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Current awareness in drug testing and analysis

1 Reviews

Francis PS, Adcock JL, Costin JW, Purcell SD, Pfeffer FM, Barnett NW// Deakin University, School Life & Environm Sci, Geelong, Vic 3217, Australia

J Pharm Biomed Anal 2008 **48** (3) 508

Chemiluminescence detection of opium poppy (*Papaver somniferum*) alkaloids

The analysis of the opium poppy (*Papaver somniferum*) alkaloids and their semi-synthetic derivatives has important applications in industrial process monitoring, clinical analysis and forensic science. Liquid-phase chemiluminescence reagents such as tris(2,2'-bipyridyl)ruthenium(II) and acidic potassium permanganate exhibit notable sensitivity and complementary selectivity for many *P. somniferum* alkaloids. This has been exploited to develop a range of analytical procedures using flow analysis, HPLC, CE and microfluidic instrumentation

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J Anal Toxicol 2008 **32** (8) 612

Stability of cyanide in cadavers and in postmortem stored tissue specimens: A review

The toxicological and postmortem analysis of fire victims' blood and tissue can disclose the type and quantity of toxic species inhaled prior to death. Assigning a level of significance to cyanide concentrations found in the blood and tissue of fire victims is often difficult due to the fact that cyanide is inherently unstable in cadavers and in stored tissue samples. The rate of transformation of cyanide in blood and tissue specimens is dependent on the initial cyanide concentration in the sample at time of death, the length of time that a sample remains in the cadaver, the length of time that a sample remains in storage, and the preservation (e.g., addition of sodium fluoride to sample) and storage conditions (e.g., temperature) of the sample

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Bunseki Kagaku 2008 **57** (10) 783

Hair analysis of drugs of abuse (Japanese, English Abstract)

Serious social problems world wide are occurring as a result of drug abuse. The development and application of analytical methods of drugs of abuse are very important for the prediction of and protection from the risk to human health. Analysis of hair is invaluable because it illustrates drug intake over a long period. The pupose of this review is to discuss the history and significance of hair analysis. Recently reported application cases are introduced

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Trends Anal Chem 2008 **27** (11) 1053

Analysis of drugs of abuse and their human metabolites in water by LC-MS^2: A non-intrusive tool for drug abuse estimation at the community level

In this article, protocols, including sample preservation, pretreatment and extraction, detection conditions, and matrix effects for drugs of abuse (DAs) in surface and sewage water, where these compounds are at detectable levels (μg/l-ng/l) are reviewed. Analytical procedures are based on LC separation and MS^2 detection. Concentrations measured in sewage water can be used to estimate overall usage of DAs in different locations

2 Sports Doping - General

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J Chromatogr A 2008 **1210** (2) 193

Implementation of gas chromatography combined with simultaneously selected ion monitoring and full scan mass spectrometry in doping analysis

A method capable of detecting over 150 components mentioned on the list of WADA is described. The analytes are extracted from urine by a combined extraction procedure using freshly distilled diethyl ether and *tert*-butyl methyl ether as extraction solvents at pH 9.5 and 14 respectively. Prior to GC-MS analysis the residues are combined and derivatised using a mixture of *N*-methyl-*N*-trimethylsilyltrifluoroacetamide, NH_4I and ethanethiol. The mass spectrometer is simultaneously operated in the full scan mode (mass range varies along with GC-oven temperature program) and in the selected ion monitoring mode. The obtained limits of detection are in compliance with the requirements set by the WADA. Besides narcotics, stimulants and anabolic androgenic agents, this method is also capable of detecting several agents with anti-estrogenic activity and some β-agonists

In order to keep subscribers up-to-date with the latest developments in their field, John Wiley & Sons are providing a current awareness service in each issue of the journal. The bibliography contains newly published material in the field of drug testing and analysis. Each bibliography is divided into 18 sections: 1 Reviews; 2 Sports Doping - General; 3 Steroids; 4 Peptides; 5 Diuretics; 6 CNS Agents; 7 Equine; 8 Recreational Drugs - General; 9 Stimulants; 10 Hallucinogens; 11 Narcotics; 12 Forensics; 13 Alcohol; 14 Tobacco; 15 Homeland Security; 16 Workplace; 17 Product Authenticity; 18 Techniques. Within each section, articles are listed in alphabetical order with respect to author. If, in the preceding period, no publications are located relevant to any one of these headings, that section will be omitted.

3 Steroids

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Rapid Commun Mass Spectrom 2008 **22** (24) 4147

The detection of androstenedione abuse in sport: A mass spectrometry strategy to identify the 4-hydroxyandrostenedione metabolite

Studies have shown that the administration of androstenedione (ADIONE) significantly increases the urinary ratio of testosterone glucuronide to epitestosterone glucuronide (T/E) - measured by gas chromatography/mass spectrometry (GC/MS) - in subjects with a normal (approximately 1) or naturally high (>1) initial values. However, the urinary T/E ratio has been shown not to increase in subjects with naturally low (<1) initial values. Such cases then rely on the detection of C_6-hydroxylated metabolites shown to be indicative of ADIONE administration. While these markers may be measured in the routine GC/MS steroid profile, their relatively low urinary excretion limits the use of gas chromatography/combustion/isotope ratio mass spectrometry (GC/C/IRMS) to specifically confirm ADIONE administration based on depleted ^{13}C content. A mass spectrometry strategy was used in this study to identify metabolites of ADIONE with the potential to provide compound-specific detection. C_4-hydroxylation was subsequently shown to be a major metabolic pathway following ADIONE administration, thereby resulting in urinary excretion of 4-hydroxyandrostenedione (4OH-ADIONE). Complementary analysis of 4OH-ADIONE by GC/MS and GC/C/IRMS was used to confirm ADIONE administration

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J Chromatogr A 2008 **1214** (1-2) 134

Application of comprehensive two-dimensional gas chromatography to sterols analysis

GCxGC for sterol analysis was investigated by separation and identification of endogenous sterols in standards, and spiked in human urine. The separation pattern of trimethylsilyl (TMS) derivatives of sterols was compared on two complementary column sets. Whilst the BPX5/BPX50 column set provides better overall separation, BPX50/BPX5 results in better peak shape and sensitivity. The match quality of GCxGC-TOFMS spectra was superior to that for analysis using 1D GC-TOFMS for sterols spiked in urine. TOFMS coupled to GCxGC enabled satisfactory identification of sterols in urine at their lowest limit of detection. This study shows that GCxGC-TOFMS yields high specificity for steroids derived from urine, with detection limits appropriate for use in doping control

Pozo OJ, Van Eenoo P, Deventer K, Grimalt S, Sancho JV, Hernandez F, Delbeke FT// UGent, Dept Clin Chem Microbiol & Immunol, DoCoLab, Technologiepark 30, BE-9052 Zwijnaarde, Belgium

Rapid Commun Mass Spectrom 2008 **22** (24) 4009

Collision-induced dissociation of 3-keto anabolic steroids and related compounds after electrospray ionization. Considerations for structural elucidation

The collision-induced dissociation of forty-one 3-keto anabolic steroids and related compounds has been studied using both triple quadrupole (QqQ) and hybrid quadrupole-time of flight (QTOF) instruments. Due to the complexity of the product ion spectra of these analytes, which generate a large number of ions, only two specific regions were studied in depth: the product ions near the precursor ion (m/z > or =M-100) and the most abundant product ions at a collision energy of 30 eV. Accurate mass measurements were used in order to obtain an unequivocal assignment of the empirical formula and the origin of each selected product ion. Analytes have been divided into eight groups according to the number and position of double bonds and the presence of functional groups such as hydroxyl- or nitrogen-containing rings. A correlation between the steroid structure and the product ions obtained has been postulated. The application of these correlations can be useful in the elucidation of feasible structures for unknown steroids and/or their metabolites

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Trends Anal Chem 2008 **27** (8) 657

