



## LIST OF CONTENTS

## Issue 1

## New Methods

A novel double coating for microemulsion electrokinetic chromatography with laser-induced fluorescence detection: as tested with amino acid derivatives

- J. Xie (Lanzhou, PR China and Henan, PR China) X. Chen, J. Zhang (Lanzhou, PR China), J. Liu (Lanzhou, PR China and Sichuan, PR China)  
J. Tian, X. Chen, Z. Hu (Lanzhou, PR China) ..... 1

Electrophoretic analysis of di- and oligosaccharides derived from glycosaminoglycans on microchip format

- Y.-k. Matsuno, M. Kinoshita, K. Kakehi (Higashi-osaka, Japan) ..... 9

A rapid and simple determination of A77 1726 in human serum by high-performance liquid chromatography and its application for optimization of leflunomide therapy

- E.N. van Roon, J.P. Yska, J. Raemaekers, T.L.Th.A. Jansen (Leeuwarden, The Netherlands), M. van Wanrooy, J.R.B.J. Brouwers (Leeuwarden, The Netherlands and Groningen, The Netherlands) ..... 17

## Pharmaceutical Applications

HPTLC method for guggulsterone. II. Stress degradation studies on guggulsterone

- H. Agrawal, N. Kaul, A.R. Paradkar, K.R. Mahadik (Pune, India) ..... 23

HPTLC method for guggulsterone. I. Quantitative determination of E- and Z-guggulsterone in herbal extract and pharmaceutical dosage form

- H. Agrawal, N. Kaul, A.R. Paradkar, K.R. Mahadik (Pune, India) ..... 33

Improved detection of Coptidis alkaloids by field-amplified sample stacking in capillary electrophoresis

- S.-W. Sun, H.-M. Tseng (Taipei, Taiwan) ..... 43

Chloride-selective membrane electrodes and optodes based on an indium(III) porphyrin for the determination of chloride in a sequential injection analysis system

- A.M. Pimenta, A.N. Araújo, M.C.S.M. Montenegro (Porto, Portugal), C. Pasquini, J.J.R. Rohwedder, I.M. Raimundo Jr. (Campinas, Brazil) ..... 49

Development and validation of a fully automated method for the chromatographic determination of content uniformity of drug tablets

- I. Toro, J.F. Dulsat, J.L. Fábregas, J. Claramunt (Barcelona, Spain) ..... 57

Method development for betamethasone and dexamethasone by micellar liquid chromatography using cetyl trimethyl ammonium bromide and validation in tablets. Application to cocktails

- D. Peña-García-Brioles, R. Gonzalo-Lumbreras, R. Izquierdo-Hornillos, A. Santos-Montes (Madrid, Spain) ..... 65

Development and validation of a novel LC/ELSD method for the quantitation of gentamicin sulfate components in pharmaceuticals

- N.C. Megoulas, M.A. Koupparis (Athens, Greece) ..... 73

Identification of 5-aminosalicylic acid, ciprofloxacin and azithromycin by abrasive stripping voltammetry

- Š. Komorsky-Lovrić, B. Nigović (Zagreb, Croatia) ..... 81

Comparison of fluorimetric, voltammetric and biosensor methods for the determination of total antioxidant capacity of drug products containing acetylsalicylic acid

- L. Campanella, A. Bonanni, D. Bellantoni, G. Favero, M. Tomassetti (Rome, Italy) ..... 91

## Biomedical Applications

Study of urinary 8-hydroxydeoxyguanosine as a biomarker of oxidative DNA damage in diabetic nephropathy patients

- G.W. Xu, Q.H. Yao, Q.F. Weng, B.L. Su, X. Zhang, J.H. Xiong (Dalian, China) ..... 101

