Elzbieta BRZEZINSKA et al., see section 32a

96 107 K. KRESTA et al., see section 32a

96 058 Alina PYKA et al., see section 23b

96 018 B. TYMAN-SZRAM, R. MUSIOL, M. SAJEWICZ, J. POLANSKI* (*Department of Organic Chemistry, Institute of Chemistry, University of Silesia, 40-006 Katowice, Poland): Thin-layer chromatographic determination of the pKa values of organic acids and bases. J. Planar Chromatogr. 18, 323-325 (2005). TLC of salicylic and benzoic acid on silica gel and of phenol, paracetamol, quinoline, 8-hydroxyquinoline, 4-bromoaniline, 2-chloroaniline, salicylic acid and benzoic acid on silica gel modified with DC 200 silicone oil with methanol - buffer in different volume ratios. TLC can be a valuable method for the determination of the pKa values of organic acids and bases. It is a simple and inexpensive alternative to the HPLC procedure.

pKa value 2d, 11a

96 003 S. BABIC, Alka 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): Use of a genetic algorithm to optimize TLC separation. J. Planar Chromatogr. 18, 112-117 (2005). Description of a method for optimization of a TLC separation based on use of a genetic algorithm. The procedure was tested by optimization of the reversed-phase HPTLC separation of a mixture of six pesticides; satisfactory results were obtained. The genetic algorithm was compared with the simplex method.

HPTLC, genetic algorithm 2e

96 035 M. TATARCZAK et al., see section 8a

3. General techniques

96 159 V. ZIVKOVIC-RADOVANOVIC et al., see section 33a

96 027 J. WANG (Wang Jie), D. WANG (Wang Dongyuan)*, G. SHE (She Gaohong), Z. WANG (Wang Zuo), J. WANG (Wang Jing), H. ZHANG (Zhang Hangxia), L. LI (Li Ledao), G. WANG (Wang Ge) (*Department of Analytical Chemistry, Shenyang Pharmaceuticak University, Shenyang 110016, China): Research on a semi-automatic sample applicator. J. Planar Chromatogr. 18, 132-140 (2005). Detailed description of a semi-automatic sample applicator. All components are commercially available at low cost; the process of assembly is very simple, especially the spraying head and the applicator mechanism. The type and the position of the spraying head, the gas pressure, and the application speed were tested. The relative standard deviations of band length and band width are < 1 % and < 2.5 %, respectively. The application speed is an important factor: the faster the application speed, the better.

sample application 3c

96 020 V.G. BEREZKIN*, A.O. BALUSHKIN, B.V. TYAGLOV, E.F. LITVIN (*A.V. Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences, Leninski pr. 29, GSP-1, 119991 Moscow, Russia): Use of low volatility mobile phases in electroosmotic thin-layer chromatography. J. Chromatogr. A 1084 (1-2), 13-17 (2005). A variant of electroosmotic thin-layer chromatography is suggested with the use of low volatility compounds as mobile phases aimed at drastically decreasing the evaporation of the mobile phase and improving the reproducibility of the method. The linear movement velocity of zones of separated compounds is experimentally shown to increase 2- to 12-fold in electroosmotic chromatography (compared to similar values in traditional TLC). The separation efficiency is also considerably increased.

quality control, electroosmotic thin-layer chromatography 3d

96 022 D. HANDLOSER (CAMAG Laboratory, Sonnenmattstrasse 11, 4132 Muttenz, Switzerland): Quality and reproducibility of chamber saturation with the new Automatic Development Chamber ADC 2. CBS 95, 10-13 (2005). HPTLC of five sulfonamides on silica gel with dichloroethane - methanol - 2-propanol - ammonia 25:5:5:1 in the twin trough chamber and ADC 2 with varied chamber saturation. Densitometric evaluation by absorbance measurement at 254 nm. Comparison of chamber saturation in conventional twin trough chamber and Automatic Development Chamber ADC 2 respectively. Reproducibility of Rf-values is better in ADC 2 due to higher quality in chamber saturation and less manual operations.

HPTLC, densitometry, Automatic Development Chamber ADC 2, chamber saturation 3d

96 023 Dorota KAZMIERCZAK, W. CIESIELSKI, R. ZAKRZEWSKI*, Monika ZUBER (*Department of Instrumental Analysis, University of Lódz, Pomorska 163, 90-236 Lódz, Poland): Application of iodine-azide reaction for detection of amino acids in thin-layer chromatography. J. Chromatogr. A 1059 (1-2), 171-174 (2004). The iodine-azide reaction was employed to TLC detection of sulphur-containing derivatives of protein and some non-protein amino acids. The derivatization reaction with phenyl isothiocyanate (PITC) took place directly on the plate before the developing step. Subsequently, the plates were sprayed with a mixture of sodium azide and starch solution in NP-TLC and in the case of RP-TLC sodium azide solution with starch incorporated into mobile phase and then exposed to iodine vapor. The spots became visible as white spots on violet-grey background. The obtained detection limits of PTC-derivatives have been compared with other visualizing techniques commonly used in TLC practice (UV254 and iodine vapor). The iodine-azide system has been proved to be the most favorable and enabled to detect quantities per spot in the range of 1-60 pmol (HPTLC) and 3-100 pmol (TLC).

HPTLC, postchromatographic derivatization, derivatization, amino acid, phenyl isothiocyanate, iodine-azide reaction 3e

96 026 Alina PYKA*, K. BOBER (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, 41-200 Sosnowiec, Poland): Visualizing agents for short-chain fatty acids in TLC. J. Planar Chromatogr. 18, 141-146 (2005). TLC of fatty acids (ethanoic, propanoic, butanoic, pentanoic, hexanoic, heptanoic, and octanoic acid) on silica gel with hexane - acetone 4:1 or acetone - water - chloroform - ethanol - aqueous ammonia 30:1:3:5:11 with chamber saturation for 30 min. Only bromocresol green, bromophenol blue, potassium permanganate, and methyl red can be used for the detection. Among the new visualizing agents dipping of the plates in an aqueous solution of alkaline blue enables the detection of the free fatty acids from propanoic to octanoic. Dipping generated better contrast regarding the spots than spraying.

qualitative identification, derivatization reagents 3e, 11c

96 024 M. LANCASTER*, D.M. GOODALL, E.T. BERGSTROEM, S. MCCROSSEN, P. MYERS (*Department of Chemistry, University of York, York YO10 5DD, United Kingdom): Quantitative measurements on wetted thin layer chromatography plates using a charge coupled device camera. J. Chromatogr. A 1090 (1-2), 165-171 (2005). This paper presents the first study of imaging of spots on thin-layer chromatographic plates whilst still wet with solvent. Imaging and quantification of Sudan II after development with dichloromethane was carried out in both reflectance and transmission modes, using a charge coupled device (CCD) camera. The relationship between peak area and sample loading was established at low sample loading, and found to be linear over an order of magnitude for both wet and dry modes with r2-values > 0.99. All data processing was carried out using the Beer-Lambert equation. Curvature at high loadings in the plots of integrated absorbance as a function of sample loading was accounted for using an empirical expression designed for use with the Kubelka-Munk treatment and apparent absorbance of the stationary phase due to scattering. Results are consistent with an effective path length significantly longer than the thickness of the sorbent layer. The limit of detection on a dry plate (0.5 ng) was found to be lower than on a wetted plate (2 ng). Precision was found to be 1-4 % RSD intra-plate and 8-14 % RSD inter-plate. Results are compared with quantification of the same analyte on dried plates.

quality control, quantitative analysis 3f

96 143 B. SPANGENBERG et al., see section 32a

96 021 J. HAN (Han Jing), D. WANG (Wang Dongyuan)*, D. WANG (Wang Dan), Y. WANG (Wang Yuping), M. ZHOU (Zhou Mi), L. LI (Li Ledao), H. ZHANG (Zhang Hongxia) (*Department of Analytical Chemistry, Shenyang Pharmaceutical University, 103 Wenhua Road, Shenyang 110016, China): Coupling development and elution, a new thin-layer chromatography technique J. Chromatogr. A 1002 (1-2), 213-219 (2003). Three methods of coupling development and elution were studied in this paper. (1) A new mode of solvent supplementation and eluate collection was developed for descending development. By using a new distributor and collector in descending development, components can be separated and eluted continuously. (2) The same effect can be realized with a slope distributor [Su et al., J. Planar Chromatogr. 14 (2001) 203] and a collector by horizontal development. (3) In-situ elution can be used to treat a developed silica plate, which can elute the separated components to the receptor without scraping them off. These three methods can be used individually, and the in-situ elution can be used with other modes of development.

coupling, development, elution 3g

96 025 M. PROSEK*, A. GOLC-WONDRA, I. VOVK, J. ZMITEK (*National Institute of Chemistry, Hajdrihova 19, 1000 Ljubljana, Slovenia): The importance of controlled drying in quantitative TLC. J. Planar Chromatogr. 18, 408-414 (2005). With RSD up to 10 % by far the largest source

of uncertainty, secondary chromatography is the main reason for poor precision in TLC. During the drying process mobile phase evaporates from the upper surface of the plate, and molecules of separated components inside the layer move up or down. The experimental results show the strong dependence of the intensity of the reflected light on the position of the spots inside the layer, giving an idea how to construct a device for drying and derivatization of TLC plates. Results obtained by the new TLC dryer significant improved reproducibility and precision.

quantitative analysis, drying process 3g

96 028 C. WIECZORREK (Department of e-commerce, MACHEREY-NAGEL GmbH & Co. KG, Valencienner Straße 11, 52355 Düren, Germany): Suitability of inexpensive image-generating systems for evaluation of thin-layer chromatography and gel electrophoresis. J. Planar Chromatogr. 18, 181-187 (2005). Cameras and image-analysis techniques have been used for the evaluation of TLC and gel-electrophoresis for several years. A computer program has been developed to enable use of inexpensive image-generating systems such as CCD cameras, webcams, or flat-bed-scanners for the evaluation of TLC and electrophoresis separations in UV and white light. TLC of flavonoids and anthraquinone dyes on silica gel with water - formic acid - methyl ethyl ketone - ethyl acetate 1:1:3:5 and toluene - cyclohexane 2:1, respectively.

densitometry, quantitative analysis, qualitative identification 3g

4. Special techniques

96 029 A. ORINAK*, G. VERING, H.F. ARLINGHAUS, J.T. ANDERSSON, L. HALAS, R. ORINAKOVA (*Institute for Chemistry, Department of Physical and Analytical Chemistry, Moyzesova 11, 041 54 Kosice, Slovakia): New approaches to coupling TLC with TOF-SIMS. J. Planar Chromatogr. 18, 44-50 (2005). A new hyphenated technique that enables coupling of TLC with time-of-flight secondary-ion mass spectrometry (TOF-SIMS) has been used for identification of gibberellic acid as model analyte. When TLC and TOF-SIMS are coupled on-line the chromatographic thin-layer must be modified to avoid TOF-SIMS background signal activity from the chromatographic material or solvents used. Two different types of TLC plates - aluminum backed silica gel and monolithic silica gel - were used. TOF-SIMS enables analyte detection with high mass resolution at a level of concentration not achieved by other methods.

TLC-MS online coupling 4e

96 030 E. TYIHAK et al., see section 5b

5. Hydrocarbons and halogen derivatives

96 030 E. TYIHAK*, A. MORICZ, P.G. OTT, G. KATAY, Z. KIRALY-VEGHELY (Plant Protection Institute, Hungarian Academy of Sciences, Budapest, Herman O. Str. 15, 1022 Budapest, Hungary): The potential of BioArena in the study of the formaldehydome. J. Planar Chromatogr. 18, 67-72 (2005). TLC of trans-resveratrol (trans-3,5-dihydroxystilbene), semicarbazide and dimedone (as standards) on silica gel using different mobile phases; OPLC on silica gel with chloroform - methanol 10:1. After drying bioautographic detection by immersion in the bacterial suspension of Pseudomonas savastanoi for 25 s. Visualization with MTT either after a short draining period or after overnight incubation.

bioautography 5b, 17a, 4e

8. Substances containing heterocyclic oxygen

96 031 Magdalena BARTNIK*, K. GLOWNIAK, A. MACIAG, M. HAJNOS (*Department of Pharmacognosy with Medicinal Plant Laboratory, Skubiszewski Medical University, Chodzki 1, 20-093 Lublin, Poland): Use of reversed-phase and normal-phase preparative thin-layer chromatography

for isolation and purification of coumarins from Peucedanum tauricum Bieb. leaves. J. Planar Chromatogr. 18, 244-248 (2005). Analytical and preparative TLC of coumarins from Peucedanum tauricum with bergapten, scopoletin, and coumarin A and B as standards on silica gel and on RP-2 with water - methanol 3:2 in horizontal chambers. Detection under UV light at 366 nm and densitometry at UV 366 and 320 nm. Re-chromatography with more selective mixtures of dichloromethane and acetonitrile 99:1 and 39:1. Identification by analytical co-chromatography with standards using mixtures of cyclohexane - ethyl acetate 3:1 and dichloromethane - acetonitrile 39:1.

herbal, preparative TLC, densitometry, Peucedanum tauricum 8a

96 032 C.B. FANG (Fang Congbing), X. WAN (Wan Xiaochun)*, C.J. JIANG (Jiang Changjun), H.Q. CAO (Cao Haiqun) (*Key Laboratory of Tea Biochemistry and Biotechnology, Ministry of Education and Agriculture, Anhui Agricultural University, Hefei 230036, Anhui, China): Comparison of HPTLC and HPLC for determination of isoflavonoids in several kudzu samples. J. Planar Chromatogr. 18, 73-77 (2005). HPTLC of isoflavonoids (puerarin, 3'-methoxypuerarin, daidzin, and daidzein) from Kudzu samples (a perennial leguminous plant of the genus Pueraria) on silica gel with chloroform - methanol - ethyl acetate- water 81:94:260:15. Quantitative determination by absorbance measurement at 254 nm. Repeatability and accuracy of HPLC compared with HPTLC are better, but separation of isoflavonoids by HPLC is time consuming and difficult. HPTLC is simple and rapid without tedious isolation of isoflavonoids. Separation and quantification of isoflavonoids from stem and leaf samples of kudzu is only achieved by HPTLC.

food analysis, quantitative analysis, qualitative identification, HPTLC, densitometry 8a

96 091 K. GLOWNIAK et al., see section 32e

96 033 Malgorzata KOZYRA*, K. GLOWNIAK, A. ZABZA, G. ZGOKA, T. MROCZEK, T. CIERPICKI, J. KULESZA, I. MUDO (*Department of Pharmacognosy with Medicinal Plant Laboratory, Skubiszewski Medical University, 1 Chodzki St., 20-093 Lublin, Poland): Column chromatography and preparative TLC for isolation and purification of coumarins from Peucedanum verticillare L. Koch ex DC. J. Planar Chromatogr. 18, 224-227 (2005). Analytical and preparative TLC of esculin, umbelliferone, bergapten, xanthotoxin, isoimperatorin, imperatorin, psoralen, cis-kellactone, pteryxin, and epoxypteryxin from Peucedanum verticillare extracts on silica gel with n-heptane - dichloromethane - ethyl acetate 4:5:10 and 3:4:3, and n-heptane - diisopropyl ether - isopropanol 32:8:5 in horizontal chambers after pre-conditioning for 10 min. Detection under UV light at 366 nm.

herbal, preparative TLC, qualitative identification 8a

96 034 Renata NOWAK*, T. TUZIMSKI (Chair and Department of Pharmaceutical Botany, Medical University, 1 Chodzki St., 20-093 Lublin, Poland): A solid-phase extraction - thin-layer chromatographic - fiber optical scanning densitometric method for determination of flavonol aglycones in extracts of rose leaves. J. Planar Chromatogr. 18, 437-442 (2005). HPTLC of quercetin and kaempferol in horizontal chambers on silica gel (prewashed with methanol) with four mobile phases, e.g.1,4-dioxane - toluene - 85 % acetic acid 6:24:1 or on cellulose with five mobile phases. Evaluation under UV light at 254 and 366 nm before and after spraying with a 2 % solution of zirconium (IV) dichloride oxide in methanol. Quantitative determination by absorbance measurement at 373 for quercetin and at 347 nm for kaempferol.

herbal, pharmaceutical research, HPTLC, densitometry, quantitative analysis 8a

96 035 M. TATARCZAK, J. FLIEGER*, H. SZUMILO (*Department of Inorganic and Analytical Chemistry, Medical University of Lublin, 20-081 Lublin, Staszica 6, Poland, JFlieger@panaceum.am.lublin.pl): Use of a graphical method to predict the retention times of selected flavonoids in HPLC from thin-layer chromatographic data. Chromatographia 61 (5-6), 307-309 (2005). Similarities and differences between the retention characteristics of octadecyl silica gel wettable with water used in TLC and RP-18 used in HPLC have been elucidated by use of the linear relationships between log k and RM. The stationary phases compared were investigated with the same mobile phases - binary mixtures of methanol and water, acetonitrile and water, and tetrahydrofuran and water. For these adsorbents of the same type but differing in specific surface area the correlation line was shifted by log (alpha system I / alpha system II). High values of the correlation coefficients obtained over the whole range of mobile phase organic modifier concentration examined indicated that the TLC systems could be used to predict HPLC conditions for flavonoid separation.

HPTLC, log k, flavonoids, prediction of retention times, MP transfer from HPTLC to HPLC 8a, 2e

96 125 Renata NOWAK et al., see section 32e

96 036 Irena VOVK*, Breda SIMONOVSKA, H. VUORELA (*Laboratory for Food Chemistry, National Institute of Chemistry, Hajdrihova 19, 1001 Ljubljana, Slovenia): Separation of eight selected flavan-3-ols on cellulose thin-layer chromatographic plates. J. Chromatogr. A 1077 (2), 188-194 (2005). HPTLC of (+)-catechin (C), (-)-epicatechin (EC), (-)-gallocatechin (GC), (-)-epigallocatechin (EGC), (-)-epicatechin gallate (ECg), (-)-epigallocatechin gallate (EGCg), procyanidin B1, and procyanidin B2 on cellulose prewashed with water (not necessary, when water was used as developing solvent) and dried with a hair dryer, with 1) water; 2) 1-propanol - water 1:4; 3) 1-propanol - water - acetic acid 4:2:1; 4) 1-propanol - water - acetic acid 2:8:1 in horizontal developing chamber (sandwich configuration). Detection with vanillin - H_3PO_4 reagent. Water enabled the separation of epimers C from EC and GC from EGC, as well as the dimers procyanidin B1 and B2. Additionally C, EGC, B1 and B2 were separated from all the other compounds. The best separation of the five main catechins (EC, GC, EGC, ECg, EGCg) present in green tea extract was achieved using 1-propanol - water - acetic acid 2:8:1. The chromatograms of oak bark extract developed in solvents with higher water content (1-propanol - water 1:4 and 1-propanol - water - acetic acid 2:8:1) showed less bands than chromatograms developed in solvents with higher organic modifier content (e.g. 1-propanol - water - acetic acid 4:2:1). It was proved that such behavior was due to the presence of procyanidins beside the main component catechin.

herbal, HPTLC, qualitative identification, postchromatographic derivatization, flavan-3-ols, green tea, oak 8b

10. Carbohydrates

96 037 M. B. ARANDA*, M. H. VEGA, R. F. VILLEGAS (*Department of Food Science, Nutrition and Dietetic, Faculty of Pharmacy, University of Concepcion, Barrio Universitario s/n Casilla 237, P.O. Box 403-0249, Concepcion, Chile): Routine method for quantification of starch by planar chromatography (HPTLC). J. Planar Chromatogr. 18, 285-289 (2005). HPTLC of glucose - after hydrolyis of starch using alpha-amylase and amyloglucosidase - on silica gel, pre-washed with methanol, treated by immersion in a 0.1 M solution of di-potassium hydrogen phosphate in methanol and activated for 30 min at 120 °C. Three-fold development was performed in a horizontal development chamber with acetonitrile - water 17:3. Detection by dipping in aniline-diphenylamine reagent, densitometry at 520 nm. Calibration was linear between 100 and 300 ng with a coefficient of determination r^2 of 0.9959. The limits of detection and quantification for starch as glucose were 0.26 and 0.51 g/100 g, respectively.

food analysis, HPTLC, densitometry 10

96 038 T. BERNARDI, Elena TAMBURINI*, G. VACCARI (*Chemistry Department, University of Ferrara, Via L. Borsari 46, 44100 Ferrara, Italy): Separation of complex fructo-oligosaccharides (FOS) and inulin mixtures by HPTLC-AMD. J. Planar Chromatogr. 18, 23-27 (2005) HPTLC-AMD of fructo-oligosaccharides and inulin mixtures (sucrose, 1-kestose, nystose, and fructosyl-nystose) on diol phases at 55-65 % relative humidity in a twin-trough chamber with an acetonitrile - acetone - water polarity gradient. Detection by derivatization with 4-aminobenzoic acid reagent and quantitation by scanning at 366 nm.

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

96 039 B.V. MCCLEARY*, P. ROSSITER (*Megazyme International Ireland Ltd., Bray Business Park, Southern Cross Rd, Bray, County Wicklow, Ireland): Measurement of novel dietary fibers. J. Assoc. Off. Anal. Chem. 87, 707-717 (2004). TLC of oligosaccharides (produced on hydrolysis of high molecular weight fructan with endo-inulinase) with fructose, glucose, kestose, and kestotetraose with n-propanol - ethanol - water 7:1:2. The plates were developed once, and spots were visualized by spraying the plates with 5 % sulfuric acid in methanol, followed by heating at 120 °C for 5 min.

food analysis, qualitative identification 10a

11. Organic acids and lipids

96 087 H. DANUTA SMOLARZ et al., see section 32e

96 041 Alina PYKA*, K. BOBER (*Silesian Academy of Medicine, Faculty of Pharmacy, Department of Analytical Chemistry, 4 Jagiellonska Street, 41-200 Sosnowiec, Poland): Investigation of homologous series of fatty acids by TLC. Part IV. Separation on RP 18 plates with ternary mobile phases. J. Planar Chromatogr. 18, 228-233 (2005.) HPTLC of heptanoic to eicosanoic acids on RP-18 (with and without concentrating zone). The best chromatographic conditions for separation of the fatty acids were RP-18 plates without concentrating zone and methanol - ethanol - water 9:9:2, and RP 18 plates with concentrating zone and methanol - ethanol - water or methanol - n-propanol - water 9:9:2. Detection by exposure to iodine vapor. Separation of acids from methanoic to butanoic and from tetracosanoic to triacontanoic acid was not possible.