Detection and characterization of anabolic steroids in doping analysis by LC-MS

GC-MS has usually been emplyed for analysis of anabolic steroids in doping investigations. However, LC-MS^2 is becoming increasingly important for this purpose. The production of non-commercially available anabolic steroids detected in clinical specimens has become a challenge for doping-control laboratories. The potential of different LC-MS and LC-MS^2 scan modes for detection of both target and unknown anabolic steroids is discussed. Several modes (e.g., selected reaction monitoring, full scan and product-ion scan) can be successfully used for the detection of target analytes. The most powerful approach seems to be to combine several scan modes in order to detect and to characterize unknown steroids and metabolites

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J Chromatogr B 2008 **876** (2) 225

Validation of an extended method for the detection of the misuse of endogenous steroids in sports, including new hydroxylated metabolites

Amongst the most misused doping agents in sports are endogenous steroids. Consequently, their presence poses a major challenge for doping control laboratories. Threshold levels do not currently allow for the detection of all endogenous steroid misuse due to great interindividual variations in urinary steroid concentrations. A technique has been developed and validated to screen for traditionally monitored endogenous steroids in doping control as well as specific hydroxylated/oxygenated metabolites in order to enhance the detection capabilities for the misuse of endogenous steroids

4 Peptides

Abellan R, Ventura R, Palmi I, Di Carlo S, Bacosi A, Bellver M, Olive R, Pacual JA, Pacifici R, Segura J, Zuccaro P, Pichini S*// *Ist Superiore Sanita, Dept Therapeut Res Med & Evaluation, Viale Regina Elena 299, IT-00161 Rome, Italy

J Pharm Biomed Anal 2008 **48** (3) 844

Immunoassays for the measurement of IGF-II, IGFBP-2 and-3, and ICTP as indirect biomarkers of recombinant human growth hormone misuse in sport. Values in selected population of athletes

IGF-II, IGFBPs -2 and -3 and ICTP have been proposed as indirect biomarkers of the recombinant human growth hormone misuse in sport. An extended intra- and inter-laboratory validation of commercially available immunoassays for these biomarkers detection was performed. ELISA assays for total IGF-II, IGFBP-2 and IGFBP-3 (IGF-II/ELISA1: DSLabs, IGFBP-2/ELISA2: Biosource, and IGFBP-3/ELISA3: BioSource) and an EIA assay for ICTP (ICTP/EIA: Orion Diagnostica) were evaluated. The inter- and intra-laboratory precision values were acceptable for all evaluated assays

5 Diuretics

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J Pharm Biomed Anal 2008 **48** (2) 462

LC-high-resolution multiple stage spectrometric analysis of diuretic compounds - Unusual mass fragmentation pathways

In this work, some unusual fragmentation steps resulting from high-resolution MS^n are investigated. Amiloride, for example, produces an intense product ion in MS^3 analysis with an apparent loss of 10Da. Water adduct formation and successive carbon monoxide elimination might explain this unusual behavior. By contrast, bendroflumethiazide MS^n spectra show three successive HF losses, in spite of the presence of a radical site in the parent structure. In addition, homolytic cleavages with radical ion production occur in the case of protonated positive ion of ethacrynic acid (loss of chlorine radical) indicating that such fragmentation behavior is not so rare as generally reported. Different ionization modes were studied and a tentative correlation with acidic-base properties was done

Silvestre CIC, Santos JLM*, Lima JLFC, Zagatto EAG// *Univ Porto, Fac Farm, Serv Quim-Fis, Rua Anibal Cunha 164, PT-4099-030 Oporto, Portugal

Talanta 2008 **77** (2) 518

Single reaction interface flow system for chemiluminescent monitoring of mannitol based on its hydroxyl radical scavenger activity

Single reaction interface flow analysis (SIFA) system was employed to monitor mannitol in pharmaceutical formulations and human urine. The technique

makes use of the inhibitory effect of the mannitol scavenger to inhibit the chemiluminescent reaction between luminol and myoglobin in the absence of H_2O_2. This technique facilitated the determination of mannitol concentrations between 25 mmol/l and 1 mol/l. It was applied to the determination of mannitol in pharmaceuticals and in human urine samples without any pretreatment process. Recovery values obtained in the analysis of spiked urine samples were between 94.9 and 105.3%

6 CNS Agents

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J Anal Toxicol 2008 **32** (9) 763

Urinary elimination of ephedrines following administration of the traditional Chinese medicine preparation Kakkon-to

Ephedrae Herba is one of the prescriptions of Kakkon-to. The major ingredients of Ephedrae Herba, ephedrines, are banned substances on the WADA list. The concentrations of urinary ephedrines were analyzed by HPLC. The result showed that ephedrine and norpseudoephedrine were excreted in the urine after taking one single dose of Kakkon-to. The study indicated that levels in urine after administering a single dose of Kakkon-to would not violate the rule of doping. However, a further study on administering the preparation for 3 times per day for 3 days showed a positive ephedrine result. Athletes should be careful when taking more than a single dose of Kakkon-to

7 Equine

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Anal Chem 2008 **80** (21) 8340

Identification of recombinant equine growth hormone in horse plasma by LC-MS/MS: A confirmatory analysis in doping control

Equine growth hormone (eGH) is suspected of being illegally administered to racehorses in order to improve physical performance and to speed-up wound healing. reGH differs from the native eGH by an additional methionine at the N-terminal (met-eGH) and has never been unambiguously detected in any type of biological matrix at trace concentrations (1-10 μg/l). A plasma sample (4 ml) was treated with ammonium sulfate at the reGH isoelectric point and the pellet was purified by solid-phase extraction. Specific peptides were generated by trypsin digestion and analyzed by LC-MS/MS. The detection limit was 1 μg/l. The method was validated according to European Union regulation (DEC/2002/657/EC) and the Association of Official Racing Chemists (AORC) requirements. In addition, it was successfully applied to determining the plasma concentrations of reGH with time using linear ion trap mass analyzer. The presence of this prohibited hormone (reGH) was also successfully detected by triple quadrupole mass spectrometry up to 48 h postadministration of reGH to a horse

Benoit M, Lingen K, Taddei LM, Heffron BT, Hurt L, Lokanc JA, Lingner K, Cardenas E, Flores S, Mayer D, Pilipiak D, Folker-Calderon D, Negrusz A*// *Univ Illinois, Animal Forensic Toxicol Lab, 2242 West Harrison St, Chicago, Il 60612, USA

J Anal Toxicol 2008 **32** (8) 667

Pyrilamine and o-desmethylpyrilamine detection in equine serum and urine

Pyrilamine (mepyramine) is an H_1-receptor antagonist used in human and veterinary medicine. It has the potential to produce central nervous system effects in horses. Urine and serum samples were initially screened by the pyrilamine ELISA kit with subsequent confirmation and quantitation utilizing a newly developed and validated GC-MS method for pyrilamine and its major metabolite *O*-desmethylpyrilamine using chlorpromazine as an internal standard. Prior to the basic extraction, urine specimens were hydrolyzed using β-glucuronidase. The urine extracts as well as the serum samples were then subjected to solid-phase extraction on Bond Elut LRC-PRS columns. Whilst pyrilamine was eliminated from the bloodstream rather quickly, the metabolite level remained in the urine for days after administration

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Biomed Chromatogr 2008 **22** (8) 912

Analysis of iridoids from *Harpagophytum* and eleutherosides from *Eleutherococcus senticosus* in horse urine

LC/ESI-MS^n methods have been previously set up to detect the administration of (i) *Harpagophytum* and (ii) preparations containing a plant capable of anti-stress properties: *Eleutherococcus senticosus*. Regarding the detection of *Harpagophytum* administration, harpagoside, harpagide and 8-*para*-coumaroyl harpagide were detected together in only one sample out of 317. Eleutheroside E was found to be the main indicator of *Eleutherococcus senticosus* administration. It was detected in post-administration samples collected from two horses having received a feed supplement containing *Eleutherococcus senticosus* for several days. Out of the 382 samples tested, eleutheroside E was found in an unexpectedly large number of urine samples (39%) of various origins and its presence cannot be only due to the sole use of herbal dietary supplements

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J Forensic Sci 2008 **53** (5) 1145