The development of a higher throughput reactive intermediate screening assay incorporating micro-bore liquid chromatography–micro-electrospray ionization–tandem mass spectrometry and glutathione ethyl ester as an in vitro conjugating agent J.R. Soglia, S.P. Harriman, S. Zhao, J. Barberia, M.J. Cole, J.G. Boyd, L.G. Contillo (Groton, CT, USA) . . . . .	105
Simultaneous determination of cefepime and grepafloxacin in human urine by high-performance liquid chromatography J.A. Ocaña González, F.J. Jiménez Palacios, M. Callejón Mochón, F.J. Barragán de la Rosa (Seville, Spain) . . . . .	117
Rapid analysis of docetaxel in human plasma by tandem mass spectrometry with on-line sample extraction A.G. Grozav, T.E. Hutson, X. Zhou, R.M. Bukowski, R. Ganapathi, Y. Xu (Cleveland, OH, USA) . . . . .	125
Development and validation of a high-performance liquid chromatographic method for the determination of cyproterone acetate in human skin S. Henry de Hassonville, P. Chiap, J.-F. Liégeois, B. Evrard, L. Delattre, J. Crommen, G. Piel, P. Hubert (Liège, Belgium) . . . . .	133
<b>Short Communications</b>	
Determination of lansoprazole in pharmaceutical capsules by flow injection analysis using UV-detection D. Yeniceli, D. Dogrukol-Ak, M. Tuncel (Eskisehir, Turkey) . . . . .	145
Adsorptive stripping voltammetric quantification of the antipsychotic drug clozapine in bulk form, pharmaceutical formulation and human serum at a mercury electrode E. Hammam, A. Tawfik, M.M. Ghoneim (Tanta, Egypt) . . . . .	149
Polarographic behaviour of Aceclofenac, Tenoxicam and Droxicam in a methanol–water mixture J.A. Acuña, M.D. Vázquez, M.L. Tascón, P. Sánchez-Batanero (Valladolid, Spain) . . . . .	157
Simple and rapid HPLC method for determination of amlodipine in human serum with fluorescence detection and its use in pharmacokinetic studies G. Bahrami, S. Mirzaei (Kermanshah, Iran) . . . . .	163
Determination of nateglinide in animal plasma by micellar electrokinetic chromatography and on-line sweeping technique H. Yan, G. Yang (Beijing, China and Baoding, China), F. Qiao (Baoding, China), Y. Chen (Beijing, China) . . . . .	169
A validated solid-phase extraction HPLC method for the simultaneous determination of the citrus flavanone aglycones hesperetin and naringenin in urine F.I. Kanaze, E. Kokkalou, M. Georgarakis, I. Niopas (Thessaloniki, Greece) . . . . .	175
Rapid quantitative analysis of oxiracetam in human plasma by liquid chromatography/electrospray tandem mass spectrometry J. Son, J. Lee, M. Lee, E. Lee (Seoul, South Korea), K. Tae Lee (Seoul, Republic of Korea), S. La, D.-H. Kim (Seoul, South Korea) . . . . .	183
Automated 96-well liquid–liquid back extraction liquid chromatography–tandem mass spectrometry method for the determination of ABT-202 in human plasma N. Xu, G.E. Kim, H. Gregg, A. Wagdy, B.A. Swaine, M.S. Chang, T.A. El-Shourbagy (Abbott Park, IL, USA) . . . . .	189
Ion-pair complex-based solvent extraction combined with chemiluminescence determination of chlorpromazine hydrochloride with luminol in reverse micelles W. Shi, J. Yang, Y. Huang (Chongqing, PR China) . . . . .	197
HPLC study of glimepiride under hydrolytic stress conditions P. Kováříková, J. Klimeš (Hradec Králové, Czech Republic), J. Dohnal, L. Tisovská (Hradec Králové, Czech Republic and Dolní Mečolupy, Czech Republic) . . . . .	205
Analysis of fluorescence excitation–emission matrix of multicomponent drugs. A case study with human placental extract used as wound healer P. Datta, D. Bhattacharyya (Calcutta, India) . . . . .	211
Determination of dopamine by flow-injection analysis coupled with luminol-hexacyanoferrate(III) chemiluminescence detection E. Nalewajko, R. Bort Ramírez, A. Kojlo (Białystok, Poland) . . . . .	219
An expression of within-plate uncertainty in sandwich ELISA Y. Hayashi, R. Matsuda, T. Maitani, K. Ito, W. Nishimura, K. Imai, M. Maeda (Tokyo, Japan) . . . . .	225
Identification and characterization of major degradation products of risperidone in bulk drug and pharmaceutical dosage forms R.S. Tomar, T.J. Joseph, A.S.R. Murthy, D.V. Yadav, G. Subbaiah and K.V.S.R. Krishna Reddy (Gandhinagar, India) . . . . .	231
Chemiluminescence detection of isoniazid using Ru(phen) <sub>3</sub> <sup>2+</sup> –isoniazid–Ce(IV) system J. Xi (Wuhan, PR China), B. Shi (Wuhan, PR China and Enshi, PR China), X. Ai, Z. He (Wuhan, PR China) . . . . .	237
HPLC method for simultaneous determination of cefprozil diastereomers in human plasma T.-H. Park, J.-K. Kim, J.-P. Jee, J.-S. Park, C.-K. Kim (Seoul, Republic of Korea) . . . . .	243

## Issue 2

**New Methods**

- UV spectroscopy and reverse-phase HPLC as novel methods to determine Capreomycin of liposomal fomulations  
C. Rossi, G. Fardella, I. Chiappini, L. Peroli, C. Vescovi, M. Ricci, S. Giovagnoli, S. Scuota (Perugia, Italy) ..... 249

- Fast separation of bacitracin on monolithic silica columns  
V. Pavli, V. Kmetec (Ljubljana, Slovenia) ..... 257

**Pharmaceutical Applications**

- Identification of low-level degradants from low dose tablets  
A. Blanchard, C. Lee, B. Nickerson, L.L. Lohr, A.J. Jensen, K.M. Alsante, T.R. Sharp, D.P. Santafianos, R. Morris (Groton, CT, USA), K.D. Snyder (San Mateo, CA, USA) ..... 265

- Rapid analysis of liquid formulations containing sodium chloride using laser-induced breakdown spectroscopy  
L. St-Onge (Boucherville, Canada), E. Kwong (Pointe-Claire-Dorval, Canada), M. Sabsabi (Boucherville, Canada), E.B. Vadas (Dorval, Canada) ..... 277