HPTLC, qualitative identification 11a

96 018 B. TYMAN-SZRAM et al., see section 2d

96 040 K. MAEDER*, Andrea RUEBE, Sandra KLEIN (*Martin-Luther-University Halle, Institute of Pharm. Technology and Biopharmacy, Wolfgang-Langenbeck-Str.4, 06120 Halle/Saale, Germany, maeder@pharmazie.uni-halle.de): Quantitation of in vitro lipolysis products with HPTLC. CBS 95, 14-15 (2005). HPTLC-AMD of lipids from drug formulations with an 11-step gradient based on ethyl acetate. Detection by dipping in an aqueous copper sulfate solution followed by heating at 150 °C for 30 min. Quantitative determination by absorbance measurement at 675 nm, evaluation of peak area with calibration according to Hill kinetics.

pharmaceutical research, quantitative analysis, HPTLC, AMD, densitometry, lipid carrier, digestion 11c

96 026 Alina PYKA et al., see section 3e

13. Steroids

96 042 T. LARSEN*, J. AXELSEN, H. WEBER RAVN (*Department of Terrestrial Ecology, National

Environmental Research Institute, Vejlsøvej 25, 8600, Silkeborg, Denmark): Simplified and rapid method for extraction of ergosterol from natural samples and detection with quantitative and semi-quantitative methods using thin-layer chromatography. J. Chromatogr. A 1026 (1-2), 301-304 (2004). A new and simplified method for extraction of ergosterol (ergosta-5,7,22-trien-3beta-ol) from fungi in soil and litter was developed using pre-soaking extraction and paraffin oil for recovery. Recoveries of ergosterol were in the range of 94-100 % depending on the solvent to oil ratio. Extraction efficiencies equal to heat-assisted extraction treatments were obtained with pre-soaking extraction. Ergosterol was detected by TLC. Detection by fluorescence measurement, quantification limit was 8 ng. Using visual evaluation of images of TLC plates photographed in UV-light the quantification limit was 16 ng.

pharmaceutical research, herbal, quality control, traditional medicine, qualitative identification, quantitative analysis, densitometry, ergosterol 13c

96 118 S. MARIHAL et al., see section 32a

14. Steroid glycosides, saponins and other terpenoid glycosides

96 043 M. GLENSK*, M. WLODARCZYK, M. RADOM, W. CISOWSKI (*Wroclaw University of Medicine, Department of Pharmacognosy, pl Nankiera 1, 50-140 Wroclaw, Poland): TLC as a rapid and convenient method for saponin investigation. J. Planar Chromatogr. 18, 167-170 (2005). TLC and HPTLC of saponins from 70 species of Acer on silica gel in a horizontal chamber with chloroform - methanol - formic acid - water 200:80:20:19. Detection by spraying with 10 % sulfuric acid in ethanol or anisaldehyde - sulfuric acid reagent, followed by heating at 110 °C for 5 min, and evaluation in visible and UV light at 366 nm. Detection also by spraying with water or blood reagent.

herbal, qualitative identification, HPTLC 14

96 044 Erzsébet HAZNAGY-RADNAI*, P. LEBER, E. TOTH, G. JANICSAK, I. MATHE (*Institute of Pharmacognosy, University of Szeged, Eötvös Str. 6, 6720 Szeged, Hungary): Determination of Stachys palustris iridoids by a combination of chromatographic methods. J. Planar Chromatogr. 18, 314-318 (2005). TLC of iridoids (e. g. aucubin, catalpol, harpagide, 8-O-acetylharpagide, ajugoside) from aqueous plant extracts on silica gel with chloroform - methanol - water 25:10:1 and 160:55:8 and ethyl acetate - formic acid 7:4. Detection by spraying with a solution of 1 % 4-dimethylaminebenzaldehyde in conc. HCl containing acetic anhydride (Ehrlich's reagent) and heating at 105 °C for 5 min. Quantitation by densitometry at 540 nm.

herbal, densitometry, quantitative analysis, preparative TLC 14, 32e

96 045 Agnieszka LUDWICZUK*, Sz. NYIREDI, T. WOLSKI (*Department of Pharmacognosy with Medicinal Plant Laboratory, Skubiszewski Medical University, 1 Chodzki Street, 20-093 Lublin, Poland): Separation of the ginsenosides fraction obtained from the roots of Panax quinquefolium L. cultivated in Poland. J. Planar Chromatogr. 18, 104-107 (2005). TLC and HPTLC of ginsenosides (Rg1, Re, Rf, Rb1, Rc, Rb2, and Rd as standards) on silica gel with chloroform - methanol - ethyl acetate - water - hexane 10:11:30:4:2. OPLC on HPTLC silica gel with the same mobile phase but containing ethyl acetate or propyl acetate. Detection by spraying with Godin's reagent (5 % sulfuric acid and 1 % vanillin in ethanol) and heating at 105 °C for 10 min. Densitometric evaluation by absorbance measurement at 540 nm.

pharmaceutical research, herbal, HPTLC, densitometry, quantitative analysis 14

96 046 A. UMEK, A. RUPERT, A. MLINARIC, J. KAC* (*Faculty of Pharmacy, Askerceva 7, 1000 Ljubljana, Slovenia): HPTLC method for the determination of acteoside in ribwort plantain

(Plantago lanceolata L.). J. Planar Chromatogr. 18, 147-150 (2005). HPTLC of acteoside from leaves of Plantago lanceolata on silica gel with ethyl acetate - formic acid - water 18:1:1. Quantitative determination by densitometry at 334 nm. Intra-day and inter-day RSD were 0.58 and 2.0 %, respectively. Instrumental precision and repeatability of the method (CV) were found to be 0.62 and 1.5 %, respectively. The average recovery was 102.3 %.

herbal, quality control, densitometry, HPTLC, quantitative analysis, Plantago lanceolata
14

15. Terpenes and other volatile plant ingredients

96 057 D.L. MARTIN et al., see section 23a

96 047 E. PASTENE*, J. ALARCON, M. AVELLO, M. NAIL, A. URBINA, D. SEPULVEDA, M. VEGA (*Universidad de Concepción, Barrio Universitario c/n. P. O. Box 237, Concepción, Chile): Application of HPTLC to the analysis of horminone in Sphacele chamaedryoides (Balbis) Briq. J. Planar Chromatogr. 18, 221-223 (2005). HPTLC of horminone on silica gel in a twin-trough chamber with hexane - dioxane 9:1. Absorbance measurement at 271 nm. For fluorescence analysis the plates were dipped in 1 % diphenylboryloxyethylamine in ethyl acetate for 2 s, followed by drying and dipping in a solution of 5 % PEG 8000 in dichloromethane for 2 s. After 15 min fluorescent zones of horminone were scanned at 366/>400 nm. Use of the fluorescence reagent successfully reduced the limits of detection and quantification to 0.75 ng/spot and 1.51 ng/spot, respectively. Linearity range was from 60-300 ng/spot.

herbal, traditional medicine, quality control, HPTLC, densitometry, quantitative analysis, Sphacele chamaedryoides (Balbis) 15a

17. Amines, amides and related nitrogen compounds

96 048 Judite LAPA-GUIMARAES*, Jana PICKOVA (*Department of Food Science, Swedish University of Agricultural Sciences, P.O. Box 7051, 75007 Uppsala, Sweden): New solvent systems for thin-layer chromatographic determination of nine biogenic amines in fish and squid. J. Chromatogr. A 1045 (1-2), 223-232 (2004). TLC of dansyl derivatives of biogenic amines (agmatine, putrescine, tryptamine, cadaverine, spermidine, histamine, spermine, tyramine and beta-phenylethylamine) with chloroform - diethyl ether - triethylamine 6:4:1, followed by chloroform - triethylamine 6:1. Quantitative determination by fluorescence measurement at 330/>400 nm. Correlation coefficients of linear regressions were higher than 0.99 for all amines, except for agmatine (0.976). Detection limits were 10 ng for tryptamine, tyramine, histamine and beta-phenylethylamine, and 5 ng for the other amines. The overall repeatability of the chromatography was 1.82 % when including agmatine and barely 1.02 % for the other amines. The accuracy ranged from 105.97 % (agmatine) to 49.92 % (tryptamine). This thin-layer chromatography method was found to be an effective and precise analytical procedure to separate and determine biogenic amines. Its main advantages compared to previous procedures are that it uses less harmful solvent (diethyl ether instead of benzene) and can separate a larger group of biogenic amines.

food analysis, HPTLC, biogenic amines 17a

96 049 K. SPEER*, S. KRETZSCHMAR, Sibylle NEUGEBAUER, D. HUEBNER (*Institute of Food Chemistry, TU Dresden, Bergstr. 66, 01062 Dresden, Germany, Karl.Speer@chemie.tu-dresden.de): Determination of histamine and other biogenic amines in fish by planar chromatography. CBS 95, 2-4 (2005). TLC of seven biogenic amines (extracted with trichloroacetic acid from fish, followed by dansylation with dansyl chloride) on silica gel (prewashed with developing solvent) with benzene - chloroform - triethylamine 10:6:7 or 10:6:2 in horizontal developing chamber over 90 mm. Quantitative determination by fluorescence measurement at 365 nm/> 400 nm. Calibration using peak area, LOD 7.5 mg/kg, LOQ 56 mg/kg.

food analysis, quality control, densitometry, quantitative analysis, biogenic amines, dansylation
17a

96 030 E. TYIHAK et al., see section 5b

18. Amino acids and peptides, chemical structure of proteins

96 050 B. BASAK, D. BANDYOPADHYAY, A. BANERJI, Asima CHATTERJEE* (*Center of Advanced Studies on Natural Products Including Organic Synthesis, Department of Chemistry, Calcutta University, 92 A. P. C. Road, Kolkata 700 009, India): Use of ninhydrin for detection of silylated amino acids. J. Planar Chromatogr. 18, 251-252 (2005). TLC of 22 silylated amino acids on silica gel with butanol. After spotting the plates with the amino acid solutions the samples were sprayed with hexamethyldisilazane reagent (1 % solution in acetone), dried, heated at 110 °C for 20 min, and cooled. The developed plates were dried, sprayed with ninhydrin (0.25 % solution in acetone), dried completely, then further heated at 110 °C for 20 min. Detection limits of this method within 10 min are comparable with those of other methods.

qualitative identification, postchromatographic derivatization 18a

96 051 D. KAZMIERCZAK, W. CIESIELSKI, R. ZAKRZEWDKI* (*Department of Instrumental Analysis, University of Lódz, Pomorska 163, 90-236 Lódz, Poland): Separation of amino acids as phenyl thiocarbamyl derivatives by normal and reversed-phase thin-layer chromatography. J. Planar Chromatogr. 18, 427-431 (2005). HPTLC of twenty-one amino acids as phenyl thiocarbamyl derivatives on silica gel and RP-18 in a horizontal chamber with twelve two-component mobile phases and seven three-component mobile phases. Two-dimensional normal-phase TLC (e. g. ethanol - chloroform 2:1 in the first and methanol- dioxane 1:1 or methanol - chloroform - dioxane 1:1:1 in the second direction) and one-dimensional RP-TLC (with acetonitrile - sodium azide solution (pH 6.5) 1:4). Detection after NP-TLC by spraying with sodium azide and starch solution followed by exposure to iodine vapor; in RP-TLC the plates were developed with mobile phase containing sodium azide and starch solution and exposed to iodine vapor without being dried.

HPTLC, qualitative identification 18a

21. Purines, pyrimidines, nucleic acids and their constituents

96 052 Nada U. PERISIC-JANJIC*, G.S. USCUMLIC, N.V. VALENTIC (*Department of Chemistry, Faculty of Sciences, Trg D. Obradovica 3, 21000 Novi Sad, Serbia and Montenegro): The retention behavior of some uracil derivatives in normal and reversed-phase chromatography. Lipophilicity of the compounds. J. Planar Chromatogr. 18, 92-97 (2005). TLC of newly synthesized uracil derivatives on silica gel with benzene - methanol, benzene - acetonitrile, benzene - isopropanol, and HPTLC on RP-18 with water - methanol and water - acetonitrile. The mechanism of retention on different TLC supports was investigated and the retention constants determined for the uracils are discussed in terms of the physicochemical properties of both the solutes and stationary and mobile phases. Detection under UV light at 254 nm.

HPTLC, qualitative identification 21a

22. Alkaloids

96 053 M. GADZIKOWSKA, A. PETRUCZYNIK, Monika WAKSMUNDZKA-HAJNOS*, M. HAWRYL, G. JOZWIAK (*Department of Inorganic Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081 Lublin, Poland): Two-dimensional planar chromatography of tropane alkaloids from Datura innoxia Mill. J. Planar Chromatogr. 18, 127-131 (2005). TLC and HPTLC of alkaloids (e.g. atropine, homatropine, L-hyoscyamine, scopolamine N-oxide, tropine, tropic acid) from Datura innoxia on silica gel with methanol - acetone - aqueous ammonia 10:8:1 or methanol - acetone - diethylamine 25:24:1; or on RP-18 with methanol - water (buffered at pH 3.4) containing 0.01 M HDEHP; 2D-TLC: first direction on silica gel with methanol - acetone - dietylamine 25:24:1 and second direction on RP-18 with methanol - water (buffered at

pH 3.4) containing 0.01 M HDEHP. Detection by spraying with Dragendorff's reagent. Densitometric evaluation at 520 nm and at 205 nm (before derivatization).

pharmaceutical research, herbal, HPTLC, qualitative identification, Datura innoxia, alkaloids 22

96 054 S. KHATOON*, M. SRIVASTAVA, A. K. S. RAWAT, S. MEHROTRA (*Pharmacognosy and Ethnopharmacology Division, National Botanic Research Institute, Rana Pratp Marg, Lucknow 226001, India): HPTLC method for chemical standardization of Sida species and estimation of the alkaloid ephedrine. J. Planar Chromatogr. 18, 364-367 (2005). HPTLC of ephedrine on silica gel in a presaturated twin-trough chamber with toluene - diethyl acetate - diethylamine 7:2:1. Quantitative determination by absorbance measurement at 200 nm. Also HPTLC of methanolic extracts of roots and aerial parts of different Sida species with toluene - chloroform - ethanol 13:30:7. Common and distinguishing bands were observed.

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

96 055 I. MALINOWSKA, M. GADZIKOWSKA, Monika WAKSMUNDZKA-HAJNOS *, A. KRAMEK (*Department of Inorganic Chemistry, Medical University, Staszica 6, 20-081 Lublin, Poland): Mobile-phase velocity - a tool for separation of alkaloids by OPLC. J. Planar Chromatogr. 18, 176-180 (2005). OPLC separation of alkaloids on silica gel using different mobile-phase velocities from 100 to 400 µL/min and investigation of this effect on properties such as retardation factors, reproducibility, efficiency, number of theoretical plates, HETP, and resolution. OPLC of tertiary alkaloid standards (allocryptopine, protopine, chelidonine) and quaternary alkaloid standards (chelerythrine, chelilutine, sanguinarine, and chelirubine) on silica gel with toluene - ethyl acetate - methanol 14:3:3 for tertiary alkaloids and toluene - ethyl acetate - methanol 83:15:2 for quaternary alkaloids.

herbal, qualitative identification, densitometry, OPLC, mobile-phase velocity 22

96 056 Anna PETRUCZYNIK *, M. WAKSMUNDZKA-HAJNOS, M. HAJNOS (*Department of Inorganic Chemistry, Medical University, Staszica 6, 20-081 Lublin, Poland): The effect of chromatographic conditions on the separation of selected alkaloids on silica layers. J. Planar Chromatogr. 18, 78-84 (2005). HPTLC of alkaloid standards (boldine, berberine, emetine, glaucine, codeine, laudanosine, narceine, narcotine, noscapine, papaverine, protopine, tubocurarine, atropine, hyoscyamine, scopolamine, quinine, cinchonine, brucine, yohimbine, strychnine, caffeine, novocaine) on silica gel with a variety of aqueous and nonaqueous mobile phases. Location of spots under UV light at 254 nm. Densitometry at 254 nm. Systems with the best selectivity and efficiency were used to separate alkaloid standard mixtures and plant extracts by 2D-TLC (e. g. methanol - water 4:1, containing 1 % ammonia, in the first direction and methanol - acetone - diisopropyl ether - diethylamine 15:15:69:1 in the second direction).

HPTLC, qualitative identification, alkaloids 22

23. Other substances containing heterocyclic nitrogen

96 057 D. L. MARTIN, B. FRIED*, J. SHERMA (*Department of Biology, Lafayette College, Easton PA 18042, USA): The absence of beta-carotene and the presence of biliverdin in the medicinal leech Hirudo medicinalis as determined by TLC. J. Planar Chromatogr. 18, 400-402 (2005). HPTLC of beta-carotene on silica gel and RP-18 with preadsorbent sample-application zones (prewashed with dichloromethane - methanol 1:1) with petroleum ether (35-60 °C) - acetonitrile - methanol 1:2:2 or petroleum ether (20-40°C) - acetone 7:3. HPTLC of biliverdin on silica gel with n-butanol - methanol - water 4:2:3. Quantitative determination by absorbance measurement at 628 nm.

clinical chemistry research, HPTLC, quantitative analysis, densitometry 23a, 15a

96 058 Alina PYKA*, M. DOLOWY, D. GURAK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, Jagiellonska 4, 41-200 Sosnowiec, Poland): Use of selected structural descriptors for evaluation of the lipophilicity of bile acids investigated by RP HPTLC. J. Planar Chromatogr. 18, 465-470 (2005). HPTLC of cholic, glycocholic, glycodeoxycholic, chenodeoxycholic, deoxycholic, lithocholic, and glycolithocholic acid on RP-18 W, RP-2, and cyano phases in a presaturated chamber with mixtures of an organic modifier (methanol, dioxane, acetonitrile, acetone) and water in different volume proportions which were varied in steps of 5 % from 35 to 80 %. Detection by spraying with a 10 % aqueous solution of sulfuric acid or by dipping in a 10 % solution of phosphomolybdic acid in ethanol and heating at 120 °C for 20 min. Investigation of relationships between lipophilicity obtained by use of RP-HPTLC, experimental and theoretical partition coefficients, and selected structural descriptors.

pharmaceutical research, HPTLC, qualitative identification 23b, 2d

27. Vitamins and various growth regulators

96 059 F. BUHL, Barbara SZPIKOWSKA-SROKA*, M. GALKOWSKA (*Institute of Chemistry, Department of Analytical Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland): Determination of L-ascorbic acid after chromatographic separation. J. Planar Chromatogr. 18, 368-371 (2005). TLC of L-ascorbic acid in aqueous extracts of pharmaceutical preparations and pepper juice on silica gel with glacial acetic acid - acetone - methanol - benzene 3:1:4:14, butanol - formic acid - water 200:10:3, and water - glacial acetic acid - ethyl methyl ketone - ethyl acetate 1:2:2:5. Detection under UV 254 nm. Quantitative determination by absorbance measurement at 588 nm after oxidation with iodate.

quality control, food analysis, qualitative identification 27

28. Antibiotics, Mycotoxins

96 061 Sandra BABIC*, D. ASPERGER, D. MUTAVDZIC, 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 sulfonamides and trimethoprim in spiked water samples by solid-phase extraction and thin-layer chromatography. J. Planar Chromatogr. 18, 423-426 (2005). HPTLC of antibiotics (sulfadimidine, sulfadiazine, sulfaguanidine, trimethoprim) on silica gel without chamber saturation in a twin-trough chamber with chloroform - methanol 89:11. Quantification by videodensitometry at 254 nm. Limit of detection was 0.05 µg per spot for sulfadimidine, sulfadiazine, and sulfaguanidine, and 0.1 µg per spot for trimethoprim.

environmental, HPTLC, densitometry, quantitative analysis 28a, 37c

96 062 Joanna NOWAKOWSKA (Medical University of Gdansk, Faculty of Pharmacy, Department of Physical Chemistry, Al. Gen. Hallera 107, 80-416 Gdansk, Poland): Normal and reversed-phase TLC separation of some macrcyclic antibiotics with non-aqueous mobile phases. J. Planar Chromatogr. 18, 455-459 (2005). HPTLC of erythromycin, troleandomycin (oleandomycin triacetate), tylosin, rifamycin B, and rifampicin on silica gel and on RP-18 in a presaturated chamber with wide-ranging mixtures containing 0 to 100 % esters or ketones in dimethyl sulfoxide or hexamethyldisiloxane. Detection by spraying with a mixture of concentrated sulfuric acid and methanol 1:4 followed by heating at 120 °C for 10 min. Chromatographic retention data and a possible retention mechanism are discussed.

pharmaceutical research, HPTLC, qualitative identification 28a

96 063 A. SZABO, B. ERDELYI*, J. SALAT, G. MATE (*Fermentation Pilot Plant, IVAX Drug Research Institute, Berlini u. 47-49, 1049 Budapest, Hungary): Densitometric determination of some bioactive guanidinium compounds without post-derivatization. J. Planar Chromatogr. 18, 203-206 (2005). TLC of primycin (a mixture of related compounds), streptomycin, dihydrostreptomycin on silica gel with A) n-butanol - water - methanol - acetic acid 4:2:1:1; and B) chloroform - methanol - water - 35% formic acid - n-butanol - formaldehyde 160:53:9:6:3:3. When using phase B repeated development improved the resolution. After development the plates were dried in a vacuum chamber at 100 °C. An efficient prewashing technique (with methanol - 35 % formic acid 1:1 followed by drying with hot air) made the TLC plates suitable for densitometric measurements at short wavelengths. Quantitative determination by absorbance measurement at 200 nm.