Substitution of human for horse urine disproves an accusation of doping

In order to detect switching and/or manipulation of samples, a DNA test was performed on a positive doping urine sample. The objective was to compare the urine DNA profile *versus* blood and hair DNA profiles from the same stallion. At first, 10 microsatellite markers were investigated to determine the horse identity. No results were obtained when horse specific markers were typed in the urine sample. In order to confirm the species origin of this sample we analyzed the mitochondrial cytochrome *b* gene. This analysis from blood and hair samples produced reproducible and clear PCR-RFLP patterns and DNA sequence match with those expected for horse, while the urine sample results were coincident with human. These results allowed us to exclude the urine sample from the questioned stallion and determine its human species origin, confirming the manipulation of urine sample

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Biomed Chromatogr 2008 **22** (6) 662

Boldenone, testosterone and 1,4-androstadiene-3,17-dione determination in faeces from horses, untreated and after administration of androsta-1,4-diene-3,17-dione (boldione)

Semi-quantitative analyses of 1,4-androstadiene-3,17-dione, testosterone, 17α- and 17β-boldenone were conducted in pre- and post-administration faeces, and in controls (untreated stallions, geldings and mares). Sample preparation comprised diethyl ether extraction, lipid removal, HPLC purification and derivatisation. 1,4-Androstadiene-3,17-dione, testosterone, 17α- and 17β-boldenone were analysed by GC-EI/MS/MS. Faeces from females in oestrus had detectable levels of boldenone isomers and testosterone

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J Vet Pharmacol Ther 2008 **31** (6) 587

Plasma concentrations of testosterone and 19-nortestosterone (nandrolone) in the nonracing intact male horse by liquid chromatography-mass spectrometry

No abstract available

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J Anal Toxicol 2008 **32** (9) 754

Comparison of liquid chromatography-mass spectrometry and radioimmunoassay for measurement of fentanyl and determination of pharmacokinetics in equine plasma

This study evaluated the validity of measuring fentanyl concentrations in equine plasma using RIA by comparing it to the established technique of LC-MS. Equine plasma samples were analyzed using a solid-phase Coat-A-Count fentanyl RIA and a validated LC-MS method. Fentanyl concentrations determined by RIA and LC-MS correlated. However, the RIA overestimated low fentanyl concentrations and underestimated high fentanyl concentrations

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J Anal Toxicol 2008 **32** (5) 387

Detection of urinary metabolites common to structurally related 17 -alkyl anabolic steroids in the horse and application to doping tests in racehorses: Methandienone, methandriol, and oxymetholone

Methandienone, methandriol, and oxymetholone are anabolic steroids possessing 17α-methyl and 17β-hydroxy groups which have been used in racehorses to enhance racing performance. Metabolites common to those of 17α-methyltestosterone and mestanolone were detected in horse urine after the administration of oxymetholone, methandienone, and methandriol. Based on analytical data, we confirmed these to be the common metabolites of five structurally related steroids, 17α-methyltestosterone, mestanolone, oxymetholone, methandienone, and methandriol. Furthermore, we detected hitherto unknown urinary metabolites of methandriol and oxymetholone in horses. On the other hand, the major metabolite of oxymetholone was mestanolone

8 Recreational Drugs - General

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J Anal Toxicol 2008 **32** (7) 511

Stability of benzodiazepines and cocaine in blood spots stored on filter paper

Drugs investigated included flunitrazepam, temazepam, oxazepam, lorazepam, nitrazepam, diazepam, and cocaine. A Guthrie card 903 was spotted with 100 µl of blood containing the drugs at concentrations of 1000 ng/ml and left overnight to dry at room temperature. The filter paper was suspended in extraction buffer for 1 h with ultrasonication. Drugs were then extracted from the buffer by solid-phase extraction using Clean Screen® columns and analyzed by LC-MS-MS. Degradation of the drugs in DBS at all storage conditions was less than for the corresponding liquid blood samples stored under similar conditions and more than 80% of each analyte could be recovered from the samples

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Environ Sci Technol 2008 **42** (23) 8841

Eliminating solid phase extraction with large-volume injection LC/MS/MS: Analysis of illicit and legal drugs and human urine indicators in US wastewaters

Large-volume followed by LC/MS/MS was developed and optimized to eliminate the need for off- and on-line solid phase extraction as a sample preparation step. Centrifugation of raw municipal influent followed by LVI was optimized for the routine determination of illicit drugs and related substances in municipal wastewaters

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J Anal Toxicol 2008 **32** (8) 530

Urine drug testing of chronic pain patients: Licit and illicit drug patterns

Confirmation data for 10,922 positive specimens were collated into 11 drug classes. The number of drug/metabolites tested (#) and number of confirmed positive specimens were as follows: amphetamines (7), 160; barbiturates (5), 308; benzodiazepines (6), 2397; cannabinoids (1), 967; carisoprodol (2), 611; cocaine (1), 310; fentanyl (1), 458; meperidine (2), 58; methadone (2), 1209; opiates (7), 8996; and propoxyphene (2), 385. Drug/metabolites were measured by gas chromatography-mass spectrometry. The frequency of illicit drug use (cannabis, cocaine, ecstasy) was 10.8%

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Subst Use Misuse 2008 **43** (10) 1326

Causes and manners of death among users of heroin, methadone, amphetamine, and cannabis in relation to postmortem chemical tests for illegal drugs

A 12-year medicolegal investigation of deceased illegal drug users (ILDU) in Stockholm, Sweden, classified on the basis of postmortem chemical tests, showed noticeable variations in causes and manners of death as well as in the distribution of suicide methods. This study offers objective information about connection between the postmortem findings of illegal drugs and the causes and manners of death of their users. However, further studies, comparing prevalence of drug use in general population and at the postmortem tests, are needed for more detailed elucidation of this connection

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J Appl Toxicol 2008 **28** (8) 998

Comparison of two extraction procedures for determination of drugs of abuse in human saliva by high-performance liquid chromatography

HPLC-DAD was used to determine morphine, 6-acetylmorphine, cocaine, benzoylecgonine, cocaethylene, methadone and 2-ethylene-1,5-dimethyl-3,3,-diphenylpyrrolidine in human saliva. The proposed method was applied to 24 saliva samples from individuals poisoned with opiates and/or cocaine

Gottardo R, Polettini A, Sorio D, Pascali JP, Bortolotti F, Liotta E, Tagliaro F*// *Univ Verona, Dept Med & Publ Hlth, Sect Forensic Med, Unit Forensic Med, IT-37134 Verona, Italy

Electrophoresis 2008 **29** (19) 4078

Capillary zone electrophoresis (CZE) coupled to time-of-flight mass spectrometry (TOF-MS) applied to the analysis of illicit and controlled drugs in blood

A new method for the determination of illicit and abused drugs in blood by CZE-EI-TOF-MS is proposed. Methamphetamine, methylenedioxyamphetamine, methylenedioxyethylamphetamine, methylenedioxymethamphetamine, methadone, cocaine, morphine, codeine, 6-acetylmorphine and benzoylecgonine were separated with CZE in an uncoated fused-silica capillary using an ammonium formate electrolyte solution. The capillary electropherograph was coupled to TOF-MS through an orthogonal electrospray ionization source, with a coaxial sheath liquid interface. Forensic drugs were identified by exact mass determination and by matching of the isotopic pattern

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J Anal Toxicol 2008 **32** (6) 408

Concentration distribution of the marijuana metabolite ^{9}-tetrahydrocannabinol-9-carboxylic acid and the cocaine metabolite benzoylecgonine in the Department of Defense urine drug-testing program

Urine drug testing has been employed for punitive purposes by the Department of Defense since December 1981. Little can be opined with certainty from a positive urine drug test as to the amount of drug ingested, when the drug was ingested, and in most instances, whether the individual felt the effects of the drug, or was under the influence of the drug found in the urine. The finding that 50% of all positive marijuana and cocaine urine metabolite concentrations in the military testing program over the three-year period of October 1, 2004 through September 30, 2007, are below a median value of 65 and 968 ng/ml, respectively, provide reference points

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J Anal Toxicol 2008 **32** (8) 605

Validation of analysis of amphetamines, opiates, phencyclidine, cocaine, and benzoylecgonine in oral fluids by liquid chromatography-tandem mass spectrometry