- Validation of the COSTIM bioassay for dendritic cell potency  
G. Shankar, M.S. Fourrier, M.A. Grevenkamp, P.A. Lodge (Bothell, WA, USA) ..... 285

- Microcalorimetric evaluation of the in vitro compatibility of amoxicillin/clavulanic acid and ampicillin/sulbactam with ciprofloxacin  
R. Chadha, N. Kashid, D.V.S. Jain (Chandigarh, India) ..... 295

- An HPLC method to evaluate purity of a steroidal drug, loteprednol etabonate  
S.-i. Yasueda, M. Higashiyama, Y. Shirasaki, K. Inada, A. Ohtori (Osaka, Japan) ..... 309

- Identification and quantification method of spiramycin and tylosin in feedingstuffs with HPLC-UV/DAD at 1 ppm level  
C. Civitareale, M. Fiori, A. Ballerini, G. Brambilla (Rome, Italy) ..... 317

- Combined effect of pH and polysorbates with cyclodextrins on solubilization of naringenin  
S. Tommasini, M.L. Calabrò, D. Raneri, P. Ficarra, R. Ficarra (Messina, Italy) ..... 327

- Use of in situ FT-Raman spectroscopy to study the kinetics of the transformation of carbamazepine polymorphs  
L.E. O'Brien (Bradford, UK), P. Timmins (Merseyside, UK), A.C. Williams, P. York (Bradford, UK) ..... 335

- Determination of cetirizine dihydrochloride, related impurities and preservatives in oral solution and tablet dosage forms using HPLC  
A.M.Y. Jaber (Dhahran, Saudi Arabia), H.A. Al Sherife, M.M. Al Omari, A.A. Badwan (Jordan) ..... 341

**Biomedical Applications**

- A rapid HPLC-DAD method for the analysis of fluoxetine and norfluoxetine in plasma from overdose patients  
C. Sabbioni, F. Bugamelli, G. Varani (Ferrara, Italy), L. Mercolini, A. Musenga, M.A. Saracino, S. Fanali (Rome, Italy), M.A. Raggi (Bologna, Italy) ..... 351

- Micellar liquid chromatography determination of some biogenic amines with electrochemical detection  
D. Bose, A. Durbanshi, M.E. Capella-Pérez, M. Gil-Agustí, J. Esteve-Romero, S. Carda-Broch (Castelló, Spain) ..... 357

- Determination of icariin and metabolites in rat serum by capillary zone electrophoresis: rat pharmacokinetic studies after administration of icariin  
J. Liu, Y.-j. Lou (Hangzhou, China) ..... 365

- HPLC-UV method for assaying 99/357, a synthetic trioxane antimalarial derivative in rat and rabbit serum  
S.K. Singh, R.P. Singh, R.C. Gupta (Lucknow, India) ..... 371

**Short Communications**

- Rapid determination of minoxidil in human plasma using ion-pair HPLC  
A. Zarghi, A. Shafaati, S.M. Foroutan, A. Khoddam (Tehran, Iran) ..... 377

- Solid-phase microextraction-gas chromatography-mass spectrometric analysis of volatile compounds in a compounded Chinese medicinal prescription, Xiao-Cheng-Qi-Tang  
Y. Sha, S. Shen, G. Duan (Shanghai, PR China) ..... 381

- Quantification intracellular levels of oligodeoxynucleotide-doxorubicin conjugate in human carcinoma cells in situ  
Y. Ren (Shanghai, China and Nanchang, China), D. Wei (Shanghai, China) ..... 387

Comparison between sodium dodecylsulphate and cetyltrimethylammonium bromide as mobile phases in the micellar liquid chromatography determination of non-steroidal anti-inflammatory drugs in pharmaceuticals C. Martínez-Algaba, L. Escuder-Gilabert, S. Sagrado, R.M. Villanueva-Camañas, M.J. Medina-Hernández (Valencia, Spain) . . . . .	393
Validation of a generic analytical procedure for determination of residual solvents in drug substances S. Klick, A. Sköld (Mölndal, Sweden) . . . . .	401
<sup>19</sup> F NMR spectroscopic study on the binding of trifluopromazine to bovine and human serum albumins K. Kitamura, M. Kume, M. Yamamoto, S. Takegami, T. Kitade (Kyoto, Japan) . . . . .	411
A method for fast determination of psoralens in oral solutions of phytomedicines using liquid chromatography A.E. Pires, N.K. Honda (Campo Grande/MS, Brazil), C.A.L. Cardoso (Dourados/MS, Brazil) . . . . .	415
LC-MS determination and bioavailability study of loperamide hydrochloride after oral administration of loperamide capsule in human volunteers J.H. Yu, H.J. Kim, S. Lee, S.-J. Hwang (Daejeon, South Korea), W. Kim (Kyonggi-do, South Korea), C.J. Moon (Seoul, South Korea) . . . . .	421