quality control, densitometry, quantitative analysis 28a

96 060 T. B. TOSTI, K. DRLJEVIC, D. M. MILOJKOVIC-OPSENICA, Z. LJ. TESIC* (*Faculty of Chemistry, University of Belgrade, P. O. Box 158, 11001 Belgrade, Serbia and Montenegro): Salting-out thin-layer chromatography of some macrolide antibiotics. J. Planar Chromatogr. 18, 415-418 (2005). TLC of macrolide antibiotics (roxithromycin, midecamycin, erythromycin, azithromycin, and erythromycin ethylsuccinate) on cellulose with aqueous ammonium sulfate solutions concentrated from 0.5 to 4.0 M. Detection by exposure to iodine vapor.

pharmaceutical research, qualitative identification 28a

96 064 Irena VOVK*, B. SIMONOVSKA (*National Institute of Chemistry, Laboratory for Food Chemistry, Hajdrihova 19, 1000 Ljubljana, Slovenia): Development and validation of a thin-layer chromatographic method for determination of chloramphenicol residues on pharmaceutical equipment surfaces. J. Assoc. Off. Anal. Chem. 88, 1555-1561 (2005). HPTLC of chloramphenicol on silica gel in a horizontal developing chamber (36 applications per plate) using n-hexane - ethyl acetate 7:13. Quantitative determination by absorbance measurement at 280 nm. Mean recovery was 95.8 %, and the coefficient of variation was 5.8 %. The detection limit was 3 ng, and the quantitation limit 10 ng.

quality control, densitometry, quantitative analysis, HPTLC 28a

29. Pesticides and other agrochemicals

96 065 H.Q. CAO (Cao Haiqun), Y.D. YUE (Yue Yongde)*, R.M. HUA (Hua Rimao), F. TANG (Tang Feng), R. ZHANG (Zhang Rong), W. FAN (Fan Wei), H.Y. CHEN (Chen Haiyan) (*International Center for Bamboo and Rattan, 100102 Beijing, China) : HPTLC determination of imidacloprid, fenitrothion and parathion in Chinese cabbage. J. Planar Chromatogr. 18, 151-154 (2005). HPTLC of imidacloprid, fenitrothion, and parathion on silica gel (prewashed with methanol and activated at 110 °C for 30 min) with hexane - acetone 7:3 in an unsaturated twin-trough chamber. Quantitative determination by absorbance measurement at 287 nm.

food analysis, HPTLC, quantitative analysis 29

96 067 J. RASMUSSEN*, O. S. JACOBSEN (*Department of Agricultural Science, The Royal Veterinary and Agricultural University (KVL), Copenhagen, Denmark): Thin-layer chromatographic methods for the analysis of eighteen different 14C-labeled pesticides. J. Planar Chromatogr. 18, 248-251 (2005). TLC of 14C-labeled pesticides (metribuzin, linuron, isopropylanilin, bentazon, metamitron, MCPA, mecoprop, isoproturon, MD-IPU, diuron, diazinon, simazine, 2,4-dichlorophenoxyacetic acid, chlorsulfuron, metsulfuron-methyl, thifensulfuron-methyl, tribenuron-methyl, triazinamine, methyl-triazinamin, terbutylazine) on silica gel and RP-18 in horizontal chamber with isopropanol - ethyl acetate - acetic acid 30:70:0.1, isopropanol - ethyl acetate - hexane - acetic acid 10:40:50:0.1 and 30:40:30:0.1, isopropanol - hexane - acetic acid 30:70:0.1,

hexane - diethyl ether 1:1, methanol - water 3:2, methanol - water - acetic acid (pH 3) 3:2, methanol - water - ethyl acetate 13:5:2, acetonitrile - water 3:2, acetonitrile - water -phosphoric acid (pH <2) 1:9. After development the plates were exposed to a phosphor screen for 24 hours and analyzed by use of a Cyclone storage phosphor system.

environmental, quality control, autoradiography 29

96 070 T. TUZIMSKI (Department of Physical Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081 Lublin, Poland): Two-stage fractionation of a mixture of pesticides by micropreparative TLC and HPLC. J. Planar Chromatogr. 18, 39-43 (2005). Micropreparative TLC of 10 pesticides (isoproturon, diuron, momolinuron, desmetryn, methiocarb, atrazine, fenitrothion, terbutryn, bromopropylate, aziprotryne) for preliminary fractionation on silica gel in horizontal DS chambers with tetrahydrofuran - n-heptane 1:4; detection under UV at 254 nm. HPTLC of the separated fractions on RP-18 W with methanol - water 3:2 and acetonitrile - water 3:2; densitometry at 254 nm.

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

96 016 T. TUZIMSKI et al., see section 2c

96 017 T. TUZIMSKI et al., see section 2c

96 069 F. TANG, S. GE, Y. YUE*, R. HUA, R. ZHANG (*International Centre for Bamboo and Rattan, 100102 Beijing, China): High-performance thin-layer chromatographic determination of carbamate residues in vegetables. J. Planar Chromatogr. 18, 28-33 (2005). HPTLC and TLC of four carbamate residues (pirimicarb, methomyl, carbofuran, carbaryl) in vegetables on silica gel (prewashed with chloroform - methanol 1:1 followed by drying at 110 °C for 30 min) with system I (two fold development with first toluene - acetone 4:1, and second dichloromethane - acetone 4:1), and system II (two fold development with first ethyl acetate - petroleum ether 3:2, and second chloroform - petroleum ether 9:1). Quantitative determination by densitometric scanning at 254 and 366 nm.

food analysis, densitometry, quantitative analysis, HPTLC 29c

96 066 H. HEGEWALD (Lacrome LDA, Rua Cesar Batista 6 D, 7000-715 Evora, Portugal, lacrome@clix.pt): Pre-chromatographic in situ derivatization of glyphosate and AMPA. CBS 95, 9 (2005). HPTLC of glyphosate and AMPA derivatized in situ on the application position of the plate with FMOC, on silica gel with n-butanol - water - acetic acid 5:1:1 over 70 mm in an unsaturated twin trough chamber. After drying dipping in paraffin - toluene 1:1 for fluorescence enhancement. Quantitative determination by fluorescence measurement with mercury lamp at 265/M 360 nm. Linear calibration using peak height, LOD 0.5 ng absolute per substance zone for glyphosate-FMOC and 0.2 ng for AMPA-FMOC.

environmental, HPTLC, densitometry, quantitative analysis, herbicide, water analysis 29d

96 068 L. SONG (Song Liyan)*, Y. ZHAO (Zhao Youcai), R. HUA (Hua Rimao) (*Environment Engineering, State Key Laboratory of Pollution Control and Resource Reuse, School of Environmental Science and Engineering, Tongji University, Shanghai 200092, China): Separation of fenoxaprop-p-ethyl biodegradation products by HPTLC. J. Planar Chromatogr. 18, 85-88 (2005). 11 HPTLC of fenoxaprop-p-ethyl and degradation products on silica gel (prewashed with methanol - chloroform 1:1 and activated at 110 °C for 30 min) in a twin-trough chamber with tolu-

ene - dichloromethane 7:3. Visualization on irradiation with an UV lamp at 236 nm. Quantitative determination by absorbance measurement at 236 nm.

agricultural, qualitative identification, densitometry, HPTLC, quantitative analysis 29d

96 073 W. WEBER*, W. SEITZ, Anna AICHINGER (*Zweckverband Landeswasserversorgung, Betriebs- und Forschungslaboratorium, Am Spitzigen Berg 1, 89129 Langenau, Germany, weber.w@lw-online.de): Ultra trace analysis of glyphosate and AMPA in water with HPTLC. CBS 95, 5-7 (2005). HPTLC of glyphosate and AMPA in surface water, in vitro-derivatized with FMOC, on silica gel (prewashed with 2-propanol by immersion for 24 h) with the organic layer of n-butanol - water - acetic acid 5:4:1 over 70 mm. Quantitative determination by fluorescence measurement with deuterium lamp at 268/M 360 nm. Linear calibration using peak area, LOD 50 ng/L.

environmental, agricultural, HPTLC, densitometry, quantitative analysis, herbicide, glyphosate, glufosinate, water analysis, in vitro derivatization 29d

32. Pharmaceutical and biomedical applications

96 074 E. A. ABOURASHED*, M. S. ABDEL-KADER, A.-A. M. HABIB (*Department of Pharmacognosy, College of Pharmacy, King Saud University, Riyadh 11451, Saudi Arabia): HPTLC determination of sildenafil in pharmaceutical products and aphrodisiac herbal preparations. J. Planar Chromatogr. 18, 372-376 (2005). HPTLC of sildenafil in four commercial products and three aphrodisiac herbal preparations on silica gel after pre-saturation with chloroform - methanol - diethylamine 90:10:1. Quantitative determination by absorbance measurement at 305 nm. Recovery was 100.6 and 98.2 % for pure and spiked samples.

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

96 076 Ratna AWATE*, V. DHAINJE, DR. VAISHALI SHIRSAT (*Bombay College of Pharmacy, Kalina, Santacruz (E), Mumbai 400098, India): Estimation of etoricoxib in tablets by HPTLC. Abstract G-35, IPC (2005). HPTLC of etoricoxib in tablets on silica gel with n-hexane - ethyl acetate 1:3. Quantitative determination by absorbance measurement at 237 nm. The method was linear within the range of 100-500 ng/spot with a recovery rate of 97.4 %. LOD was 40 ng/spot and LOQ 100 ng/spot.

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

96 077 L.I. BEBAWY*, M.F. EL TARRAS, S.A. EL SABOUR (*National Organization for Drug Control and Research, 6 Hussin Kammel El Din Dokki, P. O. Box 12311, Giza, Egypt): Determination of trimetazidine dihydrochloride in the presence of its acid-induced degradation products. J. Assoc. Off. Anal. Chem. 87, 827-833 (2004). TLC of trimetazidine dihydrochloride and degradation products (e. g. piperazine and 2,3,4-trimethoxymethyl benzene) on silica gel with methanol - ammonia 200:3. Detection under UV light at 254 nm and densitometry at 215 nm. The method was applicable over a concentration range of 2.00-9.00 µg/spot with a mean percentage accuracy of 99.86 +/- 0.92.

quality control, densitometry, quantitative analysis 32a

96 078 A. BERECKA, Anna GUMIENICZEK*, H. HOPKALA (*Department of Medicinal Chemistry, Medical University of Lublin, Chodzki Str. 6, 20-093 Lublin, Poland) : Retention behavior of new oral antidiabetic drugs in reversed-phase chromatography. J. Planar Chromatogr. 18, 61-66 (2005). TLC of three antidiabetic agents (pioglitazone, rosiglitazone, and repaglinide) on silica

gel RP-8 with buffer - organic modifier binary mobile phases of widely different composition in horizontal chambers; visualization under UV light at 254 nm. Peak-purity tests by recording the in-situ spectra in the wavelength range of 200 to 400 nm.

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

96 079 R. BHUSHAN*, S. JOSHI, M. ARORA, M. GUPTA (*Department of Chemistry, Indian Institute of Technology Roorkee, Roorkee-247 667, India): Study of the liquid chromatographic separation and determination of NSAID. J. Planar Chromatogr. 18, 164-166 (2005). TLC of non-steroidal anti-inflammatory drugs (NSAID), i.e. mefenamic acid, naproxen, ibuprofen, flurbiprofen, ketoprofen, paracetamol, and diclofenac, on silica gel with chloroform - methanol , chloroform - ethyl acetate, acetonitrile - methanol - water, acetonitrile - methanol in different proportions. Detection with iodine vapor.

quality control, pharmaceutical research, qualitative identification, preparative TLC 32a

96 081 Elzbieta BRZEZINSKA*, J. STOLARSKA (*Medical University of Lódz, Department of Analytical Chemistry, ul. Muszynskiego 1, 90-151 Lódz, Poland): Determination of the partition and distribution coefficients of biologically active compounds by reversed-phase thin-layer chromatography. J. Planar Chromatogr. 18, 443-449 (2005). TLC of six 2-[1-(4-alkylpiperazinyl)]benzothiazoles, two 2-[4-(1-alkyl)piperidinyl]benzothiazoles, three 2-(N,N'-dimethyl-1,2-ethane diamino)benzothiazoles, and 2-[1-(4-aminopiperidinyl)]benzothiazole on RP-18 in a saturated horizontal chamber with mixtures of acetone and aqueous Tris (tris(hydroxymethyl)-aminomethane) buffer (pH 7.4). The organic modifier (acetone) content varied from 40 to 85 % in 5 % increments. Detection under UV.

pharmaceutical research, qualitative identification 32a, 2d

96 082 M. CAKAR, G. POPOVIC, Danica AGBABA* (*University of Belgrade, Faculty of Pharmacy, Department of Pharmaceutical Chemistry, Vojvode Stepe 450, P. O. Box 146, 11000 Belgrade, Serbia and Montenegro): High-performance thin-layer chromatography determination of some antimycotic imidazole derivatives and preservatives in medicinal creams and a gel. J. Assoc. Off. Anal. Chem. 88, 1544-1548 (2005). TLC of antimycotics (bifonazole, clotrimazole, and miconazole) and preservatives (benzyl alcohol, benzoic acid) on silica gel with 1) ethyl acetate - n-heptane - methanol - diethylamine 30:45:10:2 for bifonazole and benzyl alcohol; 2) n-butyl acetate - n-heptane - methanol - diethylamine 30:45:10:2 for clotrimazole and benzyl alcohol; 3) n-butyl acetate - carbon tetrachloride - methanol - diethylamine 6:12:5:1 for miconazole and benzoic acid. Quantitative determination by reflectance/absorbance measurement at 230 nm (bifonazole, benzyl alcohol, miconazole, and benzoic acid) and at 210 nm (clotrimazole and benzyl alcohol). Recovery rates for all substances ranged from 98.7 to 100.7 %. The limits of detection and quantitation were 0.03 to 0.2 µg and 0.1 to 0.5 µg/spot, respectively.

quality control, densitometry, quantitative analysis 32a

96 083 B. CHAUDHARI, N. PATEL*, P. SHAH (*B.M. Shah College of Pharmacy, Modasa 383315, Gujarat, India): Development and validation of HPTLC method for the estimation of rosuvastatin calcium. Abstract G-28, IPC (2005). HPTLC of rosuvastatin on silica gel with chloroform - methanol - toluene 3:1:1. Quantitative determination by absorbance measurement. The hRf value of rosuvastatin was 53, recovery rate was between 98-102 %, LOD was 8 ng/spot and LOQ 26 ng/spot.

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

96 114 T. LJ. DJAKOVIC-SEKULIC, C. SARBU, Nada U. PERISIC-JANJIC* (*Department of Chemistry, Faculty of Science, University of Novi Sad,Trg D. Obradovica 3, 21000 Novi Sad, Serbia and Montenegro): A comparative study of the lipophilicity of benzimidazole and benztriazole derivatives by RPTLC. J. Planar Chromatogr. 18, 432-436 (2005). TLC of nine benzimidazole and benztriazole derivatives on silica gel impregnated with paraffin oil with methanol - water mixtures. After development the plates were dried and examined under UV light at 254 nm. A quantitative structure-retention relationship (QSRR) correlation study was performed.

pharmaceutical research, qualitative identification 32a, 2c

96 088 G. EDMOND, B. PHILIPPE, M. LIONEL, P. ANGELO* (*Dept. of Clinical Pharmacy, Institut Gustave Roussy, 39, rue Camille Desmoulins, 94800 Villejuif, France): Fluorescence detection combined with either HPLC or HPTLC for pharmaceutical quality control in a hospital chemotherapy production unit: Application to camptothecin derivatives. J. Pharm. Biomed. Anal. 39, 581-586 (2005). For post-production quality control of camptothecin derivatives irinotecan (CPT 11) and topotecan (TPT), HPLC and HPTLC methods have been developed which were suitable for identification, determination of purity and quantification. HPTLC on silica gel with methylene chloride - methanol - formic acid - water 82:24:2:1. After development, the plate was soaked in 15 % paraffin in n-heptane. Quantitative determination by fluorescence measurement at 366/>400 nm. The method was linear within the range of 100-1000 ng/mL for both CPT-11 and TPT. The method was validated for accuracy, precision, LOD, and LOQ.

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

96 092 Anna GUMIENICZEK*, A. BERECKA, H. HOPKALA (*Department of Medicinal Chemistry, Medical University of Lublin, Chodzki Str. 6, 20-093 Lublin, Poland): Quantitative analysis of repaglinide in tablets by reversed-phase thin-layer chromatography with densitometric UV detection. J. Planar Chromatogr. 18, 155-159 (2005). TLC of repaglinide on RP-8 with acetonitrile - phosphate buffer pH 6.0 3:2. Quantitative determination by absorbance measurement at 225 nm. Calibration in the range of 0.6-3.6 µg was linear with a good correlation coefficient (r = 0.998 +/- 0.001). Limits of quantitation and detection were 0.27 µg and 0.08 µg, respectively.

quality control, densitometry, quantitative analysis 32a

96 096 C.Q. HU (Hu Chang-Qin)*, W.B. ZOU (Zou Wen-Buo), W.S. HU (Hu Wang Sheng), X.K. MA (Ma Xiao-Kang), M.Z. YANG (Yang Min-Zhi), S.L. ZHOU (Zhou Shi-Lin), J.F. SHENG (Sheng Jin-Fang), S.H. CHENG (Cheng Shuang-Hong), J. XUE (Xue Jing) (*National Institute for the Control of Pharmaceutical and Biological Products, Beijing 100050, China): Establishment of a fast chemical identification system for screening of counterfeit drugs of macrolide antibiotics. J. Pharm. Biomed. Anal. 40, 68-74 (2006). Two TLC methods have been developed for the screening of fake (counterfeit) drugs of macrolide antibiotics. TLC on silica gel with ethyl acetate - n-hexane - ammonia 20:3:3 for 14 membered macrolides and trichloromethane - methanol - ammonia 100:5:1 for 16 membered macrolides. Detection by spraying with $KMnO_4$ solution. Different chromatographic conditions were standardized and results of color reactions and TLC were correlated to judge the counterfeiting. The method was evaluated in five different laboratories in China.

pharmaceutical research, quality control, qualitative identification 32a

96 134 Y. S. JAISWAL, G. S. TALELE*, S. J. SURANA (*Department of Pharmaceutical Chemistry, R. C. Patel College of Pharmacy, Karwand Naka, Shirpur Dhule 425 405, Maharashtra, India): A simple and sensitive HPTLC method for quantitative analysis of ethamsylate in tablets. J. Planar Chromatogr. 18, 380-383 (2005). HPTLC of ethamsylate in tablets on silica gel (pre-washed

with methanol) at 25 +/-2 °C with chloroform - methanol -acetic acid 10:5:1 in a pre-saturated twin-trough chamber. Quantitative determination by absorbance measurement at 300 nm. The validated calibration range was 500-2500 ng per spot (r = 0.997).

quality control, HPTLC, quantitative analysis 32a

96 098 B. JEROME, S. THOMAS, Isabelle LAVILLE, L. MERCIER, O. OBERLIN, V. GILLES, B. PHILIPPE, P. ANGELO* (*Dept. of Clinical Pharmacy, Institut Gustave Roussy, 39, rue Camille Desmoulins, F-94800 Villejuif, France): Quality control and stability study using HPTLC: applications to cyclophosphamide in various pharmaceutical products. J. Pharm. Biomed. Anal. 38, 180-185 (2005). A stability indicating HPTLC method is reported for estimation of cyclophosphamide in pharmaceutical preparations. HPTLC on silica gel with dichloromethane - methanol - acetic acid 97:3:2. Detection with alcoholic phosphomolybdic acid solution followed by heating at 190 °C for 10 min. Quantitative determination by absorbance measurement at 700 nm. Linearity was obtained between 0.40-1.20 mg/mL with recovery rates of 99.5 %. The method was validated for selectivity, specificity, accuracy, precision, and found to be stable for 28-70 days.

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

96 101 V. KADAKIA*, M. RAVAL, S. MISHRA (*Dept. of Pharmacognosy and Phytochemistry, APMC College of Pharmaceutical Education & Research, Himatnagar, Gujarat 383001, India): HPTLC method for simultaneous estimation of andrographolide and wedelolactone in marketed formulations. Abstract CP-53, IPC (2005). HPTLC of andrographolide and wedelolactones in several market samples on silica gel with toluene - acetone - formic acid 9:6:1. Quantitative determination by absorbance measurement at 254 nm. The hRf value of andrographolide was 52 and of wedelolactone 58. Linearity was obtained between 200-400 ng/spot and 120-200 ng/spot respectively with recovery rates of 98.1-106.7 %. A complex coumarin from Eclipta alba was used as marker.