Amphetamines (AMPs), opiates, phencyclidine (PCP), and cocaine and its metabolite benzoylecgonine (BE) in oral fluids were quantitated by an Applied Biosystems 3200 QTRAP LC-MS-MS. AMPs, opiates, PCP, cocaine, and BE were extracted from samples using liquid-liquid or solid-phase extractions and the extracts were separated on a Shimadzu HPLC prior to the MS-MS analysis. The limit of detection/quantitation for AMPs, opiates, PCP, cocaine, and its metabolite BE were 10, 10, 2, 2, and 2 ng/ml of oral fluid, respectively

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J Mass Spectrom Soc Jpn 2008 **56** (3) 123

Development of a systematic screening procedure for abused drugs without using standard compounds by gas chromatography/mass spectrometry (Japanese, English Abstract)

A procedure is proposed for drugs of abuse that does not require the use of standard compounds. It employs GC/MS with retention time locking. Target compounds were fifty-five drugs of abuse, including amphetamine, piperazine, tryptamine and phenetylamine derivatives, opiates, and benzodiazepines. The method consists of solid-phase extraction with a polar-enhanced Focus™ column followed by acetylation and GC/MS with retention time locking. An "abused drugs database," which includes the retention time, qualifier ion/target ion percentage, and calibration curve (values of slope and intercept), was produced using the novel GC/MS software, NAGINATA™

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J Anal Toxicol 2008 **32** (6) 402
Urine monitoring of diazepam abuse - New intake or not?
Testing for drugs-of-abuse in urine is requested for multiple reasons, including legal and workplace policies. In these cases under suspicion, diazepam metabolites were measured in urine samples by gas or liquid chromatography coupled to mass spectrometry. Very long elimination times were found in the described cases. Neither had in fact ingested diazepam during the study period. By the use of pharmacogenetic typing, one of the subjects was found to have a slow metabolism for CYP2C9 as well as for CYP2C19. In the second case, there was a possible drug interaction between diazepam and zolpidem

Marchei E, Colone P, Nastasi GG, Calabro C, Pellegrini M, Pacifici R, Zuccaro P, Pichini S*// *Ist Superiore Sanita, Dept Therapeut Res & Med Evaluat, Viale Regina Elena 299, IT-00161 Rome, Italy
J Pharm Biomed Anal 2008 **48** (2) 383
On-site screening and GC-MS analysis of cocaine and heroin metabolites in body-packers urine
Illicit transportation of cocaine and heroin either swallowed or inserted into the rectum and/or vagina of individuals, defined as "body-packers", is becoming increasingly common. The presence of cocaine and heroin metabolites in urine from suspected body-packers was investigated by an on-site immunochromatographic test and confirmed the obtained results by gas chromatography-mass spectrometry and X-ray examination. Urine was immediately screened on-site by Cozart rapid urine test. Irrespective of test results, individuals underwent X-ray examination and urine samples were analyzed by gas chromatography-mass spectrometry (GC-MS). Some positive results were obtained from users who were not body-packers

Miller EI, Wylie FA, Oliver JS// Univ Glasgow, Forensic Med & Sci, University Pl, Glasgow G12 8QQ, Scotland
J Anal Toxicol 2008 **32** (7) 457
Simultaneous detection and quantification of amphetamines, diazepam and its metabolites, cocaine and its metabolites, and opiates in hair by LC-ESI-MS-MS using a single extraction method
LC-MS-MS methodology was developed and validated for the simultaneous identification and quantification of amphetamines, diazepam and its metabolites, cocaine and its metabolites, and opiates from hair using a single extraction method. As part of the method development, Gemini C18, Synergi Hydro RP, and Zorbax Stablebond-Phenyl LC columns were tested with three different mobile phases. Analyte recovery and limit of detection were evaluated for two different solid-phase extraction methods that used Bond Elut Certify and Clean Screen cartridges. Phosphate buffer (pH 5.0) was chosen as the optimum hair incubation medium because of the high stability of cocaine and 6-monoacetylmorphine using this method and faster sample preparation

Montgomery DP, Plate CA, Jones M, Jones J, Rios R, Lambert DK, Schumtz N, Wiedmeier SE, Burnett J, Ail S, Brandel D, Maichuck G, Durham CA, Henry E, Christensen RD*// *Intermountain Healthcare, Dept Women & Newborns, 4403 Harrison Blvd, Ogden, Ut 84403, USA
J Perinatol 2008 **28** (11) 750
Using umbilical cord tissue to detect fetal exposure to illicit drugs: A multicentered study in Utah and New Jersey
A total of 498 umbilical cord samples were analyzed of which 157 (32%) were positive using mass spectrometric detection. The sensitivity and specificity of the ELISA-based test for each class of drugs tested were as follows: methamphetamine 97 and 97%, opiates 90 and 98%, cocaine 90 and 100%, cannabinoids 96 and 98% and phencyclidine (only 1 of the 498 umbilical cord sample was positive for phencyclidine) 100 and 100%

Pelander A, Ristimaa J, Rasanen I, Vuori E, Ojanpera I// Univ Helsinki, Dept Forensic Med, POB 40, FI-00014 Helsinki, Finland
Ther Drug Monit 2008 **30** (6) 717
Screening for basic drugs in hair of drug addicts by liquid chromatography/time-of-flight mass spectrometry
In this study, a qualitative drug screening method was adapted for screening of basic drugs in hair. The method included alkaline hydrolysis, purification with mixed-mode solid phase extraction, and analysis by LC coupled to ToF MS with automated data analysis and reporting. Identification was based on accurate mass, isotopic pattern fit, and retention time. Drug classes identified included antidepressants, antipsychotics, antiepileptics, amphetamines, opioids, beta-blockers, a benzodiazepine, a hypnotic, a local anesthetic, an antiemetic, and an antipyretic analgesic

Sato C, Furube A, Katoh R, Nonaka H, Inoue H// Natl Inst Advanced Ind Sci & Technol, Res Inst Instrumentation Frontier, Tsukuba, Ibaraki 305 8565, Japan
Jpn J Appl Phys 2008 **47** (11) 8583
Non-destructive and discriminating identification of illegal drugs by transient absorption spectroscopy in the visible and near-IR wavelength range
Nanosecond transient absorption spectroscopy with a 10-ns UV-laser pulse for the excitation light and visible-to-near-IR light for the probe light was tested for the possibility of identifying illegal drugs. The transient absorption spectra of acetonitrile solutions of *d*-methamphetamine, *dl*-3,4-methylenedioxymethamphetamine hydrochloride (MDMA), and *dl*-*N*-methyl-1-(1,3-benzodioxol-5-yl)-2-butanamine hydrochloride (MBDB) were measured. Analysis of the spectra in terms of exponential and Gaussian functions, identified drugs and discriminated them from chemical substances having similar structures. Transient absorption spectroscopy should be a non-destructive method of inspecting for illegal drugs, especially when they are dissolved in liquids. This technique may even be used for drugs packed in opaque materials if it is further extended to utilize intense femtosecond laser pulses

Uchiyama N, Kikura-Hanajiri R, Kawahara N, Goda Y// Natl Inst Hlth Sci, 1-18-1 Kamiyoga, Setagaya ku, Tokyo 158 8501, Japan
Yakugaku Zasshi 2008 **128** (10) 1499
Analysis of designer drugs detected in the products purchased in fiscal year 2006 (Japanese, English Abstract)
To inhibit drug abuse, 32 substances have been controlled in Japan since April in 2007 by the Pharmaceutical Affairs Law as designated substances (Shitei-Yakubutsu, classified as 11 tryptamines, 11 phenethylamines, 2 piperazines, 6 alkyl nitrites, 1 diterpene and 1 plant). Whereas the distributions of have decreased as a result this regulation, new designer drugs are still being found. In this study using NMR, GC-MS and LC-MS, 7 designer drugs in 15 products were detected. Three methylone derivertives (1-(3,4-methylenedioxyphenyl-2-(pyrrolidin-1-yl)-1-pentanone: MDPV, 2-methylamino-1-(3,4-methylenedioxyphenyl)butan-1-one: bk-MBDB, 2-ethylamino-1-(3,4-methylenedioxyphenyl)propan-1-one): bk-MDEA, a MDMA derivative (*N*-hydroxy-1-(3,4-methylenedioxyphenyl)-2-aminopropane: N-OH MDMA), a methamphetamine derivative (*N*-methyl-1-(4-fluorophenyl)propan-2-amine: *N*-Me-4-FMP), a tryptamine derivative (5-methoxy-*N*-ethyl-*N*-isopropyltryptamine: 5-MeO-EIPT) and indan-2-amine were detected. 5-MeO-EIPT was newly identified in this study