### Issue 3

#### New Methods

Analysis of C-3 epimerization in (24 <i>R</i> )-24,25-dihydroxyvitamin D <sub>3</sub> catalyzed by hydroxysteroid dehydrogenase T. Higashi, K. Sakajiri, K. Shimada (Kanazawa, Japan) . . . . .	429
Optimization and validation of a micellar electrokinetic chromatographic method for the analysis of florfenicol S. Hillaert, W. Van den Bossche (Ghent, Belgium) . . . . .	437
Analytical characterization of heparin by capillary zone electrophoresis with conductivity detection and polymeric buffer additives P. Mikuš, I. Valášková, E. Havránek (Bratislava, Slovak Republic) . . . . .	441
Automated robotic liquid handling/laser-based nephelometry system for high throughput measurement of kinetic aqueous solubility K.A. Dehring, H.L. Workman, K.D. Miller, A. Mandagere, S.K. Poole (Ann Arbor, MI, USA) . . . . .	447
A method for the direct analysis of drug compounds in plasma using a single restricted access material (RAM) column R. Papp, W.M. Mullett, E. Kwong (Kirkland, Canada) . . . . .	457

The characterisation of selected drugs with amine-containing side chains using electrospray ionisation and ion trap mass spectrometry and their determination by HPLC-ESI-MS

C. Joyce, W.F. Smyth, V.N. Ramachandran, E. O'Kane, D.J. Coulter (Northern Ireland, UK) . . . . .	465
---	-----

Superheated water chromatography-nuclear magnetic resonance spectroscopy and mass spectrometry of vitamins

O. Chienthavorn, R.M. Smith, S. Saha (Leics, UK), I.D. Wilson, B. Wright, S.D. Taylor, E.M. Lenz (Cheshire, UK) . . . . .	477
---	-----

#### Pharmaceutical Applications

Molecularly imprinted solid phase extraction-pulsed elution-mass spectrometry for determination of cephalexin and $\alpha$ -aminocephalosporin antibiotics in human serum S.G. Wu, E.P.C. Lai, P.M. Mayer (Ottawa, Canada) . . . . .	483
Sensitive chemiluminescence assay for risperidone in pharmaceutical preparations Z. Song, C. Wang (Xi'an, China) . . . . .	491
The effect of preprocessing methods in reducing interfering variability from near-infrared measurements of creams J. Luyperaert (Brussel, Belgium), S. Heuerding (Basel, Switzerland), Y.V. Heyden, D.L. Massart (Brussel, Belgium) . . . . .	495
Simple, sensitive and rapid LC-MS/MS method for the quantitation of cerivastatin in human plasma — application to pharmacokinetic studies N.V.S. Ramakrishna, M. Koteshwara, K.N. Vishwottam, S. Puran, S. Manoj, M. Santosh (Hyderabad, India) . . . . .	505
Development and validation of an assay for citric acid/citrate and phosphate in pharmaceutical dosage forms using ion chromatography with suppressed conductivity detection B.M. DeBorba, J.S. Rohrer (Sunnyvale, CA, USA), L. Bhattacharyya (Rockville, MD, USA) . . . . .	517
Analysis of undeclared synthetic phosphodiesterase-5 inhibitors in dietary supplements and herbal matrices by LC-ESI-MS and LC-UV S.R. Gratz, C.L. Flurer, K.A. Wolnik (Cincinnati, OH, USA) . . . . .	525