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

96 102 R. KAKDE*, V. KACHROO, P. INGALKAR (*Department of Pharmaceutical Sciences, Nagpur Univ., Nagpur 440 010, Maharashtra, India): Simultaneous estimation of pantoprazole and mosapride in their pharmaceutical preparations by HPTLC. Abstract G-9, IPC (2005). HPTLC of pantoprazole and mosapride in combined dosage form on silica gel with methanol - toluene - chloroform 4:30:15. Quantitative determination by absorbance measurement at 305 nm. The hRf value of pantoprazole was 31 and of mosapride 43, recovery was 99.9-101.1 %. Accuracy, precision, and linearity of the method were established.

quality control, HPTLC, densitometry, quantitative analysis, pantoprazole, mosapride 32a

96 104 M. KHAKPOUR, A. JAMSHIDI*, A.A. ENTEZAMI, H. MIRZADEH (*Department of Novel Drug Delivery Systems, Iran Polymer and Petrochemical Institute, P. O. Box 14185/458, Tehran, Iran): HPTLC procedure for determination of levonorgestrel in the drug-release media of an in-situ-forming delivery system. J. Planar Chromatogr. 18, 326-329 (2005). HPTLC of levonorgestrel on silica gel (prewashed with chloroform - methanol 1:1 and once with the mobile phase, dried and activated at 100 °C for 15 min) with toluene - 2-propanol 9:1 in an automatic multiple development chamber without chamber saturation. Visual examination under UV light at 254 nm; quantitation by densitometry at 250 nm.

quality control, HPTLC, quantitative analysis, densitometry, AMD 32a

96 105 L. KOMSTA, Genowefa MISZTAL* (*Medical University, Department of Medicinal Chemistry, 6 Chodzki, 20-093 Lublin, Poland): Determination of fenofibrate and gemfibrozil in pharmaceuticals by densitometry and videodensitometric thin-layer chromatography. J. Assoc. Off. Anal. Chem. 88, 1517-1524 (2005). HPTLC of fenofibrate and gemfibrozil on diol phases in horizontal chambers using the sandwich technique with hexane - tetrahydrofuran 4:1. Quantitative determination by classical densitometry at 227 nm and videodensitometry at 254 nm. Recovery in the densitometric assay was 101.4 % for fenofibrate and 100.5 % for gemfibrozil. Videodensitometry resulted in recoveries of 102.7 % and 98.8 %, respectively.

quality control, quantitative analysis, densitometry, HPTLC 32a

96 106 Dorota KOWALCZUK (Medical University of Lublin, Faculty of Pharmacy, Department of Medicinal Chemistry, 6 Chodzki Str., 20-093 Lublin, Poland): Simultaneous high-performance thin-layer chromatography - Densitometric assay of trandolapril and verapamil in the combination preparation. J. Assoc. Off. Anal. Chem. 88, 1525-1529 (2005). HPTLC of trandolapril and verapamil in 2-component mixtures and in their combination capsules on silica gel in horizontal chambers with ethyl acetate - ethanol - acetic acid 16:4:1. Quantitative determination by densitometric measurement at 215 nm. Detection and quantitation limits were found to be 1.25 and 3.75 µg/spot for TRA and 0.15 and 0.45 µg/spot for VER, respectively.

quality control, quantitative analysis, densitometry, HPTLC 32a

96 107 K. KRESTA*, P. KASTNER, J. KLIMES, V. KLIMESOVA (*Charles University in Prague, Faculty of Pharmacy in Hradec Králové, Heyrovského 1203, Hradec Králové 500 05, Czech Republic): Reversed-phase thin-layer chromatographic determination of the lipophilicity of potential antituberculotic compounds. J. Planar Chromatogr. 18, 450-454 (2005). TLC of twenty-seven 2-benzylsulfanybenzothiazole derivatives on silanized silica gel in a pre-equilibrated normal chamber with 0.05 M phosphate buffer (pH 7.4 or 3.0) and methanol as organic modifier. Detection under UV light at 254 nm.

pharmaceutical research, qualitative identification 32a, 2d

96 108 J. KRZEK*, A. KWIECIEN (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Kraków, Poland): Application of densitometry for determination of beta-adrenergic-blocking agents in pharmaceutical preparations. J. Planar Chromatogr. 18, 308-313 (2005). HPTLC of beta-adrenergic-blocking agents (acebutolol, atenolol, betaxolol, bisoprolol, labetalol, metoprolol, oyprenolol, pindolol, propanolol, sotalol, timolol) on silica gel with chloroform - methanol - ammonia 75:35:1. Quantitative determination by absorbance measurement at 270 nm for atenolol, at 240 nm for acebutolol, at 289 nm for propanolol, and at 220 nm for bisoprolol. The limits of detection and determination ranged from 30 to 400 ng and recovery was from 97.1 to 102.2 %.

quality control, HPTLC, quantitative analysis, densitometry 32a

96 109 J. KRZEK*, A. MASLANKA, P. LIPNER (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, 9 Medyczna St, 30688 Cracow, Poland): Identification and quantitation of polymyxin B, framycetin, and dexamethasone in an ointment by using thin-layer chromatography with densitometry. J. Assoc. Off. Anal. Chem. 88, 1549-1554 (2005). TLC of polymyxin B, framycetin, and dexamethasone on silica gel with methanol and methanol - n-butanol - 25 % ammonia - chloroform 14:4:9:12 for framycetin and polymyxin B. Quantitative determination by densitometry at 550 nm after detection with 0.3 % ninhydrin solution. Dexamethasone was separated with cyclohexane - ethyl acetate 2:3, quantitative determination by absorbance measurement at 245 nm. Similar accuracy, relative standard deviation values from 1.49 to 2.47 % and relative error values from 0.02 to 0.81 % are comparable to those

obtained with the reference methods.

quality control, densitometry, quantitative analysis 32a

96 110 J. KRZEK*, U. HUBICKA, J. SZCZEPANCZYK (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, Medyczna 9, 30-688 Krakow, Poland): High-performance thin-layer chromatography with densitometry for the determination of ciprofloxacin and impurities in drugs. J. Assoc. Off. Anal. Chem. 88, 1530-1535 (2005). HPTLC of ciprofloxacin and degradation products (an ethylenediamine compound, a desfluoro compound, a by-compound, and fluoroquinolonic acid) on silica gel with chloroform - methanol - 25 % ammonia 43:43:14. Quantitative determination by densitometric analysis at 330 nm for fluoroquinolonic acid and at 277 nm for the other compounds. The method showed high sensitivity (limit of detection 10 to 44 ng), a wide linearity range (3 to 20 µg/mL), and good precision (2.32 to 6.46 % relative standard deviation) and accuracy (recovery rates 98.6 to 101.5 %) for individual constituents.

quality control, densitometry, quantitative analysis, HPTLC 32a

96 085 B. D. MALI, D. S. RATHOD, M. V. GARAD* (*Regional Forensic Science Laboratory, State of Maharashtra, Cantonment, Aurangabad-431 002, India): Thin-layer chromatographic determination of diazepam, phenobarbitone, and saccharin in toddy sample. J. Planar Chromatogr. 18, 330-332 (2005). TLC of diazepam, phenobarbitone, chloral hydrate, copper sulfate, sulfadiazine, and saccharin on silica gel in a presaturated chamber with chloroform - acetic acid 9:1, n-hexane - acetone - methanol 16:6:1 and n-hexane - acetone - butanol 24:16:1. Detection by treatment with chlorine followed by spraying with o-toluidine reagent and 1 % phosphomolybdic acid.

food analysis, qualitative identification 32a

96 118 S. MARIHAL*, V. MARDANE, C. PATIL (*Department of Pharmaceutical Analysis, Goa College of Pharmacy, 18th June Road, Panaji 408001, Goa, India): HPTLC method for quantitative estimation of corticosterone in rat plasma. Abstract G-19, IPC (2005). HPTLC for estimation of corticosterone in rat plasma on silica gel with chloroform - methanol - water 9:10:1. Quantitative determination by absorbance measurement at 245 nm. Betamethasone was employed as internal standard. The extraction of plasma with ethyl acetate gave an average recovery of >85 %. The linearity was within the range of 30-300 ng/mL with LOQ being 30 ng. The method was found to be rugged and robust.

pharmaceutical research, clinical chemistry research, clinical routine analysis, HPTLC, densitometry, corticosterone 32a, 13

96 120 A. MASLANKA, J. KRZEK* (*Jagiellonian University, Collegium Medicum, Department of Inorganic and Analytical Chemistry, 9 Medyczna St, 30-688 Krakow, Poland): Densitometric high performance thin-layer chromatography - Identification and quantitative analysis of psychotropic drugs. J. Assoc. Off. Anal. Chem. 88, 70-79 (2005). HPTLC of haloperidol, amitriptyline, sulpiride, promazine, fluphenazine, doxepin, diazepam, trifluoperazine, clonazepam, and chlorpromazine in 25 selected psychotropic drugs on silica gel with 30 mobile phases, eight of them were selected based on spot location and developing time. Identification and quantification were carried out based on UV densitometric measurements. In addition to retention factors, the absorption spectra recorded directly from chromatograms were also used in qualitative analysis. Limit of detection ranged from 0.009 to 0.260 µg depending on the wavelength used. A satisfying recovery, ranging from 92.9 to 104.7 %, was achieved for individual constituents.

quality control, densitometry, quantitative analysis, HPTLC 32a

96 121 S. MEYYANATHAN*, N. KRISHNAVENI, R. GOPINATH, B. SURESH (*Dept. of Pharmaceutical Analysis, J.S.S. College of Pharmacy, Ootacamund 643001, Tamil Nadu, India): HPTLC method for simultaneous estimation of nimesulide and chlorzoxazone in their formulations. Abstract GP-18, IPC (2005). HPTLC of nimesulide and chlorzoxazone in tablets on silica gel (prewashed with methanol) with toluene - acetone - ammonia 50:50:4. Paracetamol was used as internal standard. Quantitative determination by absorbance measurement at 265 nm. Nimesulide, chlorzoxazone and paracetamol showed hRf values of 80, 73 and 42, respectively. Linearity was obtained between 0.2-1.0 mg/mL with recovery rates of 99.6-100.3 % for both compounds. The method was validated for accuracy, precision, linearity, LOD, and LOQ.

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

96 122 Genowefa MISZTAL*, L. KOMSTA (*Department of Medicinal Chemistry, Medical University, 6 Chodzki, 20-093 Lublin, Poland): Determination of bezafibrate and ciprofibrate in pharmaceutical formulations by densitometric and videodensitometric TLC. J. Planar Chromatogr. 18, 188-193 (2005). HPTLC of of bezafibrate and ciprofibrate in tablets and capsules on diol phases with hexane - tetrahydrofuran 4:1. Quantitative determination by absorbance measurement at 227 nm and videoscanning at 254 nm. Recovery measured by use of densitometry was 100.3 % (RSD 7.8 %) for bezafibrate and 98.0 % (RSD 6.1 %) for ciprofibrate. Videodensitometry resulted in recovery of 96.2 % (RSD 9.8 %) and 97.8 % (RSD 11.2 %), respectively.

quality control, densitometry, quantitative analysis, HPTLC 32a

96 123 S. MUKHERJEE, P. LOYA, P. BIRAJDAR, M. SARAF* (*Bombay College of Pharmacy, Kalina, Santacruz (E), Mumbai 400098, India): Rapid and sensitive method for the determination of epalrestat in human plasma by HPTLC. Abstract G-33, IPC (2005). HPTLC of epalrestat in plasma on silica gel with ethyl acetate - toluene - acetic acid 30:20:1. Nitrofuranloin was used as internal standard. Quantitative determination by absorbance measurement at 390 nm. Linearity was obtained the range of 0.01-0.20 µg/mL with recovery of 99-107 %. The method was validated as per ICH guidelines.

pharmaceutical research, clinical chemistry research, clinical routine analysis, HPTLC, densitometry, quantitative analysis, epalrestat 32a

96 124 Neha NATH, S. ANSARI*, M. NAWAZISH (*Department of Pharmacognosy & Phytochemistry, Faculty of Pharmacy, Jamia Hamdard 110062, New Delhi, India): Quality standard studies on the roots of Ratanjot - Arnebia nobilis (Reichb) - A controversial ayurvedic drug. The Pharma Review, Dec, 106-108 (2005). HPTLC of hexane, petroleum ether, chloroform, and methanol extracts of Arnebia nobilis (Ratanjot) roots on silica gel with n-hexane - methanol 9:1 and toluene - chloroform - methanol 14:5:1. Detection by spraying with anisaldehyde sulphuric acid reagent and densitometric fingerprint analysis by absorbance measurement at 366 nm. HPTLC fingerprinting profile provided the most reliable method for correct identification of the root.

herbal, HPTLC, densitometry, postchromatographic derivatization, qualitative identification 32a

96 126 Jolanta OBNISKA*, K. KAMINSKI (*Department of Pharmaceutical Chemistry, Jagiellonian University Medical College, Medyczna 9, 30-688 Krakow, Poland): Relationships between the lipophilicity and anticonvulsant activity of N-benzyl-2-azaspiro[4.4]nonane- and [4.5]decane-1,3-dione derivatives. J. Planar Chromatogr. 18, 240-243 (2005). TLC of twenty-one N-benzyl-2-azaspiro[4.4]nonane- and [4.5]decane-1,3-dione derivatives on RP-18 with a mixture of n-propanol and TRIS buffer (pH 7.0) in a chamber saturated for 30 min. Detection under UV light at 254 nm. Examination of chromatographic behavior revealed a linear correlation between RM values and the concentration of n-propanol in the mobile phase.

pharmaceutical research, qualitative identification 32a, 2c

96 127 Jolanta OBNISKA*, K. KAMINSKI (*Department of Pharmaceutical Chemistry, Jagiellonian University, Medical College, Medyczna 9, 30-688 Kraków, Poland): RPTLC determination of the lipophilicity of some new N-[(4-arylpiperazin-1-yl)alkyl] spirosuccinimides. J. Planar Chromatogr. 18, 384-387 (2005). TLC of thirty-seven N-[(4-arylpiperazin-1-yl)alkyl]-2-azaspiro[4.4]nonane- and [4.5]decane-1,3-dione derivatives on RP-18 in a pre-saturated chamber with n-propanol - Tris buffer (pH 7.0) mixtures. Detection under UV at 254 nm.

pharmaceutical research, qualitative identification 32a, 2c

96 128 J.V. ODOVIC, B.B. STOJIMIROVIC, Mirjana B. ALEKSIC*, D. M. MILOJKOVIC-OPSENICA, Z.L. TESIC (*Faculty of Pharmacy, University of Belgrade, P. O. Box 146, 11001 Belgrade, Serbia and Montenegro): Examination of the hydrophobicity of ACE inhibitors and their active metabolites by salting-out thin-layer chromatography. J. Planar Chromatogr. 18, 98-103 (2005). Salting-out TLC (SO TLC) of five ACE inhibitors and their active degradation products (enalapril, enalaprilat, quinapril, quinaprilat, fosinopril, fosinoprilat, lisinopril, cilazapril, cilazaprilat) on silica gel, cellulose, and polyacrylonitrile with aqueous ammonium sulfate solutions of different concentrations. Increasing the salt concentration in the mobile phase led to increased RM values for all substances. For comparison TLC on RP-18 with methanol - water. Detection by exposure to iodine vapor.

clinical chemistry research, qualitative identification 32a

96 129 M. PAI*, S. KAPADE, DR. KALPANA PATIL (*Departmental of Pharmaceutical Analysis, Goa College of Pharmacy, 18th June Road, Panaji 408001, Goa, India): Development and validation of a new sensitive method for the quantitative estimation of valdecoxib in tablets and determination of its extraction efficiency in human plasma by using HPTLC. Abstract G-29, IPC (2005). HPTLC of valdecoxib in tablets and human plasma on silica gel with methanol - water - chloroform 6:3:1. Celecoxib was used as internal standard. Quantitative determination by absorbance measurement at 254 nm. The compound was extracted from plasma with ethyl acetate showing an extraction yield of 85 %. Linearity was obtained in the range of 25-200 ng/mL, recovery rate was 99.8 % from tablets and 97.2 % from plasma.

pharmaceutical research, quality control, clinical routine analysis, HPTLC, densitometry, quantitative analysis, valdecoxib 32a

96 130 B. PATEL, K. PATEL, A. SALUJA* (*Dept. of Pharmacognosy & Phytochemistry, A.R. College of Pharmacy, Vidhyanagar, Gujarat 388120, India): HPTLC method development for estimation of stigmasterol in leptadenia reticulata. Abstract CP-31, IPC (2005). HPTLC of stigmasterol in leptadernia reticulata on silica gel with n-hexane - ethyl acetate 4:1. Quantitative determination by absorbance measurement at 525 nm after derivatization. Both hydrolyzed and unhydrolyzed samples (2N HCl) were analyzed. Unhydrolyzed samples were found to contain a higher amount of stigmasterol. Linearity was in the range of 0.16-0.48 mg/mL. Several market samples were analyzed by the proposed method.

herbal, HPTLC, densitometry, quantitative analysis, stigmasterol, leptadenia reticulata 32a

96 132 Alina PYKA*, W. KLIMCZOK (*Department of Analytical Chemistry, Faculty of Pharmacy, Silesian Academy of Medicine, 4 Jagiellonska Street, 41200 Sosnowiec, Poland): Study of lipophilicity and application of selected structural descriptors in QSAR analysis of nicotinic acid derivatives. Investigations on RP 18 WF254 plates. Part II. J. Planar Chromatogr. 18, 300-304 (2005). HPTLC of nicotinic acid, methyl nicotinate, ethyl nicotinate, isopropyl nicotinate, butyl nicotinate, hexyl nicotinate, benzyl nicotinate, and N-methylnicotinamide on RP-18 W after presaturation of the chamber with methanol - water in different volume proportions. Visualizati-

on under UV light at 254 nm.

pharmaceutical research, qualitative identification, HPTLC 32a

96 148 R. T. SANE, S. S. KAMAT, S. N. MENON, S. R. INAMDAR*, M. R. MOTE (*TDM Laboratories, Plot No. 194, Scheme No. 6, Road No. 15, Sion (E), Koliwada, Mumbai 400 022, India): Determination of rosuvastatin calcium in its bulk drug and pharmaceutical preparations by high-performance thin-layer chromatography. J. Planar Chromatogr. 18, 194-198 (2005). HPTLC of rosuvastatin calcium (with aceclofenac as internal standard) on silica gel with toluene - methanol - ethyl acetate - formic acid 60:10:30:1. Quantitative determination by absorbance measurement at 265 nm. A good determination coefficient (r2 = 0.9999) was obtained for the linearity in the range of 1.0 to 15.0 µg of sample. For formulation and bulk drug the mean percentage assay was 100.09 +/- 0.20 and 100.07 +/- 0.48, respectively. The accuracy of the method was found to be 100.62 % and precision was found to vary from 0.01 to 0.77 %.

quality control, HPTLC, densitometry, quantitative analysis 32a

96 137 N. SHAH, B. SUHAGIA, R. SHAH, C. SHAH* (*B.M. Shah College of Pharm. Edu. Res., Modasa 383315, Gujarat, India): Development and validation of a HPTLC method for the estimation of telmisartan and hydrochlorothiazide in bulk and tablets. Abstract G-5, IPC (2005). HPTLC of telmisartan and hydrochlorothiazide on silica gel with chloroform - methanol - toluene 2:5:5 with chamber saturation for 30 min. Detection by spraying with ninhydrin reagent. Quantitative determination by absorbance measurement at 272 nm. The linearity was in the range of 250-500 ng/spot for telmisartan and 200-700 ng/spot for hydrochlorothiazide, recovery was 99-101 %. Accuracy, precision and linearity of the method were established.

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

96 138 N. SHAH, S. SHAH*, V. PATEL, N. PATEL (*B.M. Shah College of Pharmacy, Modasa 383315, Gujarat, India): Development and validation of a HPTLC method for the estimation of cefuroxime axetil. Abstract G-25, IPC (2005). HPTLC of cefuroxime axetil in bulk and tablets on silica gel with chloroform - methanol - toluene 2:1:1 with chamber saturation for 30 min. Quantitative determination by absorbance measurement at 290 nm. The linearity was within the range of 300-900 ng/spot with an average recovery rate of 99.4 %. The method was validated as per ICH guidelines.

pharmaceutical research, quality control, HPTLC, densitometry, quantitative analysis, cefuroxi

96 142 A. SOCKALINGAM*, Indumathy NARAYANAREDDY, P. SHANMUGAPANDIYAN, S. SRIDHAR (*Dept. of Pharmaceutical Analysis and Chemistry, C.L.Baid Metha College of Pharmacy, Old Mahabalipuram Road, Jyothi Nagar, Thorapakkam, Chennai 600096, India): Simultaneous quantification of stavudine, lamivudine and nevirapine by UV spectroscopy, reverse phase HPLC and HPTLC in tablets. J. Pharm. Biomed. Anal. 39, 801-804 (2005). HPTLC on silica gel with chloroform - methanol 9:1. Quantitative determination by absorbance measurement at 265 nm. hRf values of stavudine (SV), lamivudine (LV) and nevirapine (NV) were 25, 67 and 87 respectively. The method was linear within the range of 0.01-0.06 mg/mL, 0.05-0.30 mg/mL and 0.06-0.40 mg/mL for SV, LV, and NV respectively, recovery rates were between 98.2 and 99.9 %. The HPTLC method was compared with the UV and HPLC methods. Accuracy, precision and ruggedness of the method were established.

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

96 143 B. SPANGENBERG*, A. SEIGEL, J. KEMPF, W. WEINMANN (*University of Applied Sciences Offenburg, Badstraße 24, 77652 Offenburg, Germany): Forensic drug analysis by means of diode-array HPTLC using Rf and UV library search. J. Planar Chromatogr. 18, 336-343 (2005). HPTLC of thirty-three compounds with benzodiazepine properties on silica gel after prewashing first with methanol and then with dichloromethane - methanol 19:1 in a saturated horizontal chamber and three optimized mobile phases: dichloromethane - methanol 19:1, ethyl acetate - cyclohexane - 25 % ammonia 50:40:0.1, and in a third run cyclohexane - acetone - methyl t-butyl ether 3:2:1. Diode-array HPTLC makes it possible to identify all the compounds with high certainty down to a level of 20 ng. An algorithm for spectral recognition which is combined with Rf values from the three separation steps into one fit factor is presented. This set of data is unique for each of the compounds investigated and enables unequivocal identification.