9 Stimulants

Aasim WRW, Gan SH, Tan SC// Univ Sains Malaysia, Sch Med Sci, Dept Pharmacol, MY-16150 Kubang Kerian, Kelantan, Malaysia
Biomed Chromatogr 2008 **22** (9) 1035
Development of a simultaneous liquid-liquid extraction and chiral derivatization method for stereospecific GC-MS analysis of amphetamine-type stimulants in human urine using fractional factorial design
A stereospecific GC-MS analysis method for amphetamine-type stimulants in human urine was recently developed. For maximum efficiency, liquid-liquid extraction and chiral derivatization of the analytes using (*R*)-(-)-α-methoxy-α-(trifluoromethyl)phenylacetyl chloride were performed simultaneously. The effects of (1) use of saturated sodium chloride in 2.0 M sodium hydroxide, (2) extraction solvent volume, (3) percentage of triethylamine, (4) derivatization reagent volume, (5) sample mixing time, (6) incubation temperature and (7) incubation time on method sensitivity and variability were assessed using a two-level, eight-run Plackett-Burman design followed by a fold- over design. The use of saturated sodium chloride solution and the derivatization reagent volume were significant factors (ANOVA, $p<0.01$). The saturated sodium chloride solution decreased sensitivity whereas an increased volume of derivatization reagent increased sensitivity. Detection limits were <or=2.3 µg/l and quantitation limits <or=7.7 µg/l

Bagheri H, Khallillian F, Ahangar LE// Sharif Univ Technol, Dept Chem, POB 11365-9516, Tehran, Iran
J Sep Sci 2008 **31** (18) 3212
Liquid-liquid-liquid microextraction followed by HPLC with UV detection for quantitation of ephedrine in urine
Liquid-liquid-liquid microextraction (LLLME) in combination with HPLC and UV detection has been used as a sensitive method for the determination of ephedrine in urine samples. Extraction process was performed into a microfilm

of toluene/benzene (50:50). The analyte was subsequently back extracted into an acidic microdrop solution (pH 2) suspended in the organic phase. The extract was then injected into the HPLC system directly. An enrichment factor of 137 along with a good sample clean-up was obtained under the optimized conditions. The calibration curve showed linearity in the range of 0.01-50 mg/l

Bartlome R, Rey JM, Sigrist MW// ETH, Inst Quantum Elect, Schafmattstr 16, CH-8093 Zurich, Switzerland
Ann Chem 2008 **80** (14) 5334
Vapor phase infrared laser spectroscopy: From gas sensing to forensic urinalysis
Numerous gas-sensing devices are based on infrared laser spectroscopy. In this paper, the technique is further developed and, for the first time, applied to forensic urinalysis. For this purpose, a difference frequency generation laser was coupled to an in-house-built, high-temperature multipass cell (HTMC). Quantitative measurements were taken on pure ephedrine and pseudoephedrine vapors. Ephedrine-positive and pseudoephedrine-positive urine samples were prepared by means of liquid-liquid extraction and directly evaporated in the HTMC without any preliminary chromatographic separation. The laser spectrometer has room for much improvement; its potential is discussed with respect to doping agents detection

Chung LW, Liu GJ, Li ZG, Chang YZ, Lee MR*// *Natl Chung Hsing Univ, Dept Chem, 250 Kuo Kuang Rd, Taichung 402, Taiwan
J Chromatogr B 2008 **874** (1-2) 115
Solvent-enhanced microwave-assisted derivatization following solid-phase extraction combined with gas chromatography-mass spectrometry for the determination of amphetamines in urine
An approach using microwave-assisted derivatization (MAD) following solid-phase extraction (SPE) combined with GC-MS was developed to determine amphetamines in urine samples. Derivatization performance was studied using various solvents and compared with the performance obtained without solvent. The highest derivatization efficiencies were obtained in ethyl acetate (EA) under microwave power of 250W for 1min

Da Silva MJ, Dos Anjos EV, Honorato RS, Pimentel MF, Paim APS*// *Univ Fed Pernambuco, Dept Quim Fundamental, Cidade Univ, BR-50740-550 Recife, PE, Brazil
Anal Chim Acta 2008 **629** (1-2) 98
Spectrophotometric cocaine determination in a biphasic medium employing flow-batch sequential injection analysis
A flow-system procedure for the spectrophotometric determination of cocaine using cobalt thiocyanate as a complexing reagent is described. In this reaction, two phases are formed: the superior (pink) contains an excess of cobalt thiocyanate solution and the lower layer (blue) contains the complex cocaine-cobalt thiocyanate. Samples and reagent are inserted through a sequential-injection valve between two air bubbles inside a reaction chamber. An optic fiber sensor connected to the chamber recorded the absorbance at 630 nm signal. The detection and quantification limits were 29.4 mg/l and 98 mg/l

Fernandez P, Aldonza M, Bermejo AM, Tabernero MJ// Fac Med, Inst Legal Med, Forensic Toxicology Serv, Santiago de Compostela, Spain
J Liq Chromatogr Relat Technol 2008 **31** (16) 2467
Bile analysis for cocaine and benzoylecgonine in overdose cases
An HPLC technique with IV detection for determining cocaine and benzoylecgonine (BEG) in human bile is described. A LiChrospher RP-18 column and methanol-phosphate buffer as mobile phase are employed. The average extraction yield was 82% for cocaine and 76% for BEG

Gunn JA, Sweeney B, Dahn T, Bell S, Newhouse R, Terell AR// West Virginia Univ, Bennett Dept Chem, Morgantown, WV 26506, USA
J Anal Toxicol 2008 **32** (7) 485
Simultaneous quantification of amphetamine and methamphetamine in meconium using ISOLUTE HM-N-supported liquid extraction columns and GC-MS
A procedure is described for the rapid extraction and quantification of amphetamine and methamphetamine from meconium using ISOLUTE® HM-N-supported liquid extraction columns and GC-MS. Extraction of both analytes was achieved using ISOLUTE® HM-N-supported liquid extraction columns containing a modified form of diatomaceous earth

Hargreaves MD, Page K, Munshi T, Tomsett R, Lynch G, Edwards HGM// Univ Bradford, Sch Life Sci, Div Chem & Forensic Sci, Raman Spectrosc Grp, Bradford BD7 1DP, England
J Raman Spectrosc 2008 **39** (7) 873
Analysis of seized drugs using portable Raman spectroscopy in an airport environment - A proof of principle study
This study demonstrates the viability of Raman spectroscopy for the rapid identification of illicit substances in their containers in an airport environment. It is demonstrated that the spectrometers are able to collect the spectra of suspect powders, including cocaine HCl and *d*-amphetamine sulphate with unknown constituents rapidly and with a high degree of discrimination

Jagerado E, Montgornery MA, Sibum M, Sasaki TA, Le Beau MA// FBI Lab, 2501 Investigation Pkwy, Quantico, Va 22135, USA
J Anal Toxicol 2008 **32** (8) 570
Rapid analysis of cocaine and metabolites in urine using a completely automated solid-phase extraction-high-performance liquid chromatography-tandem mass spectrometry method
Online solid-phase extraction (SPE) with liquid chromatographic separation and tandem mass spectrometric detection (MS-MS) for the analysis of cocaine and its metabolites in urine has been developed. An efficient online SPE procedure was developed using Hysphere™ MM anion sorbent. A gradient chromatography method with a Gemini C6-Phenyl (50 x 3.00-mm i.d., 5 µm) column was used for the separation of all compounds. Detection was by positive ion mode electrospray ionization MS-MS. Multiple reaction monitoring (MRM) was used to enhance the selectivity and sensitivity of the method. Two MRM transitions were monitored for each analyte and one transition for each internal standard. The limits of detection for the method ranged from 3 to 23 ng/ml and the limits of quantitation ranged from 7 to 69 ng/ml