Development and validation of a high-performance liquid chromatography assay and a capillary electrophoresis assay for the analysis of adenosine and the degradation product adenine in infusions P. Kießling, G.K. E. Scriba, F. Süß, G. Werner, H. Knoth, M. Hartmann (Jena, Germany) . . . . .	535
Simultaneous determination of levodopa and benserazide by stopped-flow injection analysis and three-way multivariate calibration of kinetic-spectrophotometric data M. Pistonesi, M.E. Centurión, B.S.F. Band (Bahía Blanca, Argentina), P.C. Damiani, A.C. Olivier (Rosario, Argentina) . . . . .	541
Automated simultaneous triple dissolution profiles of two drugs, sulphamethoxazole-trimethoprim and hydrochlorothiazide-captopril in solid oral dosage forms by a multicommutation flow-assembly and derivative spectrophotometry D. Tomšů, M. Catalá Icardo, J. Martínez Calatayud (Valencia, Spain) . . . . .	549
Characterization of the preferred orientation of δ-mannitol crystallites in tablets M. Koivisto, T. Virjalon, T. Heikkilä, V.-P. Lehto (Turku, Finland) . . . . .	559
A liquid chromatographic method for simultaneous determination of amoxicillin sodium and sulbactam sodium in a combination formulation P. Wang (Shenyang, China), M. Qi (Beijing, China), Y. Sun (Shenyang, China), J. Yang (Xingtai, China) . . . . .	565
Solubility and phase separation of benzocaine and salicylic acid in 1,4-dioxane–water mixtures at several temperatures M.A. Peña, P. Bustamante, B. Escalera, A. Refillo (Madrid, Spain), J.M. Bosque-Sendra (Granada, Spain) . . . . .	571
Harmonization of strategies for the validation of quantitative analytical procedures. A SFSTP proposal—part I Ph. Hubert (Liège, Belgium), J.-J. Nguyen-Huu (Vitry sur Seine, France), B. Boulanger (Mont-Saint-Guibert, Belgium), E. Chapuzet (Olivet, France), P. Chiap (Liège, Belgium), N. Cohen (Epernon, France), P.-A. Compagnon (St. Denis, France), W. Dewé (Mont-Saint-Guibert, Belgium), M. Feinberg (Paris, France), M. Lallier (Arcueil, France), M. Laurentie (Fougères, France), N. Mercier (Olivet, France), G. Muzard (Monaco, France), C. Nivet (Baugy, France), L. Valat (Mérignac, France) . . . . .	579
Flow injection-chemiluminescence determination of puerarin in pharmaceutical preparations Q. Zhang, A. Myint, L. Liu, X. Ge, H. Cui (Anhui, PR China) . . . . .	587
Acid catalysed degradation of some spiramycin derivatives found in the antibiotic bitespiramycin X. Shi, S. Zhang (Shenyang, PR China), J.P. Fawcett (Dunedin, New Zealand), D. Zhong (Shenyang, PR China) . . . . .	593
<b>Bioanalytical Applications</b>	
Degradation and configurational changes of thioridazine 2-sulfoxide C.M. de Gaitani, A.S. Martinez, P.S. Bonato (Ribeirão Preto, Brazil) . . . . .	601
Stereoselective analysis of carvedilol in human plasma using HPLC/MS/MS after chiral derivatization E. Yang, S. Wang, J. Kratz, M.J. Cyronak (King of Prussia, PA, USA) . . . . .	609
<b>Short Communications</b>	
HPTLC determination of caffeine in stimulant herbal products and power drinks E.A. Abourashed, J.S. Mossa (Riyadh, Saudi Arabia) . . . . .	617
Determination of methylxanthines in urine by liquid chromatography with diode array UV detection C.G. Zambonin, A. Aresta, F. Palmisano (Bari, Italy) . . . . .	621
Simultaneous HPLC determination of ketoprofen and its degradation products in the presence of preservatives in pharmaceuticals J. Dvořák, R. Hájková, L. Matysová, L. Nováková (Hradec Králové, Czech Republic), M.A. Koupparis (Athens, Greece), P. Solich (Hradec Králové, Czech Republic) . . . . .	625
Voltammetric determination of pyridoxine (Vitamin B <sub>6</sub> ) by use of a chemically-modified glassy carbon electrode W. Qu (Wuhan, PR China and Hubei, PR China), K. Wu, S. Hu (Wuhan, PR China) . . . . .	631
High-performance liquid chromatographic method for simultaneous determination of baicalein and baicalein 7-glucuronide in rat plasma L. Zhang, G. Lin, Z. Zuo (New Territories, Hong Kong) . . . . .	637
Interference of isonicotinyl hydrazone in the microbiological analysis of rifampicin from anti-tuberculosis FDC products containing isoniazid T.T. Mariappan (Punjab, India), T. Geetha, R. Pandey (Chandigarh, India), K.C. Jindal, S. Singh (Punjab, India) . . . . .	643
HPLC determination of mycophenolic acid and mycophenolic acid glucuronide in human plasma with hybrid material M. Bolon (Lyon, France and Bron, France), L. Jeanpierre (Bron, France), M. El Barkil (Lyon, France and Bron, France), K. Chelbi, M. Sauviat (Bron, France), R. Boulieu (Lyon, France and Bron, France) . . . . .	649

Determination of cobalt in pharmaceutical products A.-I. Stoica, M. Peltea, G.-E. Baiulescu, M. Ionica (Bucharest, Romania) . . . . .	653
Rapid quantitative analysis of oxiracetam in human plasma by liquid chromatography/electrospray tandem mass spectrometry J. Son, J. Lee, M. Lee, E. Lee, J.H. Lee, S. La, D.-H. Kim (Seoul, South Korea) . . . . .	657
Indirect photometric assay determination of gabapentin in bulk drug and capsules by capillary electrophoresis R. Sekar, S. Azhaguel (Hyderabad, India) . . . . .	663
Rapid and selective UV spectrophotometric and RP-HPLC methods for dissolution studies of oxybutynin immediate-release and controlled-release formulations M.V.S. Varma, A.M. Kaushal, S. Garg (Punjab, India) . . . . .	669

## Issue 4

### **Review**

An analytical perspective on favoured synthetic routes to the psychoactive tryptamines S.D. Brandt, S. Freeman, P. McGagh, N. Abdul-Halim, J.F. Alder (Manchester, UK) . . . . .	675
---	-----

### **New Methods**

FI-chemiluminometric study of thiazides by on-line photochemical reaction M. Ciborowski, M.C. Icardo, J.V.G. Mateo, J. Martínez Calatayud (Valencia, Spain) . . . . .	693
--	-----

Ion selective electrodes for penicillin-G based on Mn(III)TPP-Cl and their application in pharmaceutical formulations control by sequential injection analysis E.M.G. Santos, A.N. Araújo (Porto, Portugal), C.M.C.M. Couto (Porto, Portugal and Paredes, Portugal), M.C.B.S.M. Montenegro (Porto, Portugal), A. Kejzlarová, P. Solich (Hradec Králové, Czech Republic) . . . . .	701
--	-----