HPTLC, qualitative identification 32a, 3f

96 144 K. SRINIVAS*, C. NAVEEN KUMAR, Prya Susan VARGHESE, M. E. BHANOJI RAO (*Sri Venkateshwara College of Pharmacy, Srikakulam, Andhra Pradesh 532001, India): HPTLC method for quantitative determination and fingerprinting of isoleucin in trigonella foenum graecum. Abstract D-12, IPC (2005). HPTLC of methanol and ethyl acetate extracts of trigonella foenum graecum on silica gel with n-propanol - ammonia 11:9. Detection by spraying with ninhydrin reagent. Quantitative determination by absorbance measurement and in visible range. The hRf value of isoleucin was 60. Methanolic extracts contained 0.17 % isoleucin and ethyl acetate extracts 0.008 %. Accuracy, precision, linearity of the method were established.

traditional medicine, herbal, HPTLC, densitometry, quantitative analysis, postchromatographic derivatization, isoleucin 32a

96 145 S.P. SUBRAMANIYAN, S.K. DAS* (*Government of India, Department of Biochemistry, Central Drugs Laboratory, 3, Kyd St, Kolkata 700 016, India): Rapid identification and quantification of chlorpheniramine maleate or pheniramine maleate in pharmaceutical preparations by thin-layer chromatography-densitometry. J. Assoc. Off. Anal. Chem. 87, 1319-1322 (2004). TLC of chlorpheniramine and pheniramine maleate in combination with other drugs in pharmaceutical preparations of tablets, syrups, eye and ear drops etc. on silica gel with cyclohexane - chloroform - methanol - diethylamine 9:8:1:2. Detection under UV light at 254 nm and quantitative determination by scanning at 260 nm. Recoveries of CPM and PM were 100.1 +/- 0.8 % and 100.1 +/- 0.9 %, respectively.

quality control, densitometry, quantitative analysis 32a

96 015 C. SULLIVAN et al., see section 2a

96 146 E. SUMARLIK, H. TAMPUBOLON, M. YUWONO, G. INDRAYANTO* (*Assessment Service Unit, Faculty of Pharmacy, Airlangga University, Jl. Dharmawangsa dalam, Surabaya 60286, Indonesia): Densitometric determination of desloratadine in tablets, and validation of the method. J. Planar Chromatogr. 18, 19-22 (2005). TLC of desloratadine (8-chloro-6,11-dihydro-11-(4-piperidinylidene)-5H-benzo[5,6]cyclohepta[1,2-b]pyridine) on silica gel with ethyl acetate - n-butanol - 25% ammonia - methanol 21:5:4:5. Quantitative determination by absorbance/reflectance measurement at 279 nm. Peak area was linearly dependent on the amount of desloratadine within the range of 1500 to 5000 ng/spot. The relative process standard deviation was 1.78 %.

quality control, densitometry, quantitative analysis 32a

96 150 M. TATARCZAK, Jolanta FLIEGER*, H. SZUMILO (*Medical University of Lublin, Faculty of Pharmacy, Department of Inorganic and Analytical Chemistry, Staszica 6, 20-081 Lublin, Poland): Simultaneous densitometric determination of rifampicin and isoniazid by high-perfor-

mance thin-layer chromatography. J. Planar Chromatogr. 18, 207-211 (2005). HPTLC of rifampicin and isoniazid on silica gel (prewashed with methanol) in a horizontal chamber with ethyl acetate - methanol - acetone - acetic acid 5:2:2:1 after pre-saturation for 30 min. Densitometric evaluation by absorbance measurement at 345 nm for rifampicin and at 270 nm for isoniazid. For isoniazid and rifampicin CV was 0.42 and 0.16 %, relative standard error 0.01 and 0.13 %, and recovery 98.9 and 102.5 %, respectively.

quality control, HPTLC, densitometry, quantitative analysis 32a

96 151 T. TUZIMSKI*, K. SZTANKE (*Department of Physical Chemistry, Faculty of Pharmacy, Medical University, Staszica 6, 20-081 Lublin, Poland): Retention data for some carbonyl derivatives of imidazo[2,1-c][1,2,4]triazine in reversed-phase systems in TLC and HPLC and their use for determination of lipophilicity. Part 1. Lipophilicity of 8-aryl-3-phenyl-6,7-dihydro-4H-imidazo[2,1-c][1,2,4]triazin-4-ones. J. Planar Chromatogr. 18, 274-281 (2005). Determination of the lipophilicity of 13 carbonyl derivatives of imidazo[1,2-c][1,2,4]triazine by TLC on RP-18 and RP-18 W in horizontal chambers with aqueous mobile phases containing organic modifiers (methanol or dioxane). Detection under UV light at 254 and 366 nm.

lipophilicity 32a

96 153 J. VERMA*, A. JOSHI (*Department of Chemistry, K.J. Somaiya College of Science & Commerce, Vidyavihar, Mumbai 400077, India) : Simultaneous estimation of alprazolam and sertraline in tablet dosage form by HPTLC. Indian Drugs 42 (12), 805-807 (2005). HPTLC of alprazolam and sertraline in tablet dosage form on silica gel with toluene - ethyl acetate - methanol - acetic acid 90:30:20:3 without saturation. Quantitative determination by absorbance measurement at 217 nm. Accuracy, precision, and linearity were established. The linearity range was 20-100 ng for alprazolam and 100-500 ng for sertraline. Recovery rates were between 99.8-100.5 % for both drugs.

pharmaceutical research, HPTLC, quantitative analysis, densitometry, alprazolam, sertraline 32a

96 084 P. CHEN (Chen Ping)*, Y. ZHU (Zhu Yinglong), Q. WEI (Wei Qiang), B. YAN (Yan Bianjie), (*Shangxi Acad. TCM, Xian, Shanxi 710003, China): (Study of the quality standard for Zhike Pingchuan capsules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (3), 275-278 (2005). TLC of the extracts on silica gel with 1) chloroform - ethyl acetate - methanol - water 15:40:22:10; 2) n-butanol - ammonia - ethanol 5:2:1; 3) the lower phase of chloroform - ethyl acetate - methanol - water 8:8:3:2. Detection 1) by spraying with 10 % H_2SO_4 in ethanol followed by heating at 105 ºC until the spots appear; 2) by spraying with 5 % potassium iodobismuthate solution. Identification by fingerprint technique. Quantification of ginsenoside Rb1 by HPLC.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, ginsenoside Rb1 32c

96 089 Y. FU (Fu Yue)*, Q. CHEN (Chen Qingtang), X. DONG (Dong Xun) (*Natural Drug Inst., Yunnan Baiyao Group Co., Ltd., Qunming, Yunnan 650032, China): (Study of the quality analysis of Lidanzhitong tablets) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (4), 410-414 (2005). TLC of the extracts of the title Chinese traditional patent medicine on silica gel with 1) chloroform - ethyl acetate - methanol - formic acid 400:25:50:1 2) the lower phase of chloroform - methanol - water 32:17:5; 3) n-hexane - chloroform - water 15:8:2. Detection 1) by spraying with 5 % vanillin - H_2SO_4 solution and heating mildly until the spots are visualized; 2) by spraying with 2 % AlCl3 in methanol and inspection under UV 365 nm; 3) by exposing to iodine vapor. Identification by fingerprint techniques. Quantification of paeoniflorin by HPLC.

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

96 094 CH. GUO (Guo Changqiang)*, LI XU (Xu Ligui), X. YAN (Yan Xuesheng), T. TU (Tu Tao), ZH. YU (Yu Zhongyuan) (*Shangdong Provin. Inst. TCM, Shandong, Jinan 250014, China): (Study of the quality standard for Biaoshi Ganmao granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27(5), 538-541 (2005). TLC of Biaoshi Ganmao granule extracts on silica gel developed with 1) chloroform - methanol - water 28:10:1, 2) chloroform - methanol - ammonia 40:10:1, 3) n-hexane - diethyl ether - glacial acetic acid 50:50:1, 4) ethyl acetate - methanol - water 100:17:13, 5) toluene - ethyl acetate - formic acid 20:10:1. Detection 1) under UV 365 nm, 2) by spraying with 2 % ninhydrin solution followed by heating at 105 ºC until the spots are visualized, 3) under UV 254 nm, 4) by spraying with $AlCl_3$ solution. Identification by fingerprint technique. Quantification of puerarin by HPLC. The results for four batches of real life sample are given.

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

96 111 Y. LI (Li Yuhong)*, G. ZHU (Zhu Guoqiang), Y. WU (Wu Yuxin), G. LI (Li Ge) (*People's Hosp., Xinjiang Region, Urumuqi, Xinjiang 830001, China): (The quality standard for compound Xuelian capsules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27(5), 526-529 (2005). TLC of the extracts on silica gel with 1) ethyl acetate - formic acid - water 10:1:2; 2) cyclohexane - chloroform - methanol 10:6:1. Detection 1) by spraying with 10 % H_2SO_4 in ethanol followed by heating at 105 ºC until the spots are visualized; 2) by exposing to iodine vapor and inspection under UV 365 nm. Identification by fingerprint technique. Monitoring of the dosage limit of aconitine in the medicine by comparison with the standard. Quantification of tetrahydropalmatine by HPLC. The results are given for 10 batches of real life samples.

herbal, pharmaceutical research, traditional medicine, quality control, qualitative identification, HPTLC, aconitine, D1-tetrahydropalmatine 32c

96 112 J. LIU (Liu Junkang)*, X. XU (Xu Xinyan), D. LIU (Liu Diwei), Y. SU (Su Yali), X. TAO (Tao Xiyuan), (*Xinjiang Aoton. Region Inst. Drog Cont., Wulumuqi Xinjiang 830002, China): (Study of the quality standard for Haolan Ganmao granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (3), 279-282 (2005). TLC of the extracts of the title Chinese traditional patent medicine on silica gel with 1) ethyl acetate - methanol - ammonia 17:2:1; 2) n-butanol - glacial acetic acid - water 19:5:5. Detection 1) under UV 365 nm; 2) by spraying with ninhydrin solution followed by heating at 105 ºC until the spots are visualized. Identification by fingerprint technique. Quantification of paracetamol and pseudoephedrine arginine by HPLC.

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

96 113 Y. LIU (Liu Yanju)*, SH. LI (Li Shuiqing), X. LU (Lu Xizhen) (*Hubei Coll. TCM, Wuhan, Hubei 430061, China): (Determination of stachydrine chloride in Fugong capsules by thin-layer chromatography) (Chinese) Chinese J. Hosp. Pharm. (Zhongguo Yiyuan Yaoxue Zazhi) 25 (9), 894-896 (2005). TLC of the extracts on silica gel plates with acetone - ethanol - hydrochloric acid 10:6:1. Detection by spraying with potassium iodobismuthate solution. Identification by comparison with the standard. Quantification by densitometry at 510 nm. Validation of the method by investigation of linearity range (2.2 µg - 10.8 µg, r = 0.9994); precision (RSD = 1.05 %, n = 5); reproducibility of five time assay towards the same sample (RSD = 0.31 %); and standard addition recovery (98.1 %, RSD = 2.15 %, n = 5). The results for five real life samples are given.

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

96 115 X. LU (Lu Xinyan)*, H. ZHAO (Zhao Huaiqing), CH. ZHAO (Zhang Chao), SH. TANG (Tang Shuhan) (*Sch. Pharm., Shenyang Univ. Pharm., Shenyang, Liaoning 110016, China): (Separation of diosgenin in Trigonella foenum-graecum L. and its compound preparations by thin-layer chromatography) (Chinese). Chinese J. Chromatogr. (Sepu) 23 (2), 216-217 (2005). TLC on silica gel by 2-fold development with cyclohexane - ethyl acetate 10:1 followed by cyclohexane - ethyl acetate 2:1. Detection by spraying with H_2SO_4 - ethanol 1:10 followed by heating at 105 ºC for 5 min. Visualization under UV 365 nm.

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

96 133 Q. QIN (Qin Qing)*, B. GAO (Gao Baoshuan), (*Hebei Inst. Cont. Med. App. and Drug Package Materials, Shijiazhuang, Hebei 050061, China): (Study of the quality Standard for Compound Songluo granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (3), 272-275 (2005). TLC of the extracts on silica gel with 1) n-hexane - chloroform - methanol 15:8:2; 2) n-hexane - ethyl acetate 9:1; 3) chloroform - benzene - glacial acetic acid 7:2:1. Detection 1) by exposing to iodine vapor; 2) under UV 365 and 254 nm. Identification by comparison with the standard. Quantification of usnic acid by densitometry at 290 nm. Validation by investigating the linearity range (0.50 - 2.50 µg/spot, r = 0.999), precision (RSD = 2.26 %, n= 5, within plate, and 2.97 %, n = 5, plate-to-plate), and standard addition recovery (97.5 %, RSD = 2.61, n = 5).

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

96 139 J. SHI (Shi Juan)*, Y. ZHANG (Zhang Yujie), CH. XUN (Xun Chuanfa), (*Sch. Med., Xian Jiaotong Univ. Xian, Shanxi 710061, China): (Study of the quality standard for Shenguo granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27(5), 535-538 (2005). TLC of the extracts on silica gel with 1) benzene - ethyl acetate - formic acid 15:2:1; 2) n-hexane - ethyl acetate - formic acid 60:20:1; 3) chloroform - methanol - ammonia 40:10:1; 4) chloroform - methanol - water 13:7:2. Detection 1) under UV 365 nm; 2) by spraying with 3 % ninhydrin solution followed by heating at 105 ºC until the spots are visualized; 3) by spraying with 10 % H_2SO_4 solution in ethanol followed by heating until the spots are visualized. Identification by fingerprint technique. Quantification of emodin by densitometry at 445 nm. Validation of the method by investigation of its linearity range (0.1 µg - 1.0 µg, r = 0.998); precision (RSD = 1.05 % n = 6); reproducibility of six time assay towards the same sample (RSD = 1.24 %); and standard addition recovery (96.7 %, RSD = 1.75 %, n = 6). The results for three batches of real life sample are given.

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

96 140 X. SHU (Shu Xiaohua)*, M. ZHOU (Zhou Meijuan), M. DAI (Dai Meihua), L. ZHANG (Zhang Liqun) (*Dep. R & D, Jiangxi Huiren Pharm. Co., Ltd., Jiangxi, Nanchang 330052, China): (Comparison of methods for determination of tanshinone IIA in Huoxue Huayu granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27(4), 483-485 (2005.) TLC of tanshinone IIA extracted from the title Chinese traditional patent medicine on silica gel with benzene - ethyl acetate 19:1. Quantitative determination by densitometry at 470 nm. Also determination of the compound by HPLC.

herbal, pharmaceutical research, traditional medicine, quality control, qualitative identification, densitometry, quantitative analysis, comparison of methods, tanshinone IIA 32c

96 149 F. TANG (Tang Fushan)*, H. JIAO (Jiao Haisheng), W. QIU (Qiu Wen), F. WANG (Wang Faqin) (*No.2 Hosp., Lanzhou Univ., Langzhou, Gansu 730030, China): (Study of the quality standard

for Yinxue tablets) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (3), 385-388 (2005). TLC of the extracts on silica gel with 1) ethyl acetate - acetone - formic acid - water 5:5:1:1; 2) chloroform - ethyl acetate - methanol - water 8:20:11:5; 3) n-hexane - ethyl acetate 9:1. Detection by spraying with 1) 5 % H_2SO_4 and 2) 10 % H_2SO_4, both in ethanol followed by heating at 105 ºC until the spots are visualized; 3) under UV 365 nm. Identification by comparison with the standard. Quantification of geniposide by HPLC. The analysis results for three real life samples are given.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, densitometry, Fructus Gardeniae, Ralix Astragali, Ralix Angelicae Sinensis, geniposide 32c

96 156 Y. ZOU (Zou Yang)*, X. YE (Ye Xiaochuan), G. WANG (Wang Guangzhong), F. DENG (Deng Fen), H. JIAO (Jiao Hexiang) (*Hubei Acad. TCM, Wuhan, Hubei 430074, China): (Development of the analysis method for the quality control of compound Shouwu granules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27 (4), 407-410 (2005). TLC of the extracts on silica gel with 1) benzene - ethanol 2:1 and 4:1; 2) n-butanol - glacial acetic acid - water 7:1:2; 3) chloroform - methanol 9:1. Detection under UV 365 and 254 nm. Identification by fingerprint technique. Quantification of 2, 3, 5, 4'-tetrahydroxystilbene-2-O-beta-D-glucoside by HPLC. The analysis results for a group of real life samples are given.

pharmaceutical research, traditional medicine, quality control, herbal, qualitative identification, 2, 3, 5, 4'-tetrahydroxystilbene-2-O-beta-D-glucoside 32c

96 075 H. AGRAWAL, N. KAUL, A.R. PARADKAR, K. R. MAHADIK* (*Department of Quality Assurance Techniques, Bharati Vidyapeeth Deemed University, Poona College of Pharmacy, Erandwane, Pune 411038, Maharashtra, India, krmahadik@rediffmail.com): Standardization of crude extract of neem seed kernels (Azadirachta indica A. Juss) and commercial neem based formulations using HPTLC and extended length packed-columns SFC method. Chromatographia 62 (3-4), 183-195 (2005). Two chromatographic techniques are described for the separation and quantitative determination of azadirachtin A and B, salannin, and nimbin present in the crude extract of neem seed kernels and commercial neem based formulations. HPTLC separation of markers on silica gel with ethyl acetate - benzene 7:3. Visualization under UV 254 nm. The other technique was based on extended length packed column supercritical fluid chromatographic (PC-SFC) separation of the markers. Validation of both methods in terms of precision, robustness, recovery, limits of detection and quantitation. The analysis of variance (ANOVA) and Student's t-test were applied to correlate the results of quantitative determination of markers by means of HPTLC and PC-SFC method.

pharmaceutical research, traditional medicine, quality control, HPTLC, quantitative analysis, qualitative identification, comparison of methods, azadirachtin A and B, salannin, nimbin, neem seed kernels 32e

96 080 V. BILUSIC VUNDAC, Z. MALES*, M. PLAZIBAT, P. GOLJA, B. CETINA-CIZMEK (*Department of Pharmaceutical Botany, Faculty of Pharmacy and Biochemistry, University of Zagreb, Schrottova 39, 10000 Zagreb, Croatia): HPTLC determination of flavonoids and phenolic acids in some Croatian Stachys taxa. J. Planar Chromatogr. 18, 269-273 (2005). HPTLC of flavonoids (hyperoside, isoquercitrin, luteolin, luteolin 7-O glucoside, rutin, vitexin, quercetin, quercitrin as standards) and phenolic acids (caffeic and chlorogenic acid) on silica gel after presaturation with ethyl acetate - acetic acid - formic acid - water 100:11:11:26. Detection by spraying with natural products reagent, followed by spraying with PEG. Visualization under UV light at 254 and 366 nm. Quantitative evaluation by video-densitometry.

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

96 087 H. DANUTA SMOLARZ*, E. MEDYNSKA, G. MATYSIK (*Department of Pharmaceutical Botany, Medical University, 1 Chodzki Str., Lublin, Poland): Determination of emodin and phenolic acids in the petioles of Rheum undulatum and Rheum rhaponticum. J. Planar Chromatogr. 18, 319-322 (2005). HPTLC of emodin and phenolic acids (protocatechuic, homoprotocatechuic, caffeic, syringic, vanillic, ferulic, p-hydroxyphenylacetic, alpha-resorcylic, p-coumaric, gallic and ellagic acid) on silica gel in horizontal chambers with toluene - dichloromethane - ethyl acetate 4:4:1. Also two-dimensional TLC of phenolic acids on cellulose with benzene - methanol - acetic acid - acetonitrile 16:2:1:1 in the first direction and sodium formate - formic acid - water 10:1:200 in the second direction. After drying the chromatograms were observed under UV light at 254 nm before and after treatment with ammonia vapor. Derivatization was performed by spraying with either diazotized sulfanilic acid in 20 % sodium carbonate solution or with 2 % aqueous iron(III) chloride. Detection limits between 10 and 64 ng. Videodocumentation and quantitation by densitometry.

herbal, food analysis, HPTLC, quantitative analysis, qualitative identification 32e, 11a

96 152 V. V. DIGHE, A. A. GURSALE*, R. T. SANE, S. MENON, S. C. RAJE (*TDM Laboratory, plot no. 194, Scheme No. 6, Road No. 15, Sion (E), Koliwada, Mumbai 400 022, India): Quantification of eugenol in Cinnamomum tamala Nees and Eberm. leaf powder by high-performance thin-layer chromatography. J. Planar Chromatogr. 18, 305-307 (2005). HPTLC of methanolic extracts of Cinnamomum tamala leaves and eugenol on silica gel with toluene - ethyl acetate - formic acid 90:10:0.1. Detection and quantitation by densitometry at 280 nm.

herbal, food analysis, traditional medicine, HPTLC, quantitative analysis, densitometry, Cinnamomum tamala 32e

96 090 F. GBAGUIDI, G. MUCCIOLI, G. ACCROMBESSI, M. MOUDACHIROU, Joelle QUETIN-LECLERCQ* (*Laboratoire de Pharmacognosie, Unité CHAM, Université Catholique de Louvain, UCL 72 30, av. E. Mounier 72, 1200 Bruxelles, Belgium): Densitometric HPTLC quantification of 2-azaanthraquinone isolated from Mitracarpus scaber and antimicrobial activity against Dermatophilus. J. Planar Chromatogr. 18, 377-379 (2005). HPTLC of 2-azaanthraquinone from plant extracts on silica gel with toluene - ethyl acetate - methanol 40:9:1. Quantitative determination by absorbance measurement at 310 nm. Calibration was linear in the range of 10-100 µg/mL. The method was repeatable and precise with RSD between 0.98 and 1.59 % intra-day and between 3.41 and 5.56 % inter-day. Limits of detection and quantification were 3 and 6 µg/mL.