Jagerdeo E, Montgomery MA, LeBeau MA, Sibum M// FBI Lab, 2501 Investigation Pkwy, Quantico, Va 22135, USA
J Chromatogr B 2008 **874** (1-2) 15
An automated SPE/LC/MS/MS method for the analysis of cocaine and metabolites in whole blood
Analysis of cocaine and its metabolites (benzoylecgonine, ecgonine methyl ester, ecgonine and cocaethylene) from whole blood was performed utilizing online solid-phase extraction (SPE) with HPLC separation and tandem MS detection. Pretreatment of samples involved only protein precipitation and ultracentrifugation. An efficient online solid-phase extraction (SPE) procedure was developed using Hysphere MM anion sorbent. A gradient chromatography method with a Gemini C6-Phenyl (50mmx3.00mm i.d., 5µm) column was used for the complete separation of all components. Analysis was by positive ion mode EI-MS, using multiple reaction monitoring (MRM) to enhance the selectivity and sensitivity of the method

Mercolini L, Mandrioli R, Saladini B, Conti M, Baccini C, Raggi MA*// *Univ Bologna, Fac Pharm, Dept Pharmaceut Sci, Lab Pharmacotoxicol Anal, Via Belmeloro 6, IT-40126 Bologna, Italy
J Pharm Biomed Anal 2008 **48** (2) 456
Quantitative analysis of cocaine in human hair by HPLC with fluorescence detection
Cocaine concentrations in hair are a reliable marker of exposure to the drug and an original LC method has been developed for its determination. The chromatographic analysis was carried out on a Hydro-RP C18 column, using a mobile phase containing a phosphate buffer-acetonitrile-methanol (75:15:10, v/v/v). Native cocaine fluorescence was monitored with mirtazapine as the internal standard. Sample pre-treatment was carried out by incubative extraction with 0.1M HCl followed by solid-phase extraction with C2 cartridges. Good linearity was obtained over a working range of 0.3-100.0 ng/mg

Sun JY, Xu XY, Wang CY, You TY*// *Chinese Acad Sci, Grad Sch, Changchun Inst Appl Chem, State Key Lab Electroanal Chem, CN-130022 Changchun, Peoples Rep China
Electrophoresis 2008 **29** (19) 3999
Analysis of amphetamines in urine with liquid-liquid extraction by capillary electrophoresis with simultaneous electrochemical and electrochemiluminescence detection
Amphetamines including methamphetamine, 3,4-methylenedioxyamphetamine and 3,4-methylenedioxymethamphetamine were separated and detected by CE using simultaneous electrochemical (EC) and electrochemiluminescence (ECL) detection (CE-EC/ECL). Parameters which influence the separation and detection performance, such as the detection potential, the pH value and concentration of the running buffer, the separation voltage and the pH of the detection buffer, were investigated. For practical application, a liquid-liquid extraction with ethyl acetate procedure was developed for urine sample pretreatment and extraction efficiencies higher than 90% were obtained

10 Hallucinogens

Bonadio F, Margot P, Delemont O, Esseiva P*// *Univ Lausanne, Inst Police Sci, Ecole Sci Criminelles, CH-1015 Lausanne, Switzerland

Forensic Sci Int 2008 **182** (1-3) 52

Headspace solid-phase microextraction (HS-SPME) and liquid-liquid extraction (LLE): Comparison of the performance in classification of ecstasy tablets (Part 2)

HS-SPME was assessed as an alternative to liquid-liquid extraction (LLE) currently used for MDMA profiling. Drug seizures were analysed using both extraction techniques followed by GC-MS. A previously validated method provided data for HS-SPME, whereas LLE data were collected applying a harmonized methodology developed and used in the European project CHAMP. After suitable pre-treatment, similarities between sample pairs were studied using the Pearson correlation. Both methods were capable of distinguishing between samples coming from the same pre-tabletting batches and samples coming from different pre-tabletting batches

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J Anal Toxicol 2008 **32** (8) 653

Tetrahydrocannabinol and two of its metabolites in whole blood using liquid chromatography-tandem mass spectrometry

An analytical procedure for the determination of Δ^9-tetrahydrocannabinol (THC), 11-nor-9-carboxy-Δ^9-tetrahydrocannabinol (THCA), and 11-hydroxy-Δ^9-tetrahydrocannabinol (11-OH-THC) in whole blood has been developed and validated using LC with tandem MS. Cannabinoids present in the blood samples were quantified using solid-phase extraction followed by MS detection in positive electrospray ionization mode. The monitoring of the qualifying transition and requirement for its presence within a specific ratio to the primary ion has the potential of limiting the sensitivity of the assay, however, the additional confidence in the final result as well as forensic defensibility were considered to be of greater importance

Del Mar Ramirez Fernandez M, De Boeck G, Wood M, Lopez-Rivadulla M, Samyn N// Fed Publ Serv Justice, Natl Inst Criminalistics & Criminology, Chaussee de Vilvorde 100, BE-1120 Brussels, Belgium

J Chromatogr B 2008 **875** (2) 465

Simultaneous analysis of THC and its metabolites in blood using liquid chromatography-tandem mass spectrometry

A simple, rapid and highly sensitive and specific method for the extraction and quantification of Δ^9-tetrahydrocannabinol (THC), 11-hydroxy-Δ^9-tetrahydrocannabinol (11-OH-THC) and 11-nor-9-carboxy-Δ^9-tetrahydrocannabinol (THC-COOH) in blood is presented. The method was fully authenticated and comprised simultaneous liquid-liquid extraction (LLE) of the three analytes with hexane:ethyl acetate (90:10, v/v) into a single eluant followed by separation and quantification using LC-MS/MS. Chromatographic separation was achieved using a XBridge C_{18} column eluted isocratically with methanol:0.1% formic acid (80:20, v/v). Selectivity of the method was achieved by a combination of retention time and two precursor-product ion transitions. No instability was observed following repeated freezing and thawing or in processed samples

Gong XY, Kuban P, Scholer A, Hauser PC*// *Univ Basel, Dept Chem, Spitalstr 51, CH-4056 Basel, Switzerland

J Chromatogr A 2008 **1213** (1) 100

Determination of -hydroxybutyric acid in clinical samples using capillary electrophoresis with contactless conductivity detection

A method for the determination of GHB in urine and serum samples with CE using capacitively coupled contactless conductivity detection (CE-C^4D) was developed. The optimized separation buffer consisting of 20 mM of arginine, 10 mM of maleic acid and 30 μM of cetyltrimethylammonium bromide (CTAB) contained 5 mM vancomycin to facilitate the separation of γ-hydroxybutyric acid from β-hydroxybutyric acid (BHB), which is also present in clinical samples. The detection limits in the clinical samples were found to be about 2 μg/ml. The determination of GHB in both types of samples was carried out directly after a fourfold dilution without requiring any derivatization or extraction procedures

Goodwin RS, Darwin WD, Chiang CN, Shih M, Li SH, Huestis MA*// *NIH/NIDA, CDM, IRP, 251 Bayview Blvd, Baltimore, Md 21224, USA

J Anal Toxicol 2008 **32** (8) 562

Urinary elimination of 11-nor-9-carboxy- ^{9}-tetrahydrocannnabinol in cannabis users during continuously monitored abstinence

The time course of 11-nor-9-carboxy-Δ^9-tetrahydrocannnabinol (THCCOOH) elimination in urine was characterized in 60 cannabis users during 24 h monitored abstinence on a closed research unit for up to 30 days. Six thousand, one hundred fifty-eight individual urine specimens were screened by immunoassay and confirmed for THCCOOH by GC-MS following base hydrolysis and liquid-liquid or solid-phase extraction. In 60%, the maximum creatinine normalized concentration occurred in the first urine specimen; in 40%, peaks occurred as long as 2.9 days after admission. Data provide guidelines for interpreting urine cannabinoid test results and suggest appropriate detection windows for differentiating new cannabis use from residual drug excretion