Plasmon-waveguide resonance spectroscopy applied to three potential drug targets: cyclooxygenase-2, hepatitis C virus RNA polymerase and integrin $\alpha V\beta 3$ S. Devanathan (Tucson, AZ, USA), M.C. Walker (Chesterfield, MO, USA), Z. Salamon, G. Tollin (Tucson, AZ, USA) . . . . .	711
--	-----

Simultaneous determination of dysprosium and iron in urine by capillary zone electrophoresis coupled to cloud point extraction C. Ortega (San Luis, Argentina), S. Cerutti, R.A. Olsina, L.D. Martínez, M.F. Silva (San Luis, Argentina and CONICET, República Argentina) . . . . .	721
--	-----

### **Pharmaceutical Applications**

Separation and determination of synthetic impurities of difloxacin by reversed-phase high-performance liquid chromatography R.N. Rao, V. Nagaraju (Hyderabad, India) . . . . .	729
---	-----

Assay for the simultaneous determination of acetaminophen–caffeine–butalbital in human serum using a monolithic column C. Pistros, J.T. Stewart (Athens, GA, USA) . . . . .	737
--	-----

Direct determination of diclofenac in pharmaceutical formulations containing B vitamins by using UV spectrophotometry and partial least squares regression M.M. Sena, Z.F. Chaudhry, C.H. Collins, R.J. Poppi (Campinas, Brazil) . . . . .	743
---	-----

Development of a liquid chromatographic method for ear drops containing neomycin sulphate, polymyxin B sulphate and dexamethasone sodium phosphate M. Pendela, E. Adams, J. Hoogmartens (Leuven, Belgium) . . . . .	751
--	-----

Development and validation of a liquid chromatographic method for determination of related-substances of mosapride citrate in bulk drugs and pharmaceuticals R. Nageswara Rao, D. Nagaraju, S.N. Alvi (Hyderabad, India), S.B. Bhirud (Mumbai, India) . . . . .	759
--	-----

ICH guidance in practice: establishment of inherent stability of secnidazole and development of a validated stability-indicating high-performance liquid chromatographic assay method M. Bakshi, S. Singh (Punjab, India) . . . . .	769
--	-----

Near infrared spectroscopy for qualitative comparison of pharmaceutical batches Y. Roggo, C. Roeseler, M. Ulmschneider (Basel, Switzerland) . . . . .	777
--	-----

Development and validation of a LC-method for determination of catechin and epicatechin in aqueous extractives from leaves of <i>Maytenus ilicifolia</i> L.A.L. Soares (Natal, Brazil and Porto Alegre, Brazil), A.L. Oliveira, G.G. Ortega, P.R. Petrovick (Porto Alegre, Brazil) . . . . .	787
---	-----

Investigation of radio sterilization and dosimetric features of sulfacetamide sodium Ş. Çolak, M. Korkmaz (Ankara, Turkey) . . . . .	791
---	-----