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

96 091 K. GLOWNIAK*, K. SKALICKA, A. LUDWICZUK, K. JOP (*Department of Pharmacognosy with Medicinal Plant Garden, Medical University, 1 Chodzki Str., 20-093 Lublin, Poland) : Phenolic compounds in the flowers of Lavatera trimestris L. (Malvaceae). J. Planar Chromatogr. 18, 264-268 (2005). TLC in horizontal chambers of phenolic acids (caffeic, p-coumaric, ferulic, protocatechuic, gentisic, chlorogenic, isovanillic, gallic, syringic, vanillic and p-hydroxybenzoic acid) from Lavatera trimestris flowers on cellulose with water - acetic acid 3:17, toluene - ethyl formate - formic acid 5:4:1, chloroforrm - acetic acid - water 4:1:1 and of flavonoids (rutoside, kaempferol 3-rhamnoglucoside, hyperoside, isoquercetin, luteolin 7-glucoside, quercitrin, isorhamnetin 3-glucoside, luteolin, apigenin, quercetin, kaempferol) on silica gel with n-propanol - ethyl acetate - water 7:2:1, ethanol - 25 % ammonia - water 20:1:4, ethyl acetate - formic acid - water 10:2:3, ethyl acetate - formic acid - glacial acetic acid - water 100:11:11:27 and 50:5:5:9. Also two-dimensional TLC of phenolic acids on cellulose (after conditioning in the chamber for 5 min with benzene - methanol - acetic acid 94:1:5) with benzene - methanol - acetic acid - acetonitrile 16:2:1:1 in the first direction and with sodium formate - formic acid - water 1:0.1:20 in the second direction. Detection under UV light at 254 and 366 nm before and after treatment with ammonia vapor. Also derivatization by spraying with a 2 % aqueous solution of iron(III)

chloride, diazotized sulfanilic acid in 20 % sodium carbonate solution, and diazotized p-nitroaniline for phenolics and with a 1 % methanolic solution of natural products reagent A for flavonoids.

herbal, qualitative identification, Lavatera trimestris, phenolics 32e, 8a

96 093 M. GUNTHER, P. SCHMIDT* (*Dept. of Pharmaceutical Technology, University of Tuebingen, Auf der Morgenstelle 8, 72076 Tuebingen, Germany): Comparison between HPLC and HPTLC-densitometry for the determination of harpagoside from Harpagophytum procumbens CO_2-extracts. J. Pharm. Biomed. Anal. 37, 817-821 (2005). CO2 extracts of Harpagophytum procumbens root was evaluated by HPLC and HPTLC for harpagoside contents. HPTLC on silica gel with ethyl acetate - methanol - water 77:15:8 in saturated ADC chamber. Detection by dipping into anisaldehyde reagent followed by drying at 120 °C for 5 min. Quantitative determination by absorbance measurement at 509 nm. The linearity range was 0.04-0.40 mg/mL. The HPTLC method was less time consuming than HPLC, needing almost no sample pre-treatment. 15 different CO2-extracts of the plant were analysed.

herbal, densitometry, comparison of methods, quantitative analysis, postchromatographic derivatization, HPTLC, harpagoside, Harpagophytum procumbens 32e

96 095 Urszula HACHULA*, S. ANIKIEL, M. SAJEWICZ (*Institute of Chemistry, Department of Analytical Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland) : Application of densitometry and spectrophotometry for determination of gallic acid in tea after chromatographic separation. J. Planar Chromatogr. 18, 290-293 (2005). TLC of gallic acid in tea extracts (gallic acid, caffeine, (+)-catechin, and tannic acid as standards) on silica gel in an unsaturated chamber with chloroform - ethyl acetate - formic acid 5:4:1. Densitometric measurement at 289 nm. Limit of detection 0.1 µg per spot.

food analysis, quantitative analysis, densitometry 32e

96 044 Erzsébet HAZNAGY-RADNAI et al., see section 14

96 097 V. JAIN, V. PRASAD*, P. MISHRA, R. PAL (*Pharmaceutics Division, CDRI, Lucknow 226 001, UP, India): HPTLC method for analysis of guggulsterone in formulations and Guggul resin extract. Abstract G-26, IPC (2005). A simple HPTLC method is reported for analysis of guggulsterones E and Z in herbal extract and market formulations containing commiphora mukul. Guggulsterones were extracted from crude extract and formulations by ethyl acetate. HPTLC on silica gel with n-hexane - ethylacetate 3:1. Quantitative determination by absorbance measurement at 250 nm. hRf value of E guggulsterone was 38 and of Z guggulsterone 46, linearity range for both isomers was 200-5000 ng/mL. The method was validated as per ICH guidelines.

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

96 103 N.S. KANAKI, M. RAJANI* (*B. V. Patel Pharmaceutical Education and Research Development (PERD) Centre, Thaltej-Gandhinagar Highway, Thaltej, Ahmedabad 380 054, Gujarat, India): Development and validation of a thin-layer chromatography-densitometric method for the quantitation of alliin from garlic (Allium sativum) and its formulations. J. Assoc. Off. Anal. Chem. 88, 1568-1570 (2005). HPTLC of alliin on silica gel with n-butanol - acetic acid - water 3:1:1 at 25 +/- 2 °C and 40 % relative humidity. Detection by dipping in ninhydrin reagent (0.3 g ninhydrin in 100 mL n-butanol and 3 mL acetic acid) for 2 s, followed by heating at 110 °C for 5 min. Quantitative determination by densitometric evaluation of peak areas at 540 nm. Linearity within the range of 250-1500 ng/spot, correlation coefficient of 0.998 and RSD of 2.87 %; mean recovery 98.4 %.

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

96 116 S. MANIMARAN, V. GOVINDAN*, R. SRINIVASAN, M. NANJAN, B. SURESH (*Dept. of Pharmacognosy and Phytochemistry, J.S.S College of Pharmacy, Ootacamund 643001, Tamil Nadu, India): Estimation of solanesol in various species of genus solanum by HPTLC. Abstract DP-41, IPC (2005). HPTLC of solanesol in n-hexane extracts of shade dried and freeze dried leaves of Solanum tuberosum, S. trilobactum, S. xanthocarpum, S. nigrum, and S. toruum on silica gel with chloroform - ethanol 48:1. Quantitative determination by absorbance measurement at 254 nm. Solanum tuberosum contained the highest amount of solanesol out of the 5 analyzed species.

herbal, HPTLC, densitometry, quantitative analysis 32e

96 119 C. MARUTOIU, L. OPREAN, O.-F. MARUTOIU, Maria-Loredana SORAN*, C. TIGAE, M. C. GONCEA (*'Lucian Blaga' University of Sibiu, Faculty of Food Technology, 7-9 Ion Ratiu Street, 2400 Sibiu, Romania): Quality control of commercial mustard by thin-layer chromatography. J. Planar Chromatogr. 18, 282-284 (2005). TLC and HPTLC of horseradish and mustard samples on silica gel with iso-propanol - 25 % ammonia 9:1 containing different volumes of water (1, 2, 3, or 5 parts). After development the compounds were visualized under UV light at 254 nm or by exposure to iodine vapor.

food analysis, quality control, qualitative identification, HPTLC 32e

96 125 Renata NOWAK*, M. HAWRYL (*Department of Pharmaceutical Botany, Medical University, 1 Chodzki St, 20-093 Lublin, Poland): Application of densitometry to the determination of catechin in rose-hip extracts. J. Planar Chromatogr.18, 217-220 (2005) TLC of rose-hip extracts and (+)-catechin and (-)-epicatechin as standards on cellulose and silica gel in a horizontal chamber, saturated for 15 min, with the upper phase of ethyl acetate - water - formic acid - acetic acid 125:20:3:2. Visualization under UV light at 254 and 365 nm before and after spraying with bis-diazotized sulfanilamide. Quantitative determination by absorbance measurement at 254 nm.

herbal, quantitative analysis, densitometry 32e, 8b

96 100 U. K. PATIL, V. K. DIXIT* (*Department of Pharmaceutical Sciences, Dr Harizingh Gour University, Sagar (M. P.) 470003, India): Densitometric standardization of herbal medical products containing Evolvulus alsinoides by quantification of a marker compound. J. Planar Chromatogr. 18, 234-239 (2005). TLC of Evolvulus alsinoides extracts and the marker EA 1, i.e. 3beta,23,24-trihydroxyolean-12-en-28-oic acid, on silica gel with n-hexane - ethyl acetate 7:3 in a saturated chamber. The marker EA 1 was detected under UV light at 366 nm as a yellow fluorescent band of Rf 0.8. Quantitative determination by absorbance measurement at 232 nm (=UV max of EA 1). Limit of detection 11.6 ng; satisfactory recovery from 93.3 to 96.6 %. The marker was isolated from the aerial parts of Evolvulus alsinoides by preparative TLC.

herbal, quality control, densitometry, quantitative analysis, preparative TLC, Evolvulus alsinoides, EA 1 32e

96 136 Anne SCHIBLI*, E. REICH (*CAMAG Laboratory, Sonnenmattstrasse 11, 4132 Muttenz, Switzerland): Modern TLC: A key technique for identification and quality control of botanicals and dietary supplements. J. Planar Chromatogr. 18, 34 -38 (2005). Considering the latest technical and methodological developments, modern high-performance thin-layer chromatography, also known as planar chromatography, is a reliable and powerful analytical technique, in full compliance with current good-manufacturing practice (cGMP). With the proper equipment TLC is the method of choice when many samples must be analyzed at low cost per sample. Advantages of HPTLC are shown in the analysis of botanicals: 1) Identification (separation of Stephania tetrandra root extracts with tetrandrine as standard on silica gel with toluene - ethyl acetate - methanol -ammonia 100:100:50:3; detection under UV at 254 and 366 nm, under white light after derivatization with iodine, and under UV at 366 nm after derivatization with anisaldehyde. 2)

Semi-quantitative assessments in process control and stability tests (separation of fatty acids of Saw Palmetto products on RP-18 by two fold development with dichloromethane - acetic acid - acetone 2:4:5. 3) Quantification of marker compounds, like curcumin measured at 366 nm/>400 nm on silica gel with toluene - acetic acid 4:1. 4) Choice of stationary phase (separation of flavonoids on conventional TLC plates and on HPTLC plates with formic acid - water - ethyl methyl ketone - ethyl acetate 10:10:30:50 and detection with natural products reagent; switching to HPTLC reduced analysis time to a quarter and gave sharper bands). 5) Choice of mobile phase; 6) Derivatization and 7) Chromatogram evaluation.

herbal, quality control, HPTLC, densitometry, qualitative identification 32e

96 141 N.P. SINGH, A.P. GUPTA, A.K. SINHA*, P.S. AHUJA (*Natural Plant Products Division, Institute of Himalayan Bioresource Technology, Post Box No. 6, Palampur 176061, Himachal Pradesh, India): High-performance thin layer chromatography method for quantitative determination of four major anthraquinone derivatives in Rheum emodi. J. Chromatogr. A 1077 (2), 202-206 (2005). HPTLC of physcion, chrysophanol, emodin and chrysophanol glycoside in Rheum emodi on RP-18 with methanol - water - formic acid 80:19:1. Quantitative determination by absorbance measurement at 445 nm. The calibration curves were linear in the range of 20-100 ng for physcion, 80-400 ng for chrysophanol and emodin, and 200-1000 ng for chrysophanol glycoside. The method was found to be reproducible and convenient for quantitative analysis of anthraquinone derivatives in the methanolic extract of rhizomes of Rheum emodi collected from three different locations of Western Himalaya, India.

pharmaceutical research, traditional medicine, HPTLC, qualitative identification, quantitative analysis, Rheum emodi, anthraquinones, physcion, chrysophanol, emodin, chrysophanol glycoside 32e

96 147 B. SZABO, A. LAKATOS, T. KOSZEGI, G. KATAY, L. BOTZ* (*Pécs University, Medical School, Pharmaceutical Institute, H-7624 Pécs, Honvéd u. 3, Hungary): Thin-layer chromatography-densitometry and liquid chromatography analysis of alkaloids in leaves of Papaver somniferum under stress conditions. J. Assoc. Off. Anal. Chem. 88, 1571-1577 (2005). TLC and HPTLC of narceine, morphine, codeine, thebaine, papaverine, and narcotine on silica gel with toluene - acetone - ethanol - 25% ammonia 20:20:3:1. Detection with Dragendorff's reagent with sodium nitrite, densitometric evaluation at 520 nm. For UV-detection, Naturstoff reagent A (diphenylboric acid 2-amino ethyl ester) was used; quantitation by densitometry at 310 nm.

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

96 154 V. WAGH, S. GUPTA*, M. SAMANTA, B. SURESH (*J.S.S College of Pharmacy, Ootacamund 643001, Tamil Nadu, India): Analysis of forskolin from an herbal extract and its opthalmic formulations by HPTLC. Abstract DP-15, IPC (2005). HPTLC of forskolin in herbal extract and opthalmic preparation (prepared from methanolic extracts of Coleus forskohlii roots) on silica gel with toluene - ethyl acetate 17:3. Quantitative determination by absorbance measurement at 292 nm. The method was found to be reproducible, accurate and precise.

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

96 155 Valeria WIDMER, Anne SCHIBLI, E. REICH* (*CAMAG Laboratory Services, Sonnenmattstr. 11, 4132 Muttenz, Switzerland): Quantitative determination of beta-asarone in Calamus by high-performance thin-layer chromatography. J. Assoc. Off. Anal. Chem. 88, 1562-1567 (2005). HPTLC of beta-asarone (cis-2,4,5-trimethoxy-1-propenylbenzene) and alpha-asarone in Calamus rhizome on caffeine-impregnated silica gel (prepared by immersion of conventional silica gel plates into a solution of 80 g/L caffeine in dichloromethane for 1 s followed by drying at room temperature for 5 min, then heating at 80 °C for 5 min) with toluene - ethyl acetate 93:7.

Quantitative determination by absorbance measurement at 313 nm. The method was validated in terms of stability of sample during chromatography, specificity for beta-asarone, linearity (25-300 ng, including samples containing 0.05-0.7 % beta-asarone), accuracy and precision. Recovery was 100.0-100.8 %, limit of detection 6.4 ng, and limit of quantitation 12.7 ng. The method allows proper identification of Calami rhizoma raw material, and the specific, accurate, and precise quantification of beta-asarone and alpha-asarone

food analysis, herbal, densitometry, quantitative analysis, HPTLC, qualitative identification, Acorus calamus 32e

96 131 K.M. PATIL, S.L. BODHANKAR* (*Department of Pharmacology, Bharati Vidyapeeth Deemed University, Poona College of Pharmacy, Pune 411038, Maharashtra, India): High-performance thin-layer chromatographic determination of lamotrigine in serum. J. Chromatogr. B 823 (2), 152-157 (2005). HPTLC of lamotrigine (extracted from serum by ethyl acetate) on silica with toluene - acetone - ammonia 14:6:1. Densitometric measurement at 312 nm, hRf of lamotrigine at 54. The analytical method has excellent linearity (r = 0.998) in the range of 20-300 ng/spot. This assay range is adequate for analyzing human serum, as it corresponds to lamotrigine concentrations measured in human serum from epileptic patients. The method was validated for sensitivity, selectivity, extraction efficiency, accuracy and intra and inter-day reproducibility. The limit of detection and limit of quantification were found to be 6.4 and 10.2 ng, respectively. Good accuracy and high precision (CV) is reported, i.e. in the range of 92.1-97.1 % and 0.5-2.6 % respectively. The method was applied for determination of serum lamotrigine levels in epileptic patients and in pharmacokinetic study of lamotrigine administered orally to rabbits.

clinical chemistry research, densitometry, quantitative analysis, qualitative identification, HPTLC, lamotrigine 32f

96 099 ZH. JIN (Jin Zhu)*, H. WEI (Wei Hong), B. LI (Li Bingjun), Q. ZHAO (Zhao Quancheng), X. GONG (Gong Xuguo) (*Jinlin Tianyao Sci. & Tech. Co., Ltd., Changchun, Jilin 130012, China): (Study of the quality standard for Gubiling capsules) (Chinese). J. Chinese Trad. Patent Med. (Zhongchengyao) 27(5), 529-532 (2005). TLC of extracts on silica gel developed with 1) ethyl acetate - ethanol 4:1; 2) ethyl acetate - methyl ethyl ketone - formic acid - water 10:1:1:1; 3) cyclohexane - acetone 10:3. Detection 1) under UV 254 nm; 2) by spraying with 5 % AICl3 in ethanol followed by heating at 105 ºC for 5 min, and inspection under UV 365 nm; 3) by spraying with 5 % vanillin in H_2SO_4 followed by heating at 105 ºC until the spots are visualized. Identification by fingerprint technique. Quantification of ginsenoside Rg1 by HPLC. The results for three real life samples are given.

pharmaceutical research, herbal, quality control, traditional medicine, qualitative identification, densitometry, ginsenoside Rg1 32g

96 117 S. MANIMARAN*, S. GULSHAN, S. PARUL, L. NANJAN, M. CHINNASWAMY, B. SURESH (*Department of Phytopharmacy & Photomedicine, J.S.S. College of Pharmacy, Rockland, Ootacamund 643001, Tamil Nadu, India): Analysis of Milagai Thailam for its capsaicin and piperine content by HPTLC. Indian Drugs 42 (12), 802-804 (2005). HPTLC of Milagai Thailam on silica gel with toluene - acetone 7:3 with chamber saturation for 30 min. Quantitative determination by absorbance measurement at 254 nm. The hRf value of capsaicin was 17 and of piperine 27. Recovery rates were in the range of 99.3-99.5 %. The sample contained 0.97 mg/10 mL capsaicin and 0.6 mg/10 mL piperine. The method was found suitable for other herbal formulations containing capsaicin and piperine.

herbal, pharmaceutical research, HPTLC, densitometry, quantitative analysis, capsaicin, piperine 32g

33. Inorganic substances

96 159 V. ZIVKOVIC-RADOVANOVIC, G. VUCKOVIC* (*Faculty of Chemistry, University of Belgrade, P.O. Box 158, 11001 Beograd, Serbia and Montenegro, gordanav@chem.bg.ac.yu): Poly(ethylene glycol) as impregnator for silica gel in salting-out thin-layer chromatography of some Co(III) complexes. Chromatographia 62 (1-2), 91-97 (2005). Silica gel impregnation with polyethylene glycol of different molecular mass (400, 1000, 1540, 4000, and 5500) was investigated for salting-out thin-layer chromatography of 15 mixed aminocarboxylato Co(III) complexes using eight ammonium sulphate solutions as mobile phases. Regularities established earlier for non-impregnated adsorbents are also valid in this work. Polyethylene glycol of high molecular mass increases the hydrophobicity of the adsorbent. Positive linear dependence of RM values and of salting-out efficiency on average polyethylene glycol molecular mass was usually observed. In contrast with non-impregnated silica gel, separation was achieved between complexes with the smallest hydrocarbon groups.

herbal, qualitative identification, polyethylene glycols, salting-out thin layer chromatography, Co(III) complex 33a, 3b

96 157 P.A. MOHAMED NALAR*, J.U. JEURKAR, K.V. RAMANA RAO (*Jawaharlal Nehru Aluminium Research Development and Design Centre, Nagpur 440023, India): Thin layer chromatographic study of bauxite and quantitative estimation of co-existing Al^{3}+, Fe^{2}+ and Ti4+. Chinese J. Chromatogr. (Sepu) 23 (5), 555-561 (2005). Use of TLC in combination of spectrophotometry and titrimetry to evaluate chromatographic characteristics of bauxite constituents. Examination of the retention behaviors of four constituents (Al^{3+}, Fe^{2+}, Ti^{4+}, Si^{4+}) on modified silica gel and cellulose with a mobile phase containing aqueous sodium chloride, formic acid and hydrochloric acid. Ternary separation of Al-Fe-Ti was achieved on silica gel H. Study of pH influence, presence of impurity elements in the samples, nature of the stationary phase on the ternary separation, and of detection limits of bauxite constituents; detection of silica in bauxite on cellulose plates. Quantitative determination of Al^{3}+, Fe^{2}+, and Ti4+ on silica gel H impregnated with sodium formate.

quantitative analysis, HPTLC, silica, aluminium, iron, titanium, bauxite 33

96 158 Maria-Loredana SORAN*, T. HODISAN, M. CURTUI, D. CASONI (*National Institute of Research and Development for Isotopic and Molecular Technology, 72-103 Donath Street, 400293 Cluj-Napoca, Romania): TLC separation of rare earths using di(2-ethylhexyl)dithiophosphoric acid as complexing agent. J. Planar Chromatogr. 18, 160-163 (2005). TLC of rare earths (La(III), Ce(III), Pr(III), Sm(III), Gd(III), Er(III)) on silica gel and silica gel impregnated with 2.5 M ammonium nitrate with different mixed mobile phases containing di(2-ethylhexyl)dithiophosphoric acid as a complexing agent. The best results were obtained by use of ethyl methyl ketone - tetrahydrofuran - 1 M di(2-ethylhexyl)dithiophosphoric acid 17:8:1. Double development was used to obtain better separation of consecutive rare earths.

qualitative identification, quantitative analysis, densitometry 33

35. Other technical products and complex mixtures

96 160 Soheila HONARY*, H. JALILI (Mazandaran University of Medical Science, School of Pharmacy, Sari, Iran): HPTLC determination of antioxidants in the polymer container of parenteral infusion fluid. J. Planar Chromatogr. 18, 403-404 (2005). HPTLC of antioxidants (Irgafos 168, Irganox 1010, Irganox 1078, Irganox 1330, and BHT) and hexane extracts of polymer granules on silica gel (prewashed with chloroform -methanol 1:1) in an unsaturated twin-trough chamber with hexane - methanol 1:4. Quantitative determination of Irganox 1078 by absorbance measurement at 254 nm.

pharmaceutical research, quality control, HPTLC 35b

96 162 W. LIANG (Liang Wenbo)*, X. LIU (Liu Xongmin), J. LIANG (Liang Jngjuan), P. LI (Li Piaoying), F. SHEN (Shen Fang) (*Chem. & Chem. Eng., Guangxi Univ., Nanning, Guangxi 530004, China): (Study of the procedure for the determination of omega-pentandecalactone by thin-layer chromatography) (Chinese). Chinese J. Chromatogr. (Sepu) 23 (2), 217-218 (2005). TLC of the title compound on silica gel with chloroform - benzene 7:3. Detection 1) by spraying with 10 % H_2SO_4 in ethanol followed by heating at 120 ºC for 10 min; 2) by spraying with 10 % phosphomolybdic acid in ethanol followed by heating at 120 ºC for 10 min, then exposing to ammonia vapor. Quantification by comparison of the separated zone size with the standard. Optimization of the mobile phase by investigation of the influence of the composition of the developing solvent on Rf values. Optimization of the visualization condition by investigating the relationship between the sample dosage and the visualization results.

densitometry, quantitative analysis, postchromatographic derivatization, omega-pentadecalactone 35c

96 163 N. SIKDER*, N.R. BULAKH, A.K. SIKDER, B.R. GANDHE (*High Energy Materials Research Laboratory, Sutarwadi, Pune 411 021, India): High-performance thin-layer chromatographic analysis of the organic components of composite modified double-base propellants. J. Planar Chromatogr. 18, 57-60 (2005). HPTLC of the organic components of composite modified double-base (CMDB) propellants (with nitroglycerine, carbamite, diethyl phthalate, dibutyl phthalate, 2-nitrodiphenylamine as standards) on silica gel with chloroform - cyclohexane 7:3. Detection under UV light at 254 nm. Densitometric scanning in absorbance mode at 210 nm. Comparison of the UV spectra of the separated compounds with those of the standards.