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J Mol Struct 2008 **887** (1-3) 269

Choosing between GC-FTIR and GC-MS spectra for an efficient intelligent identification of illicit amphetamines

A comparative analysis of several expert systems produced for the identification of illicit amphetamines based on their GC–FTIR and GC–MS spectra is provided. The systems were constructed using Artificial Neural Networks (ANNs), and are dedicated to the recognition of amphetamines. Structure–activity relationships are incorporated into the knowledge base, allowing the systems to identify the amphetamines according to their toxicological activity (stimulant or hallucinogenic). Comparisons indicate that GC–FTIR data are much more relevant for the efficiency of the expert systems, probably due to the fact that these spectra constitute a "fingerprint" of the molecular structures

Holler JM, Bosy TZ, Dunkley CS, Levine B, Past MR, Jacobs A// Armed Forces Inst Pathol, Armed Forces Med Examiner System, Div Forensic Toxicology, Rockville, Md 20850, USA

J Anal Toxicol 2008 **32** (6) 428

^{9}-Tetrahydrocannabinol content of commercially available hemp products

Δ^9-Tetrahydrocannabinol (THC) is the main psychoactive compound present in marijuana. THC can also be found, as a contaminant, in some commercially available hemp products marketed in health food stores and on the internet as a good source of essential fatty acids. Analytical results are separated into two groups, products tested prior to and after publication of 21 CFR Part 1308, "clarification of listing of tetrahydrocannabinols." The data presented are a summary of 79 different hemp products tested for THC. THC was separated by a liquid-liquid or solid-liquid extraction, depending upon the product matrix. The amounts indicate that THC levels in currently marketed hemp products are significantly lower than in those products available before 2003 and reported in previous studies

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J Forensic Sci 2008 **53** (5) 1061

Developmental validation of a *Cannabis sativa* STIR multiplex system for forensic analysis

A developmental validation study based on recommendations of the Scientific Working Group on DNA Analysis Methods (SWGDAM) was conducted on a multiplex system of 10 *Cannabis sativa* short tandem repeat loci. Amplification of the loci in four multiplex reactions was tested across DNA from dried root, stem, and leaf sources, and DNA from fresh, frozen, and dried leaf tissue with a template DNA range of 10.0-0.01 ng. The loci were amplified and scored consistently for all DNA sources when DNA template was in the range of 10.0-1.0 ng. Some allelic dropout and PCR failure occurred in reactions with lower template DNA amounts. Overall, amplification was best using 10.0 ng of template DNA from dried leaf tissue indicating that this is the optimal source material. Cross species amplification was observed in *Humulus lupulus* for three loci but there was no allelic overlap. This is the first study following SWGDAM validation guidelines to validate short tandem repeat markers for forensic use in plants

Iwata YT, Kuwayama K, Tsujikawa K, Miyaguchi H, Kanamori T, Inoue H// Nat Res Inst Police Sci, 6-3-1 Kashiwanoha, Kashiwa, Chiba 277 0882, Japan

Rapid Commun Mass Spectrom 2008 **22** (23) 3816

Evaluation method for linking methamphetamine seizures using stable carbon and nitrogen isotopic compositions: A complementary study with impurity profiling

Drug profiling, extraction of physical and/or chemical profiles from abused drug samples, is useful for inferring and characterizing links between samples originating from the same and different seizures, and supports drug crime investigations. An evaluation method is described for linking methamphetamine (MA) seizures using stable carbon and nitrogen isotopic compositions concurrently with gas chromatographic impurity profiling, which is one of the major methods of drug profiling. Several sets of MA seized in Japan, whose investigative information indicated linkages, were analyzed. The impurity profile of each set of seizures was quite similar and hierarchical cluster analysis showed a sample classification that was relatively consistent with the investigative information. The results showed that complementary use of stable-isotopic compositions with impurity profiling provides useful information for evaluating the links between seizures

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Scand J Clin Lab Invest 2008 **69** (1) 113

Determination of amphetamine, methamphetamine, MDA and MDMA in human hair by GC-EI-MS after derivatization with perfluorooctanoyl chloride

A procedure for classical amphetamines and their methylenedioxylated derivatives involving liquid-liquid extraction of hydrolysed hair spiked with deuterated internal standards and direct derivatization with perfluorooctanoyl chloride has been developed. After evaporation of the organic phase and dissolution in butylacetate, the derivatized compounds were injected into a GC-MS. The method, including the derivatization procedure, is simple and robust with a sensitivity that is satisfactory for measurement of amphetamines and ecstasy in hair from abusers

Kato N, Fujita S, Ohta H, Fukuba M, Toriba A, Hayakawa K// Kanagawa Prefectural Police Headquarters, Sci Crime Lab, 155-1 Yamashita cho, Naka ku, Yokohama, Kanagawa 231 002, Japan

J Forensic Sci 2008 **53** (6) 1367

Thin layer chromatography/fluorescence detection of 3,4-methylenedioxymethamphetamine and related compounds

A rapid and sensitive method is described for the detection of six methylenedioxylatedphenethylamines, 3,4-methylenedioxymethamphetamine (MDMA); 3,4-methylenedioxyamphetamine; 3,4-methylenedioxyethylamphetamine; *N*-methyl-1-(3,4-methylenedioxyphenyl)-2-butamine; *N*-methyl-1-(3,4-methylenedioxyphenyl)-3-butamine; and 3,4-methylenedioxydimethylamphetamine, by TLC with fluorescence detection following spraying with a reagent consisting of sodium hypochlorite, potassium hexacyanoferrate (III), and sodium hydroxide. The detection limits for MDMA and the above related compounds were 50 ng

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Arch Pharm Res 2008 **31** (12) 1644

Simultaneous determination of methamphetamine, 3,4-methylenedioxy-*N*-methylamphetamine, 3,4-methylenedioxy-*N*-ethylamphetamine, *N,N*-dimethylamphetamine, and their metabolites in urine by liquid chromatography-electrospray ionization-tandem mass spectrometry

A LC-ESI-MS/MS technique was developed and validated for the simultaneous detection and quantification of seven amphetamine derivatives (amphetamine (AP), methamphetamine (MA), 3,4-methylenedioxy-*N*-amphetamine (MDA), 3,4-methylenedioxy-*N*-methamphetamine (MDMA), 3,4-methylenedioxy-*N*-ethylamphetamine (MDEA), *N,N*-dimethylamphetamine (DMA), and *N,N*-dimethylamphetamine-*N*-oxide (DMANO)) in human urine. Seven deuterium-labeled compounds were prepared for use as internal standards to quantify the analytes. Urine was combined with sodium carbonate buffer solution before solid phase extraction (SPE). An Oasis HLB SPE column followed by chromatographic separation on a Capcell Pak C18 MG-II column and electrospray mass spectrometry with multiple reaction monitoring were used for selective and sensitive detection

Larson SJ, Holler JM, Magluilo J, Dunkley CS, Jacobs A// Armed Forces Inst Pathol, Armed Forces Med Examiner System, Div Forensic Toxicology, Rockville, Md 20850, USA

J Anal Toxicol 2008 **32** (6) 438

Papain adulteration in 11-nor- ^{9}-tetrahydrocannabinol-9-carboxylic acid-positive urine smaples

The adulteration of urine samples is an ongoing problem in forensic drug-testing laboratories, even in the military where the practice of observed collections is performed. It has been reported that papain, a cysteine protease, could be successfully used as a urine adulterant, altering the concentration of 11-nor-Δ^{9}-tetrahydrocannabinol-9-carboxylic acid (THCCOOH) in urine samples. The current study analyzes the effects of latex papain (Sigma, 10 mg/ml) and Lawry's® Adolph's Meat Tenderizer (papain is an active ingredient, 10 mg/ml) on immunoassays (FPIA, EMIT, KIMS) and gas chromatography-mass spectrometry (GC-MS) analysis for biological samples. A decrease in response averaged over the course of the study was observed with FPIA (Abbott, 22%) and EMIT (Syva®) Dade Behring, 26%, Microgenics, 10%) screening assays by the addition of latex papain to the samples. An increase in response was found using the KIMS (Roche) assay (156% increase). In addition, the GC-MS results (27% decrease) demonstrate that papain affects both the screening and confirmation assays. The addition of meat tenderizer caused decrease in the FPIA (Abbott, 11%) screening assay and GC-MS results (22%) similar to the latex papain while having varied results on the other screening assays