Separation of neutral dihydropyridines and their enantiomers using electrokinetic chromatography A. Van Eeckhaut, M.R. Detaevernier, Y. Michotte (Brussels, Belgium) . . . . .	799
<b>Bioanalytical Applications</b>	
In vitro microdialysis sampling of docetaxel V.J.A. Schuck, I. Rinas, H. Derendorf (Gainesville, FL, USA) . . . . .	807
An API LC/MS/MS quantitation method for ansamitocin P-3 (AP3) and its preclinical pharmacokinetics Z. Liu (Columbus, OH, USA), H.G. Floss (Seattle, WA, USA), J.M. Cassady, J. Xiao, K.K. Chan (Columbus, OH, USA) . . . . .	815
Evaluation of metabolic variation in normal rat strains from a statistical analysis of $^1\text{H}$ NMR spectra of urine T.M.D. Ebbels, E. Holmes, J.C. Lindon, J.K. Nicholson (London, UK) . . . . .	823
Determination of saquinavir and ritonavir in human plasma by reversed-phase high-performance liquid chromatography and the analytical error function V. Albert, P. Modamio, C.F. Lastra, E.L. Mariño (Barcelona, Spain) . . . . .	835
Metabonomics, dietary influences and cultural differences: a $^1\text{H}$ NMR-based study of urine samples obtained from healthy British and Swedish subjects E.M. Lenz, J. Bright, I.D. Wilson, A. Hughes, J. Morrisson (Macclesfield, UK), H. Lindberg (Lund, Sweden), A. Lockton (Macclesfield, UK)	841
DNA-modified carbon paste electrode applied to the study of interaction between Rifampicin (RIF) and DNA in solution and at the electrode surface S.T. Girousi, I.Ch. Gherghi, M.K. Karava (Thessaloniki, Greece) . . . . .	851
<b>Short Communications</b>	
Analysis of plant extracts by NIRS: simultaneous determination of kavapyrones and water in dry extracts of <i>Piper methysticum</i> Forst. M. Gaub, Ch. Roeseler, G. Roos, K.-A. Kovar (Tuebingen, Germany) . . . . .	859
Development of a rapid RP-HPLC method for the determination of clonazepam in human plasma I.F. Bares, F. Pehourcq, C. Jarry (Bordeaux, France) . . . . .	865
A high-performance liquid chromatographic method for determination of praziquantel in plasma W. Hanpitakpong, V. Banmairuoi, B. Kamanikom, A. Choemung, K. Na-Bangchang (Pathumthani, Thailand) . . . . .	871
Simple and rapid determination of adenosine in human synovial fluid with high performance liquid chromatography-mass spectrometry L.-F. Huang, F.-Q. Guo, Y.-Z. Liang, B.-Y. Li, B.-M. Cheng (Changsha, PR China) . . . . .	877
A liquid chromatographic-tandem mass spectrometric method for the determination of YM466, a novel Factor Xa inhibitor, in rat plasma Y. Mano, T. Usui, H. Kamimura (Tokyo, Japan) . . . . .	883
On-line assay of the S-enantiomers of enalapril, ramipril and pentopril using a sequential injection analysis/amperometric biosensor system R.-I. Stefan, J.F. Staden (Pretoria, South Africa), C. Bala (Bucharest, Romania), H.Y. Aboul-Enein (Riyadh, Saudi Arabia) . . . . .	889
A validated cold vapour-AAS method for determining mercury in human red blood cells O.S. Ertas, H. Tezel (Izmir, Turkey) . . . . .	893
Adsorptive properties of cefpodoxime proxetil as a tool for a new method of its determination in urine M. Aleksić, M. Ilić, V. Kapetanović (Belgrade, Serbia and Montenegro) . . . . .	899
Overestimation of rifampicin during colorimetric analysis of anti-tuberculosis products containing isoniazid due to formation of isonicotinyl hydrazone T.T. Mariappan, K.C. Jindal, S. Singh (Punjab, India) . . . . .	905
Determination of rosiglitazone in coated tablets by MEKC and HPLC methods P. Gomes, J. Sippel, A. Jablonski, M. Steppe (Porto Alegre, Brazil) . . . . .	909
Study of the interaction between monoammonium glycyrrhizinate and bovine serum albumin Y.-J. Hu, Y. Liu (Wuhan, PR China), J.-B. Wang, X.-H. Xiao (Beijing, PR China), S.-S. Qu (Wuhan, PR China) . . . . .	915
MMSPE-RP-HPLC method for the simultaneous determination of methimazole and selected metabolites in fish homogenates L. Hollosi, A. Kettrup, K.-W. Schramm (Neuherberg, Germany) . . . . .	921
Determination and in-process control of zolpidem synthesis by high-performance liquid chromatography L. Laviana, C. Mangas (Oviedo, Spain), F. Fernández-Marí, M. Bayod (Madrid, Spain), D. Blanco (Oviedo, Spain) . . . . .	925

**Corrigendum**

Corrigendum to "A validated assay for measuring doxorubicin in biological fluids and tissues in an isolated lung perfusion model: matrix effect and heparin interference strongly influence doxorubicin measurements" [J. Pharm. Biomed. Anal. 33 (2003) 475–494]

A. Kümmerle, T. Krueger, M. Dusmet, C. Vallet, Y. Pan, H.B. Ris, L.A. Decosterd (Lausanne, Switzerland) ..... 929

**Issue 5****Review**

The sacred cow: the questionable role of assay methods in characterising the quality of bulk pharmaceuticals  
S. Görög (Budapest, Hungary) ..... 931

**New Methods**

A rapid and simple procedure for the determination of cannabinoids in hemp food products by gas chromatography-mass spectrometry  
M. Pellegrini, E. Marchei, R. Pacifici, S. Pichini (Rome, Italy) ..... 939

Integrated on-line sample clean-up using cation exchange restricted access sorbent for the LC determination of atropine in human plasma coupled to UV detection

O. Rbeida, B. Christiaens, Ph. Hubert (Liège, Belgium), D. Lubda (Darmstadt, Germany), K.-S. Boos (Munich, Germany), J. Crommen, P. Chiap (Liège, Belgium) ..... 947

Near-infrared (NIR) spectroscopy for the non-destructive and fast determination of geographical origin of Angelicae gigantis Radix  
Y.-A. Woo, H.-J. Kim, K.-R. Ze, H. Chung (Seoul, South Korea) ..... 955

Development and validation of a fully automated LC method for the determination of cloxacillin in human plasma using anion exchange restricted access material for sample clean-up

O. Rbeida, P. Chiap (Liège, Belgium), D. Lubda (Darmstadt, Germany), K.-S. Boos (Munich, Germany), J. Crommen, Ph. Hubert (Liège, Belgium) ..... 961

Chemiluminescence determination of amikacin based on the inhibition of the luminol reaction catalyzed by copper

J.M. Ramos Fernández, J.M. Bosque-Sendra, A.M. García-Campaña, F. Alés Barrero (Granada, Spain) ..... 969

**Pharmaceutical Applications**

High performance liquid chromatographic and thin layer densitometric methods for the determination of risperidone in the presence of its degradation products in bulk powder and in tablets

Z.A. El-Sherif (Giza, Egypt), B. El-Zeany (Cairo, Egypt), O.M. El-Houssini (Giza, Egypt) ..... 975

Determination of residual solvents and investigation of their effect on ampicillin trihydrate crystal structure