HPTLC, qualitative identification, quantitative analysis, densitometry, explosives 35c

96 161 W. LI, T.J. MORGAN, A. A. HEROD*, R. KANDIYOTI (*Department of Chemical Engineering and Chemical Technology, South Kensington Campus, Imperial College London, London SW7 2AZ, United Kingdom): Thin-layer chromatography of pitch and a petroleum vacuum residue. Relation between mobility and molecular size shown by size-exclusion chromatography. J. Chromatogr. A 1024 (1-2), 227-243 (2004). TLC of coal tar pitch and a petroleum vacuum residue with pyridine, acetonitrile, toluene and pentane. The bands of material detected were recovered in 1-methyl-2-pyrrolidinone (NMP) solvent and examined by size-exclusion chromatography (SEC) in NMP eluent. The relation between elution time in SEC and mobility on the TLC plate indicated that molecular size increased steadily with increasing immobility on the plate. This relation was reinforced by UV fluorescence spectroscopy in that the fluorescence moved to longer wavelengths with increasing immobility. The molecular size of the material excluded from the porosity of the SEC column remains undefined; some excluded material was found in all of the fractions from both samples. The valley of zero intensity separating the retained material from the excluded material may suggest a change of structure from near-planar in the retained region to three-dimensional in the excluded region.

coal tar; petroleum 35d

37. Environmental analysis

96 164 I. ALI*, V. K. GUPTA, P. SINGH, H. V. PANT (*National Institute of Hydrology, Roorkee 147 667, India): RPTLC analysis of haloperidol and its metabolites in wastewater after solid-phase extraction. J. Planar Chromatogr. 18, 388-390 (2005). TLC of haloperidol and metabolites on RP-18 previously equilibrated for 30 min with methanol containing 0.001 % triethylamine at 27 +/- 1 °C (room temperature). Detection by treatment with iodine vapor. Quantitative determination at 230 nm.

environmental, qualitative identification 37c

96 061 Sandra BABIC et al., see section 28a

96 165 M. SAJEWICZ (Institute of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland): Use of densitometric TLC for detection of selected drugs present in river water in South Poland. J. Planar Chromatogr. 18, 108-111 (2005). TLC of josamycin, sulfamethoxazole, carbamazepine, diclofenac, and iopromide after SPE on RP-18 with acetonitrile - water - acetic acid 5:5:2. Quantitative determination by absorbance measurement at 220 nm. Detectability of substances was lower than 1 µg (1.25 µg for sulfamethoxazole) per applied sample. The water was contaminated with all five drugs, concentrations found ranged from 0.017-1.314 µg / L water.

environmental, densitometry, quantitative analysis 37c

38. Chiral separation

96 166 Branka LUCIC*, D. RADULOVIC, Z. VUJIC, D. AGBABA (*Institute of Pharmaceutical Chemistry and Drug Analysis, Faculty of Pharmacy, Vojvode Stepe 450, P. O. Box 146, 11 000 Belgrade, Serbia and Montenegro): Direct separation of the enantiomers of (+/-)-metoprolol tartrate on impregnated TLC plates with D-(-)-tartaric acid asa chiral selector. J. Planar Chromatogr. 18, 294-299 (2005). TLC of (+/-)-metoprolol tartrate and (-)-alprenolol tartrate on silica gel, with concentrating zone, on RP-18, on LiChrospher silica gel and on alumina, previously impregnated with the mobile phase (ethanol - water 7:3, containing D-(-)-tartaric acid as chiral selector). Detection under UV light at 254 nm and densitometric scanning at 230 nm. Direct separation of the enantiomers of (+/-)-metoprolol tartrate was achieved.

quality control, densitometry, quantitative analysis 38

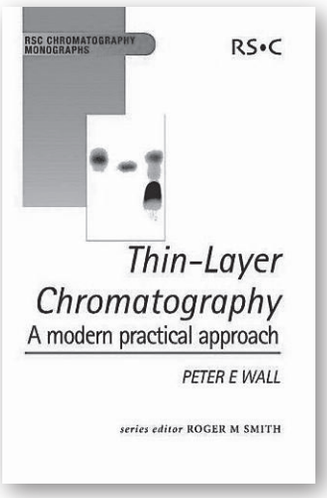
96 167 A.M. SIOUFFI*, P. PIRAS, C. ROUSSEL (*Université Paul Cezanne, UMR 6180, 13397, Marseille, France): Some aspects of chiral separations in planar chromatography compared with HPLC. J. Planar Chromatogr. 18, 5-12 (2005). Review of the latest achievements in chiral separation by planar chromatography since 2001 (chiral ligand exchange; cellulose and derivatives; coated or impregnated layers; cyclodextrins; miscellaneous; diastereoisomers; conclusions). The emphasis is on cellulose derivatives and, especially, microcrystalline cellulose triacetate (MCTA) showing that TLC has some interesting features compared with HPLC. Some enantiomer separations have been successfully achieved by TLC whereas no data are available for HPLC. For tribenzoyl cellulose derivatives general trends for resolution by both TLC and HPLC are discussed. Furtheron reasons for the scarcity of publications on chiral separations by either planar chromatography or overpressured layer chromatography are discussed. The possibilities of PC for chiral separations are rather unexploited.

review 38, 1

Peter E. Wall

Thin-Layer Chromatography – A modern practical approach

The Royal Society of Chemistry (RSC),
Cambridge, 2005
ISBN 0-85404-535-X



In his book, Peter Wall addresses the whole TLC process from a practical point of view. Besides the book of Elke Hahn-Deinstrop "Applied Thin-Layer Chromatography – Best Practice and Avoidance of Mistakes" (recension see CBS 80), within 5 years a further practical book was devoted to the technique and modern instrumentation accompanied by many examples. Both books have a similar approach. Whereas the book of Peter Wall focuses the given state of TLC on 184 pages, the book of Elke Hahn-Deinstrop gives many hints and detailed advices on 304 pages. Both books derive from an own long-term experience in TLC, however the two books differ to some extent in grade of practical approach and own profile.

In 8 chapters relevant topics are covered ranging from sorbents, sample pretreatment, application, development, detection and quantification to coupling techniques. Many well-chosen examples, figures and tables illustrate the theoretical aspects. Information is concentrated and thus a real good introduction to the single topics is given. However, a chapter about documentation and the powerful visual impression of planar chromatograms is missing. The very new developments regarding sorbents, like the UTLC plates, or the latest trends about coupling TLC with MS are also not covered. Information to modern instrumentation is based on the product experience of the author and regrettably mainly slanted towards one manufacturer although others have good instrumentation as well.

The really moderate price of Euro 118.50 or $ 139.– allows for the medium print quality and missing colored images of planar chromatograms. All in all the book provides a compact introduction into the state of modern TLC and the contemporary look of high performance TLC. Thus the book is primarily directed towards the staff, practising chromatographers and beginners to provide guidance and to obtain general knowledge on the technique.

Prof. Dr. Wolfgang Schwack

Institute of Food Chemistry
University of Hohenheim
Stuttgart, Germany

Erste Erfahrungen mit der Dünnschicht-Chromatographie

16

▲ *Chemielehrer Stefan Grabe (untere Reihe 2. von links) und der Leistungskurs Chemie des Christoph-Jacob-Treu-Gymnasiums in Lauf a. d. Pegnitz.*

Im Rahmen des Projektes der Bundesregierung Deutschland »Center of Excellence – Zentrum für Schulqualität« hat sich das Christoph-Jacob-Treu-Gymnasium in Lauf a. d. Pegnitz u.a. das Ziel gesetzt, Experten aus der Industrie als Referenten einzuladen, um den Unterricht praxisnah zu gestalten.

Die Dünnschicht-Chromatographie (DC) ist für den Einsatz in der Schule von allen chromatographischen Verfahren am besten geeignet – als bildliches Verfahren kann sie den Begriff »Chromatographie« sehr gut verdeutlichen. Um den Schülern einen Einblick in die Leistungsfähigkeit der modernen Dünnschicht-Chromatographie zu ermöglichen, sollte auch in den Schulen eine instrumentelle Ausstattung gegeben sein, vor allem hinsichtlich Auftragung und Entwicklung, möglichst jedoch auch ein Dokumentationssystem, denn das Bild ist eine der Stärken der DC.

Aus der pharmazeutischen Industrie konnte Frau Elke Hahn-Deinstrop gewonnen werden. Kursleiter Stefan Grabe unterrichtete zuvor die 17 Schüler/-innen in der Planar-Chromatographie.

Während einer kurzen Einführung in die Geschichte der Chromatographie erläuterte Frau Hahn-Deinstrop die Bedeutung der Arbeiten von Friedrich Runge, Michail Tswett und natürlich Egon Stahl. Mit allen modernen Fertigschichten hingegen ist der Name von Heinz E. Hauck verbunden, der seit ca. 30 Jahren bei Merck in Darmstadt für die Forschung und Entwicklung in der DC/HPTLC verantwortlich zeichnet.

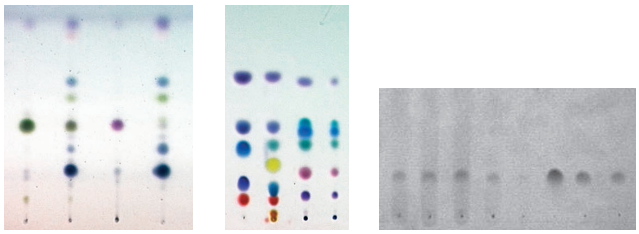
17

▲ *»Center of Excellence – Zentrum für Schulqualität«: Praktischer Teil mit Frau Elke Hahn-Deinstrop (2. von links)*

Im praktischen Teil des ersten Labortages lernten die Schüler dann unterschiedliche Sorbentien, verschiedene Fliessmittel und den Einfluss der Kammersättigung kennen. Farbstoffmischungen zeigten hierbei eindruckvoll das Prinzip der Chromatographie.

Der zweite Nachmittag stand im Zeichen von Monographien des Europäischen Arzneibuches. Geprüft wurden verschiedene Pfefferminz-, Kiefernnadel-, Fichtennadel- und Lavendelöle auf ihre Identität. Besonders spannend fanden die Schüler die Überprüfung der Droge »Safran« auf Verfälschungen – über die Methode haben wir bereits im CBS 91 berichtet.

Am dritten Unterrichtstag wurden Arzneipflanzen und deren Zubereitungen (z.B. Tabletten und Tropfen) untersucht, wie Roter Sonnenhut (*Echinaceae purpurea*) und die Blätter des *Ginkgo biloba*-Baumes. Außerdem wurde der Wirkstoff Coffein in geröstetem und gemahlenem Kaffee sowie in rohem Wildkaffee bestimmt.



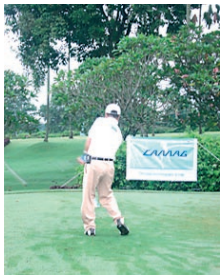
18

▲ *Chromatogramme des Chemie-Leistungskurses zeigen die Identifizierung von ätherischen Ölen, die Trennung von Farbstoffen sowie Koffein in verschiedenen Kaffeesorten und Tee*

Lust auf einen Beruf im naturwissenschaftlichen Bereich? Der Workshop in Lauf hat einen Puzzlestein dazu beigetragen.

Fragen zu den Prüfvorschriften richten Sie bitte direkt an Frau Hahn-Deinstrop, E-mail: elke.hahn_deinstrop@arcor.de

25 Jahr-Jubiläum von PT. ABADINUSA USAHASEMESTA – CAMAG´s Vertretung in Indonesien


19


20

▲ *Das »CAMAG Loch« am Golf-Turnier (links) Herr Yahya Kurniawan, General Manager (rechts)*


21

▲ *Das Verkaufsteam (stehend von links nach rechts): Bayu, Sapri, Kustono, Paul, Riza, Aris, Johan, Rio; (sitzend) Lusi, Mrs. Titi BP (Finanz- und Administrationsmanager) und Lenny*

PT. AGADINUSA USAHASEMESTA, Raden Saleh 45 G, Jakarta, ist seit 1983 CAMAG Exklusivvertreter für Indonesien. In den vergangenen 23 Jahren wurden zusammen mit CAMAG viele Aktivitäten durchgeführt, die zur Marktentwicklung für die Planar-Chromatographie in Indonesien beitrugen.

Ein Teil der 25 Jahr-Jubiläumsfeier von ABADINUSA USAHAMSEMSTA war ein Golfturnier im Emeralda Golf und Country Club in Jakarta im Dezember 2005. 98 eingeladene betroffene Personen und Freunde des Hauses feierten diesen denkwürdigen Anlass der langen erfolgreichen Zusammenarbeit mit CAMAG.

Herr Yahya Kurniawan and sein Verkaufsteam werden weiterhin aktiv bleiben, um den Marktanteil der CAMAG-Produkte zu steigern und CAMAG´s Marktführer-Position in der modernen Planar-Chromatographie zu halten.

Fortschritte bei Chromacim SAS, unserer Vertretung in Frankreich


22

▲ *Herr Pierre Devidal, Frau Brigitte Marandet, Herr Pierre Bernard-Savary (links nach rechts)*


23

▲ *Die neue Chromacim-Geschäftsstelle in Voiron*

Chromacim wurde im März 2002 gegründet von Pierre Bernard-Savary gemeinsam mit Christian Gfeller, Bereichsleiter Finanzen von CAMAG Muttenz. Das sich günstig entwickelnde Geschäft erforderte den Umzug in grössere Räume in Voiron. Gegenwärtig arbeiten dort drei Personen am Erfolg der HPTLC in Frankreich:

Herr Pierre Devidal, Elektronik-Ingenieur, ist zuständig für alle Service-Aktivitäten einschliesslich IQ/OQ und 21 CFR part 11.

Frau Brigitte Marandet ist zuständig für die Administration einschliesslich Angebotswesen, Lagerbewirtschaftung und Versand von Ersatzteilen und Verbrauchsmaterial. (Grössere Geräte werden direkt aus der Schweiz an unsere Kunden in Frankreich versandt.)

Herr Pierre Bernard-Savary, Geschäftsführer von Chromacim, blickt auf 15 Jahre Tätigkeit für CAMAG zurück, davon die ersten 10 Jahre als CAMAG Produktspezialist bei Merck France. 1997 gründete er den »Club CCM« (französischer HPTLC-Club) und war 2003 Chairman des »International Symposium for HPTLC« in Lyon. Das nächste Symposium dieser Reihe wird vom 9.–11. Oktober 2006 in Berlin stattfinden (www.hptlc.com).

Quantifizierung von ITX in Lebensmitteln mittels HPTLC/FLD gekoppelt mit ESI-MS und DART-MS

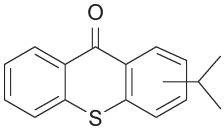
◀ *Prof. Dr. Wolfgang Schwack und Dr. Gerda Morlock**

24

Die Arbeitsgruppe von Prof. Dr. Schwack, Universität Hohenheim, Stuttgart, beschäftigt sich aktiv mit der Planar-Chromatographie (siehe CBS 94). Ende November 2005 sorgte Isopropylthioxanthon (ITX) als neu entdeckte Lebensmittel-Kontaminante in der Öffentlichkeit für Aufruhr. In der Literatur ist bisher keine analytische Methode zur Quantifizierung von ITX in betroffenen Lebensmitteln bekannt. Aufgrund des erforderlichen hohen Probendurchsatzes wurde diese neue empfindliche und selektive planar-chromatographische Methode basierend auf Fluoreszenzdetektion (FLD) und der Bestätigung positiver Ergebnisse mittels ESI-MS (Elektrospray-Ionisation-Massenspektrometrie) und DART-MS (Direct Analysis in Real Time-Massenspektrometrie) entwickelt [1].

Einleitung

Isopropylthioxanthon (ITX) wird als Fotoinitiator in UV-härtenden Offset-Druckfarben eingesetzt, mit denen z. B. Mehrschichtverbunde oder Kunststoffbecher aussenseitig bedruckt werden. Kürzlich wurde es in Spuren in Milch und Baby-Milchprodukten nachgewiesen; als Folge wurden weit über 30 Millionen Liter Milch in Italien, Frankreich, Spanien und Portugal zurückgezogen.



▲ *Strukturformel von Isopropyl-9H-thioxanthen-9-on (ITX)*

Durch Migration durch den Packstoff oder durch Abklatsch beim Transport kann ITX auf die Lebensmittelkontaktseite gelangen und von dort in das Lebensmittel übergehen. Offensichtlich sind fetthaltige Lebensmittel stärker betroffen als wasserhaltige. Neben Baby-Milchprodukten sind auch andere Milchprodukte, Milch, Sojabohnengetränke, Fruchtsäfte/-nektare sowie weitere Getränke und Lebensmittel, die auf die gleiche Weise verpackt werden, wie Kakaopulver oder Olivenöl, betroffen und auf Abwesenheit von ITX zu prüfen.

Aufgrund der nachgewiesenen Konzentrationen bis zu einigen 100 µg/kg wird ITX zwar als nicht gesundheitsgefährdend eingestuft – allerdings gibt es nur vereinzelt Studien über ITX und diese nur zu dessen Gentoxizität, zu wenige für eine abschliessende gesundheitliche Risiko-Bewertung. Zur Zeit ist nicht nur das Monitoring von ITX von Interesse, sondern auch von weiteren Fotoinitiatoren, wie 2-Ethylhexyl-4-dimethylaminobenzoat (EHDAB) wie auch 4,4'-Bis(diethylamino)-benzophenon und 4,4'-Bis(dimethylamino)-benzophenon.

Die Methode entspricht den Interessen der Lebensmittelindustrie und -kontrolle, die Abwesenheit von ITX in betroffenen Lebensmitteln effektiv bei einem hohen Probendurchsatz zu prüfen. Die Ergebnisse weisen die moderne Planar-Chromatographie als eine schnelle und kosteneffektive alternative Methode aus, um ITX in Milch-basierten oder fetten Matrizes im unteren µg/kg-Bereich zu bestimmen. Nur positive Befunde werden mittels online ESI-MS im SIM-Modus oder DART-MS bestätigt, um das Massenspektrometer minimal zu beanspruchen – ein weiterer Vorteil der HPTLC.

Probenvorbereitung

4 mL Milch bzw. 4 g Joghurt werden mit Cyclohexan - Ethylacetat 1:1 (v/v) mittels ASE (accelerated solvent extraction) extrahiert. Vor der Extraktion werden 25 µL einer 2,4-Diethyl-9H-thioxanthen-9-on (DTX)-Lösung (8 µg/mL) als interner Standard zugegeben. Der Extrakt wird über 4 g wasserfreiem Natriumsulfat getrocknet, filtriert und mittels Gefriertrocknung zur Trockne eingedampft. Der Rück-

stand wird in 1.5 mL Acetonitril aufgenommen, zur Trockne eingeengt und erneut in 250 µL Acetonitril aufgenommen.

Für Sojaöl und Margarine wird eine einfache Verteilung von ITX in Acetonitril durchgeführt. 1 mL Acetonitril und 25 µL DTX-Lösung werden zu 1 g Fett hinzugefügt (plus 200 mg Magnesiumsulfat für Margarineproben) und bei 50 °C für 30 min bei 600/min geschüttelt. Die klare obere Acetonitril-Phase wird für die Auftragung eingesetzt.

Standardlösung

ITX (3.2 µg/mL) und DTX (8 µg/mL) werden jeweils in Acetonitril gelöst.

Schicht

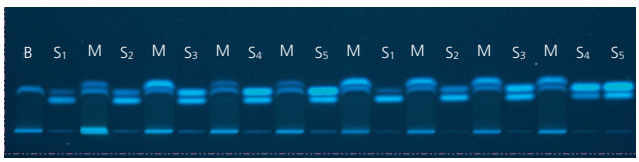
HPTLC-Platten Kieselgel 60 (Merck) 20 x 10 cm

Probenauftragung

Strichförmig mit DC-Probenautomat 4, 18 Bahnen, Auftragevolumen 30 µL Probelösung und 3 - 30 µL Standardlösung, Bandlänge 7 mm (Bahnabstand 9 mm), unterer Randabstand 8 mm, seitlicher Randabstand 20 mm. Zur internen Standardauswertung werden alle ITX-Standardzonen mit 3 µL DTX-Standardlösung übersprüht (24 ng/Zone).

Chromatographie

In einer Horizontal-Entwicklungskammer mit Toluen – n-Hexan (4:1, v/v); Laufstrecke 50 mm vom unteren Plattenrand. Nach der Chromatographie wurde die Platte für 1 min im warmen Luftstrom getrocknet. Die Entwicklung wird beidseitig anti-parallel durchgeführt, um einen Durchsatz von 36 Trennungen in 7 min Laufzeit zu ermöglichen. Pro Einzel-Trennung werden weniger als 0.2 min Trennzeit und 0.3 mL Lösungsmittel benötigt.



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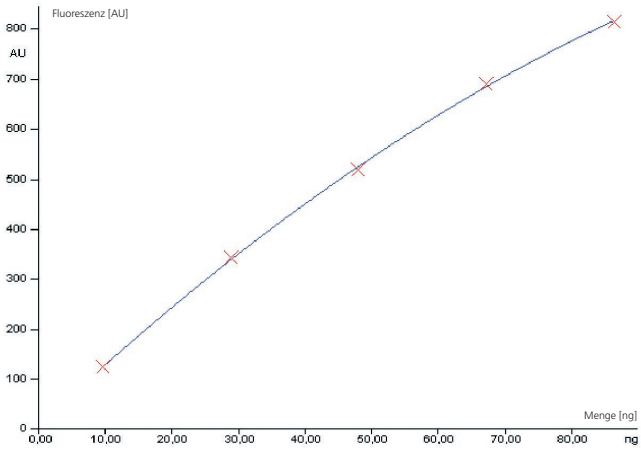
▲ *Chromatogramm zur Bestimmung von ITX in Milch (hR_F 26, interner Standard DTX bei hR_F 20); Beleuchtung unter UV 254/>400 nm; B: Blindwert (Milch mit DTX), S_1-S_5: Standardniveau 1–5, M: dotierte Milchproben*

Densitometrie

TLC-Scanner 3 mit winCATS-Software; Fluoreszenzmessung bei UV 254/>400 nm; polynome Kalibration über die Peakhöhe



26

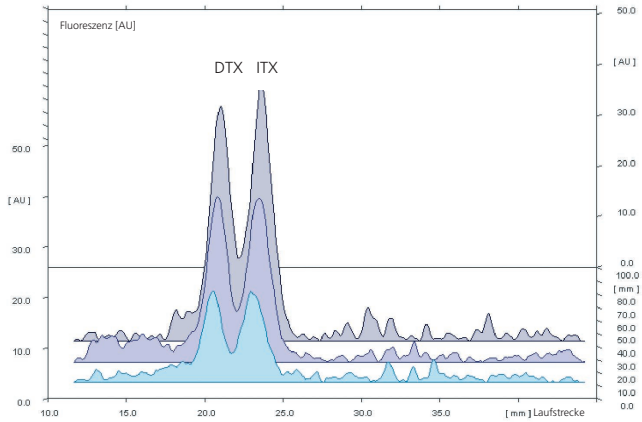
▲ *Polynome Regression von ITX ($y = -0{,}037\ x^2 + 12.610\ x + 5.994$, sdv = ± 1.51 %, r = 0.99981) im Arbeitsbereich von 20–200 µg/kg (9.6–86.4 ng/Zone)*

Dokumentation

Mit DigiStore 2 Dokumentationssystem; Aufnahme bei UV 254/>400 nm

Ergebnisse und Diskussion

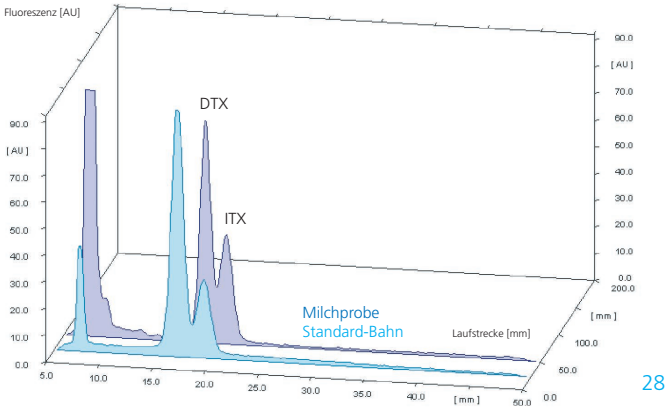
Die Nachweisgrenzen für ITX und DTX liegen jeweils bei 64 pg/Zone (S/N = 3). In fetter Matrix (dotiertes Butterfett) liegt die Nachweisgrenze unter 1 µg/kg.



27

▲ *Überlagerte Bahnen von 64 pg, 128 pg und 192 pg DTX und ITX; die Nachweisgrenzen (S/N = 3) für ITX und DTX liegen beide bei 64 pg und die entsprechenden Bestimmungsgrenzen bei 192 pg.*

Im Arbeitsbereich (20–200 µg/kg) wies die polynome Regression von ITX eine relative Standardabweichung (sdv) von ± 1.51 % (r = 0.99981) auf. Der Variationskoeffizient der Wiederholpräzision der ITX-Bestimmung (n = 9, 32 ng ITX) liegt bei 1.1 %; in dotierten Proben, i.e. Milch, Joghurt, Sojaöl und Margarine, dotiert mit 20, 50 und 100 µg/kg, wurden Variationskoeffizienten (je n = 4) zwischen ± 1.0 und 6.4 % ermittelt. Insgesamte Wiederfindungsraten (n = 8, ermittelt bei 20 bzw. 50 und 100 µg/kg, korrigiert über internen Standard) lagen bei 130 % für Milch und Joghurt sowie 70 % für Margarine und 97 % für Sojaöl.

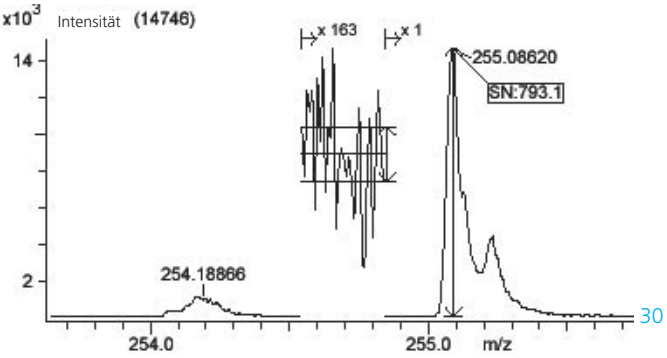


28

▲ *Überlagerung einer ITX Standard-Bahn und einer Milchprobe-Bahn dotiert mit DTX bei 50 µg/kg (interner Standard) und ITX bei 20 µg/kg*

Zur Absicherung positiver Befunde im µg/kg-Bereich wird das ESI-Massensignal der betroffenen ITX-Zonen im SIM–Modus (selective ion monitoring) bei *m/z* 255 und 277 aufgenommen mittels eines Stempelkopf-basierten Extraktors [2].

Als eine weitere Bestätigungsvariante wurde DART [3], welche an ein Flugzeit-Massenspektrometer (TOF/MS) gekoppelt war, eingesetzt. Diese neue Ionenquelle arbeitet unter Umgebungsluft mit einem angeregten Gasstrom. Ihr Einsatz im Bereich der Planar-Chromatographie wurde in ersten Untersuchungen erfolgreich demonstriert.



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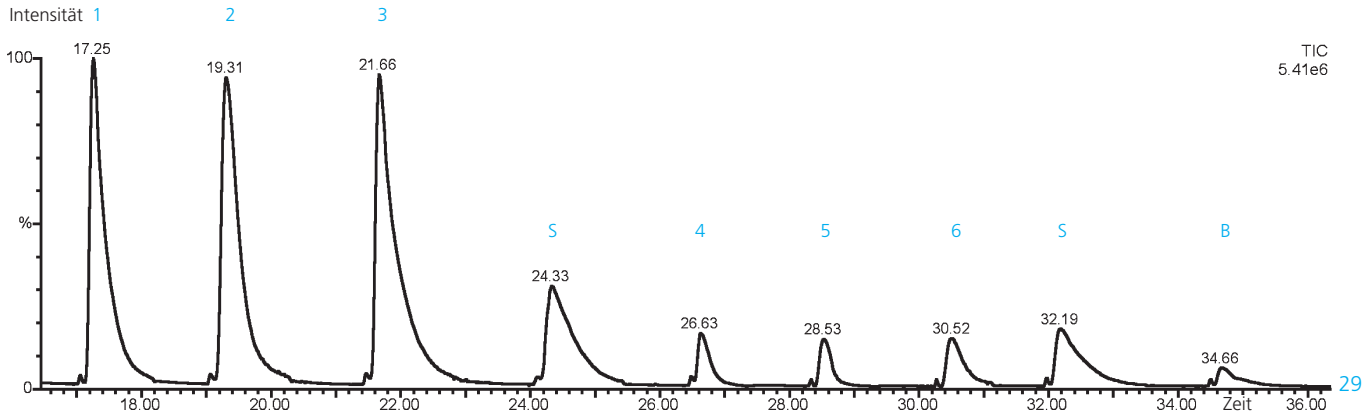
▲ *HPTLC/DART-TOF-Spektrum einer 48 ng ITX-Zone bei m/z 255.0862*

Weitere Informationen sind bei den Autoren auf Anfrage erhältlich.

*Dr. Gerda Morlock, Institut für Lebensmittelchemie, Universität Hohenheim, Garbenstr. 28, D-70599 Stuttgart, gmorlock@uni-hohenheim.de

[1] G. Morlock, W. Schwack, Anal Bioanal Chem im Druck, 2006.
[2] H. Luftmann, Anal Bioanal Chem 378, 964-968, 2004.
[3] R.B. Cody, J.A. Laramee, H.D. Durst, Anal Chem 77, 2297–2302, 2005.

Dank an Herrn Yoshihisa Ueda (JEOL (Europe) S.A.) und Dr. Wiesmann (JEOL (Germany) GmbH) für die Unterstützung bei der Aufnahme der HPTLC-DART/TOF-Spektren.



29

▲ *SIM-Elutionsprofile aufgenommen bei m/z 255 und 277 von 3 ITX-Zonen je in einer Joghurtprobe dotiert mit 100 ppb (Peak 1–3) und 20 ppb (Peak 4–6), daneben Extraktionen von Standardzonen S und Blindwert B*

Bestimmung der Verfälschung von pflanzlichen Arzneimitteln mit Glibenclamid

31

▲ *Herr Faizan Ahmad, Dr. Mazen Ali Naji und Dr. M. Kamil (links nach rechts)*

Im Gesundheitsamt (General Authority for Health Services) des Emirates von Abu Dhabi ist die Abteilung für Pharmakognosie des ZCHRTM (Zayed Complex For Herbal Research & Traditional Medicine) auf die Untersuchung von pflanzlichen und traditionellen Arzneimitteln spezialisiert. Dr. Kamil* und seine Gruppe, unter der Leitung von Dr. Mazen Ali Naji, beschäftigen sich mit der Standardisierung und Qualitätskontrolle von pflanzlichen Arzneimitteln, die vor allem aus »Medizin-Pflanzen« der Vereinigten Arabischen Emirate stammen. Der dünnschicht-chromatographische Fingerprint ist dabei sehr wichtig, um Verfälschungen von pflanzlichen Arzneimitteln mit Arzneistoffen nachzuweisen – nicht nur zwecks Detektion, sondern auch für deren quantitative Bestimmung.

Einführung

Pflanzliche Arzneimittel, die in unterentwickelten Ländern und Entwicklungsländern hergestellt werden und in den Verkehr kommen, werden z. Zt. nicht hinsichtlich aller Qualitätsparameter untersucht. Einige Arzneimittelhersteller nutzen diesen Umstand und verfälschen ihre Produkte mit Arzneistoffen, um die Wirkung des Arzneimittels zu verstärken, jedoch ohne mögliche Nebenwirkungen zu berücksichtigen. Das wiederangestiegene Interesse an pflanzlichen Arzneimitteln und deren zunehmende Vermarktung in den letzten Jahren hat die Qualitätskontrolle und Standardisierung pflanzlicher Drogen und Zubereitungen wichtiger als je zuvor gemacht.

Im Rahmen der Qualitätskontrolle eines antidiabetisch-wirkenden pflanzlichen Arzneimittels wurde Glibenclamid darin gefunden. Glibenclamid ist ist ein antidiabetisch wirkender Arzneistoff und unter Handelsnamen wie Daonil, Euglucon oder Betanase erhältlich. Zur qualitativen als auch quantitativen Bestimmung von Glibenclamid in verfälschten pflanzlichen Arzneimitteln wurde folgende DC-Methode entwickelt.

Die steigende Anzahl von phytochemischen Arzneimittel-Präparationen untermauert die Bedeutung der Standardisierung von pflanzlichen Drogen und deren Zubereitungen. DC, HPLC und UV-Spektroskopie wurden eingesetzt und miteinander verglichen hinsichtlich der Quantifizierung von Glibenclamid. Die DC-Methode zeigte sehr zufriedenstellende Ergebnisse, die vergleichbar mit denen der HPLC und UV-Spektroskopie waren.

Probenvorbereitung

Die Tabletten werden gemahlen; 5 g Tablettenpulver wurde mit 60 mL Methanol im Vortex geschüttelt und anschließend im Ultraschallbad behandelt. Die Lösung wurde 2 min bei 2000 U/min zentrifugiert, filtriert und zu 100 mL mit Methanol aufgefüllt.

Standardlösungen

25 mg Glibenclamid wurde in 25 mL Methanol gelöst (1 mg/mL). Verschiedene Verdünnungen, i.e. 500, 350 und 250 µg/mL, wurden hergestellt.

Schicht

DC-Platten Kieselgel 60 F_{254} (Merck) 20 x 20 cm (bei Verwendung von HPTLC-Platten kann die Auflösung verbessert werden)

Probenauftragung

Je 10 µL der Standard- und Probelösung werden punktförmig mit Nanomat 4 aufgetragen.

Chromatographie

In einer Doppeltrogkammer mit Toluen – Ethylformiat – Ameisensäure 5:4:1 nach Kammersättigung

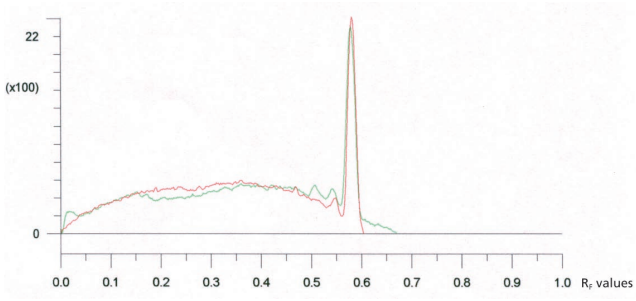
für 30 min; Laufstrecke vom unteren Plattenrand 150 mm (bei Verwendung von HPTLC-Platten wird die Lauftrecke auf 60 mm reduziert und somit auch die Laufzeit).

Dokumentation und Videodensitometrie

Mit VideoStore 2 Dokumentationssystem Aufnahme bei UV 365/>400 nm; Integration und Quantifizierung mittels VideoScan-Software.

32

◀ *DC-Platte beleuchtet mit UV 365 nm; Bahn 1: DC-Fingerprint eines mit Glibenclamid verfälschten pflanzlichen Arzneimittel-Extraktes; Bahn 2 bis 4: Glibenclamid-Standard 250, 350 und 500 µg/mL (die darunterliegende Zone in den Bahnen 2–4 ist eine Verunreinigung im Glibenclamid-Standard)*



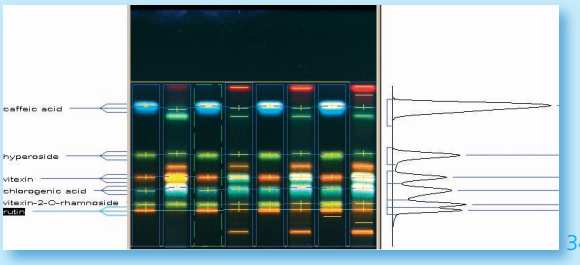
33

▲ *Chromatogramm-Überlagerung des pflanzlichen Arzneimittel-Extraktes (grüne Kurve) und des 250 µg/mL Glibenclamid-Standards (braune Kurve)*

Ergebnisse und Diskussion

Das DC-Chromatogramm eines Extraktes einer antidiabetisch-wirkenden pflanzlichen Arzneimittel-Tablette zeigt deutlich die blaue Zone des Glibenclamid-Standards bei hR_F 58; weitere polare Verbindungen in der Tablette sind auf der Startzone fixiert. Die Konzentration von Glibenclamid in der Droge wurde mit 250 µg/mL bestimmt, i.e. 0.50 % Glibenclamide in der Drogenzubereitung (w/w). Die Ergebnisse mittels UV-Spektroskopie (0.57 %) und HPLC (0.52 %) wiesen denselben Konzentrationsbereich von Glibenclamid im Arzneimittel aus.

*Dr. Mohammad Kamil (Tel 009712-5036214, email: drkamil2005@yahoo.co.in), Abteilungsleiter Pharmakognosie, und Dr. Mazen Ali Naji (Tel 09712-5036400, email: druae@hotmail.com), Generaldirektor, Zaed Complex For Herbal Research & Traditional Medicine (ZCHRTM), General Authority for the Health Services for the Emirates of Abu Dhabi, P.O. Box 29300, Abu Dhabi, United Arab Emirates



34

Digitale Bildauswertung VideoScan

Dr. Kamil, General Authority for the Health Services for the Emirates of Abu Dhabi, und sein Team quantifizieren Glibenclamid, das zur Verfälschung von pflanzlichen Arzneimitteln eingesetzt wird, mittels der VideoScan-Software. VideoScan erlaubt die Auswertung der mit DigiStore oder VideoStore 2 aufgenommenen Bilder. Das Programm ist einfach und schnell in der Bedienung, bietet flexible Anwendungen, z.B. Vergleich von Bahnen verschiedener Chromatogramme, Auswertung von Bahnen mit variierendem Abstand, schräg liegende Messbahnen, usw. Die Chromatogramme können zu beliebiger Zeit, auch noch Jahre nach der Bilderfassung, ausgewertet werden. Die quantitative Auswertung (Option) erfolgt wahlweise über Peakhöhe und/oder Peakfläche, und es besteht die Auswahl zwischen Einstandard- und Mehrbereichs-Kalibrierung (linear oder polynom).

Die wichtigsten Eigenschaften des VideoScan-Programms in Kürze:

- Einfach und schnell in der Bedienung
- Die Integration der Analogkurven kann wahlweise automatisch oder manuell durchgeführt werden.
- Die quantitative Auswertung erfolgt wahlweise über Peakhöhe und/oder Peakfläche.
- Es besteht die Auswahl zwischen Einstandard- und Mehrbereichs-Kalibrierung (linear oder polynom).

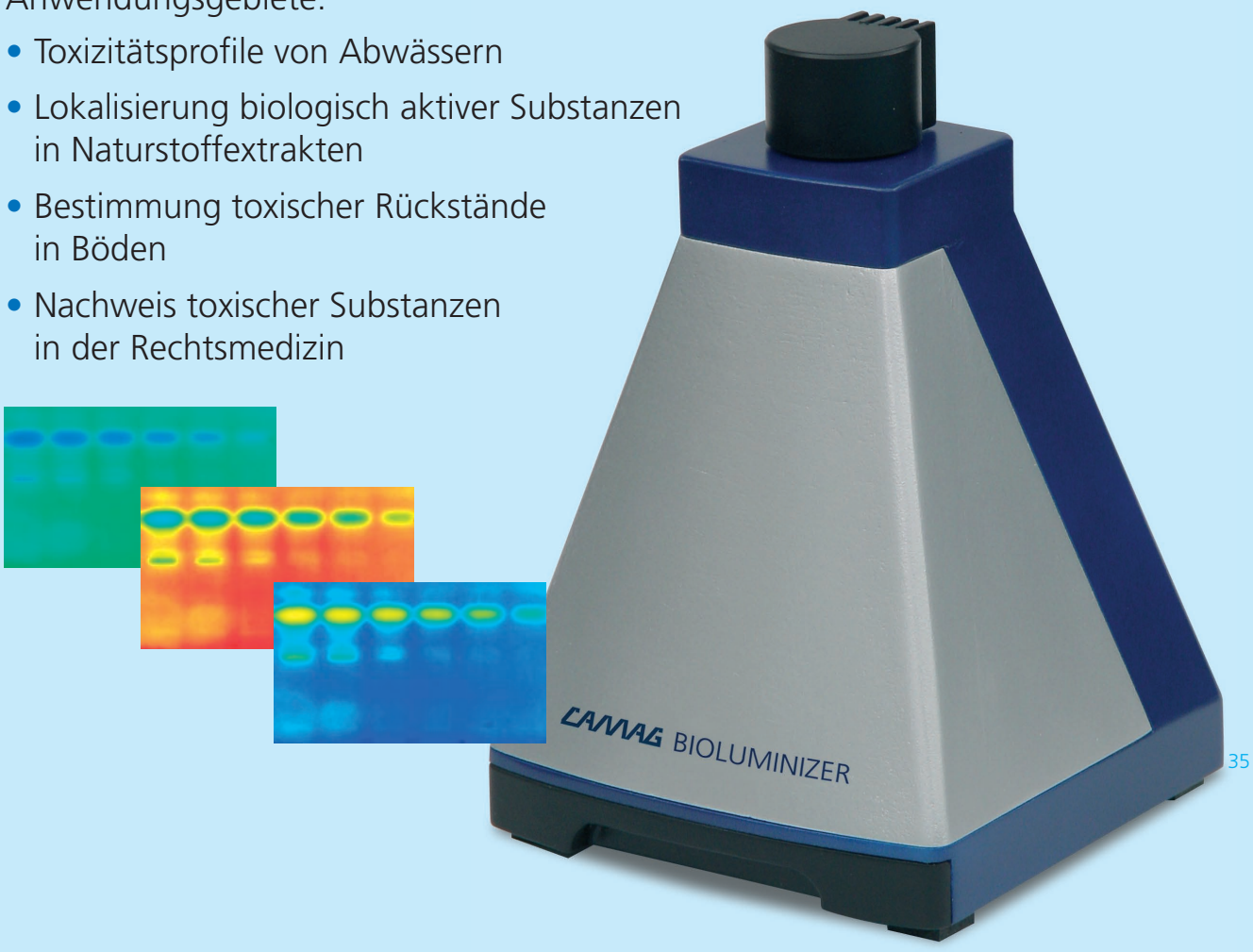
Das VideoScan Programm entspricht den Anforderungen von GMP/GLP und ist IQ/OQ qualifizierbar.

BioLuminizer™: Wirkungsdetektion durch Planar-Chromatographie/ Biolumineszenz-Kopplung

Luminographisches Toxizitätsscreening im Anschluss an eine dünnschicht-chromatographische Trennung ermöglicht die Identifizierung von biologisch aktiven Substanzen in heterogen zusammengesetzten Gemischen

Anwendungsgebiete:

- Toxizitätsprofile von Abwässern
- Lokalisierung biologisch aktiver Substanzen in Naturstoffextrakten
- Bestimmung toxischer Rückstände in Böden
- Nachweis toxischer Substanzen in der Rechtsmedizin



35

BioLuminizer™ wurde spezifisch für diese Anwendung entwickelt. Kompakt und benutzerfreundlich liefert dieses Detektionssystem aussagekräftige Bilder in hoher Bildqualität und Auflösung bei kurzen Belichtungszeiten.

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CBS 96 D 996.1309