S. Nojavan, A. Ghassempour (Tehran, Iran), Y. Bashour, M.K. Darbandi (Tabriz, Iran), S.H. Ahmadi (Tehran, Iran) ..... 983

Comparative study between capillary electrophoresis and high performance liquid chromatography in 'guarana' based phytopharmaceuticals

L.L. Sombra, M.R. Gómez, R. Olsina, L.D. Martínez, M.F. Silva (San Luis, Argentina) ..... 989

Analysis of rhubarb anthraquinones and bianthrone by microemulsion electrokinetic chromatography

S.-W. Sun, P.-C. Yeh (Taipei, Taiwan) ..... 995

Development and validation of HPLC methods for the enantioselective analysis of bambuterol and albuterol

A. Bartolinčić, V. Drušković, A. Šporec, V. Vinković (Zagreb, Croatia) ..... 1003

Stability studies on some benzocycloheptane antihistaminic agents

M.A. Abounassif, H.A. El-Obeid, E.A. Gadkariem (Riyadh, Saudi Arabia) ..... 1011

The rutin/β-cyclodextrin interactions in fully aqueous solution: spectroscopic studies and biological assays

M.L. Calabro, S. Tommasini, P. Donato, R. Stancanelli, D. Raneri, S. Catania, C. Costa, V. Villari, P. Ficarra, R. Ficarra (Messina, Italy) ..... 1019

Chemical fingerprint and metabolic fingerprint analysis of Danshen injection by HPLC-UV and HPLC-MS methods

J.-l. Zhang, M. Cui, Y. He, H.-l. Yu, D.-a. Guo (Beijing, PR China) ..... 1029

**Bioanalytical Applications**

Development, validation, and implementation of a multiplex immunoassay for the simultaneous determination of five cytokines in human serum  
C.A. Ray, R.R. Bowsher (Indianapolis, IN, USA), W.C. Smith, V. Devanarayanan (Greenfield, IN, USA), M.B. Willey, J.T. Brandt, R.A. Dean (Indianapolis, IN, USA) ..... 1037

Simultaneous determination of niacin, niacinamide and nicotinuric acid in human plasma P. Pfuhl, U. Kärcher, N. Häring, A. Baumeister (Neu-Ulm, Germany), M.A. Tawab, M. Schubert-Zsilavecz (Frankfurt am Main, Germany) . . . . .	1045
Potentiometric flow injection analysis of mebeverine hydrochloride in serum and urine H. Ibrahim, Y.M. Issa, H.M. Abu-Shawish (Cairo, Egypt) . . . . .	1053
Development and validation for high selective quantitative determination of metformin in human plasma by cation exchanging with normal-phase LC/MS/MS N. Koseki, H. Kawashita, M. Niina, Y. Nagae, N. Masuda (Ibaraki, Japan) . . . . .	1063
Elimination of autosampler carryover in a bioanalytical HPLC-MS/MS method: a case study P.T. Vallano, S.B. Shugarts, E.J. Woolf, B.K. Matuszewski (West Point, PA, USA) . . . . .	1073
Detection of levodopa, dopamine and its metabolites in rat striatum dialysates following peripheral administration of L-DOPA prodrugs by mean of HPLC-EC G. Cannazza (Modena, Italy), A. Di Stefano (Chieti, Italy), B. Mosciatti (Modena, Italy), D. Braghierioli (Chieti, Italy), M. Baraldi (Modena, Italy), F. Pinnen, P. Sozio (Chieti, Italy), C. Benatti, C. Parenti (Modena, Italy) . . . . .	1079
<b>Short Communications</b>	
N-Deethylation and N-oxidation of etamiphylline: identification of etamiphylline-N-oxide in greyhound urine by high performance liquid chromatography-mass spectrometry M.C. Dumasia, P. Teale (Cambridgeshire, UK) . . . . .	1085
Simultaneous determination of pyrimethamine, sulfadiazine and N-acetyl-sulfadiazine in plasma for monitoring infants in treatment of congenital toxoplasmosis J.K. Johannessen, I. Christiansen, D.R. Schmidt, E. Petersen, S.H. Hansen (Copenhagen, Denmark) . . . . .	1093
Analysis of pyrazine 2,5-dipropionic acid in 5-aminolevulinic acid-loaded urological and topical delivery vehicles: methodology and assay validation P.A. McCarron, R.F. Donnelly, A. David Woolfson, G.P. Andrews (Belfast, UK) . . . . .	1099
Development and application of a novel UV method for the analysis of ascorbic acid W. Zeng, F. Martinuzzi, A. MacGregor (Toronto, Canada) . . . . .	1107
Determination of dipyridamole in pharmaceutical preparations using square wave voltammetry R.A. de Toledo, M. Castilho, L.H. Mazo (São Carlos, Brazil) . . . . .	1113
Flow injection spectrophotometric determination of Al in hemodialysis solutions J.L. Rodrigues, C.S. de Magalhães, P.O. Luccas (Alfenas, Brazil) . . . . .	1119
Calender . . . . .	1125
List of contents . . . . .	1129
Author Index . . . . .	1139
Subject Index . . . . .	1145
Guide for Authors . . . . .	I