Mueller M, Peters FT, Ricaurte GA, Maurer HH*// *Univ Saarland, Inst Expt & Clin Pharmacol & Toxicol, Dept Expt & Clin Toxicol, DE-66421 Homburg, Germany

J Chromatogr B 2008 **874** (1-2) 119

Liquid chromatographic-electrospray ionization mass spectrometric assay for simultaneous determination of 3,4-methylenedioxymethamphetamine and its metabolites 3,4-methylenedioxyamphetamine, 3,4-dihydroxymethamphetamine, and 4-hydroxy-3-methoxymethamphetamine in rat brain

An LC-MS assay for MDMA with electrospray ionization was developed after homogenization of rat brain and enzymatic conjugate cleavage. The method was successfully validated with respect to selectivity, linearity, accuracy, precision, recovery, and matrix effect

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J Planar Chromatogr Mod TLC 2008 **21** (6) 465

Isolation and identification of amphetamines in urine by thin-layer chromatography

No abstract available

Sauer C, Peters FT, Staack RF, Fritschi G, Maurer HH*// *Univ Saarland, Inst Expt & Clin Pharmacol & Toxicol, Dept Expt & Clin Toxicol, DE-66421 Homburg, Saar, Germany

Forensic Sci Int 2008 **181** (1-3) 47

Metabolism and toxicological detection of the designer drug *N*-(1-phenylcyclohexyl)-3-methoxypropanamine (PCMPA) in rat urine using gas chromatography-mass spectrometry

Studies on the metabolism and the toxicological detection of the phencyclidine-derived designer drug PCMPA in rat urine are described using GC-MS. Intake of a common drug users' dose of PCMPA could be detected in rat urine by the authors' systematic toxicological analysis (STA) procedure using full-scan GC-MS after acid hydrolysis, liquid-liquid extraction and microwave-assisted acetylation. The STA should be suitable for proof of an intake of PCMPA also in human urine assuming similar metabolism

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J Chromatogr B 2008 **876** (2) 266

A validated gas chromatographic-electron impact ionization mass spectrometric method for methamphetamine, methylenedioxymethamphetamine (MDMA), and metabolites in mouse plasma and brain

Simultaneous quantification of methamphetamine (MAMP), amphetamine, hydroxy-methamphetamine, methylenedioxymethamphetamine (MDMA, ecstasy), methylenedioxyamphetamine, 3-hydroxy-4-methoxy-methamphetamine, and 3-hydroxy-4-methoxy-amphetamine in mouse plasma and brain was achieved using solid phase extraction and GC-electron impact ionization MS in selected-ion monitoring mode. Recoveries were greater than 91%

Schroeder JL, Marinetti LJ, Smith RK, Brewer WE*, Clelland BL, Morgan SL// *Univ Sth Carolina, Dept Chem & Biochem, 109 Sumter St, Columbia, SC 29208, USA

J Anal Toxicol 2008 **32** (8) 659

The analysis of ^{9}-tetrahydrocannabinol and metabolite in whole blood and 11-nor- ^{9}-tetrahydrocannabinol-9-carboxylic acid in urine using disposable pipette extraction with confirmation and quantification by gas chromatography-mass spectrometry

Essential to forensic laboratories is the desire to find a more sensitive, rapid method of analyzing Δ^9-tetrahydrocannabinol (THC) and metabolite in biological specimens. Disposable pipette extraction (DPX) is a valuable method in extracting THC and 11-nor-Δ^9-tetrahydrocannabinol-9-carboxylic acid (THCc) in blood and THCc in urine. Less waste and solvent usage; smaller specimen volume; clean chromatograms; and utilization of lowcost equipment and consumables were achieved using this method. Prior to extraction, urine specimens were hydrolyzed and proteins precipitated from blood

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J Anal Toxicol 2008 **32** (8) 705

Trace evidence of *trans*-phenylpropene as a marker of smoked methamphetamine

A deceased 31-year-old male was found with paraphernalia that indicated that he may have been smoking abused drugs prior to death. Methamphetamine, cocaine and markers of thermal degradation of methamphetamine and cocaine were also detected in the paraphernalia. GC-MS detected *trans*-phenylpropene as a marker of smoked methamphetamine and anhydroecgonine methyl ester as a marker of smoked cocaine. Both *trans*-phenylpropene and anydroecgonine methyl ester were detected in the urine of the decedent, connecting the link between the paraphernalia for smoking and the ingestion of the pyrolysis products of methamphetamine and cocaine

Toennes SW, Ramaekers JG, Theunissen EL, Moeller MR, Kauert GF// Univ Frankfurt, Inst Forensic Toxicol, Ctr Legal Med, Kennedyallee 104, DE-60596 Frankfurt, Germany

J Anal Toxicol 2008 **32** (7) 470

Comparison of cannabinoid pharmacokinetic properties in occasional and heavy users smoking a marijuana or placebo joint

Δ^9-Tetrahydrocannabinol (THC) results obtained with occasional users were in contrast to those of the heavy users who admitted cannabis use on 4-25 occasions during the previous week. Of the 12 heavy users, 10 exhibited up to 12.3 µg/l THC prior to smoking. During the 8 h after smoking, the distribution and elimination patterns were comparable to those of the occasional users and the concentrations returned to 68-196% (median 110%) of the initial values. The results indicate caution in that cannabinoid blood concentrations from heavy users in a late elimination phase may be difficult to distinguish from concentrations measured in occasional users after acute cannabis use

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Forensic Sci Int 2008 **182** (1-3) 13

Identification of impurities and statistical classification of methamphetamine hydrochloride drugs seized in China

Methamphetamine hydrochloride samples were analyzed using GC-MS and GC-FID. Major impurities detected include 1,2-dimethyl-3-phenylaziridine, ephedrine/pseudoephedrine, 1,3-dimethyl-2-phenylnaphthalene, 1-benzyl-3-methylnaphthalene. These data are suggestive of ephedrine/pseudoephedrine as the main precursor of the methamphetamine hydrochloride samples. Additionally the presence of 1,3-dimethyl-2-phenylnaphthalene, 1-benzyl-3-methylnaphthalene is indicative that they were synthesized *via* the more specific ephedrine/hydriodic acid/red phosphorus method

11 Narcotics

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J Anal Toxicol 2008 **32** (9) 744

Comparison of nonhydrolysis and hydrolysis methods for the determination of buprenorphine metabolites in urine by liquid chromatography-tandem mass spectrometry

A highly sensitive and selective method for the direct determination of buprenorphine (BUP), norbuprenorphine (NBUB), buprenorphine-3-glucuronide, and norbuprenorphine-3-glucuronide in urine was developed and validated. Analytes were extracted by solid-phase extraction on Bond Elut C18, followed by LC-EI tandem MS analysis using a Synergy Polar RP column. Gradient elution was based on a mobile phase consisting of ammonium formate adjusted to pH 3 and acetonitrile. Acceptance criteria for linearity, precision, and recovery were achieved for all analytes. No interference was detected with other common drugs. The described method was compared with an in-house hydrolysis method using 21 real urine case samples. BUP and NBUP were detected using both methods, with higher concentrations obtained using the direct method

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J Anal Toxicol 2008 **32** (8) 601

Rapid detection of opioids in vitreous humor by enzyme immunoassay

Comprehensive and rapid screening of specimens that enter a postmortem forensic toxicology laboratory is essential. Although blood and urine specimens are most commonly utilized for immunoassay screening, this study illustrates the use of vitreous humor for similar purpose. Cases submitted for drug analysis were screened for the presence of opioids in vitreous humor by the Microgenics Cloned Enzyme Donor Immunoassay (CEDIA) DAU opiate assay. Codeine and oxycodone were readily identified by the GC-NPD screen; however, morphine, hydrocodone, hydromorphone, and 6-acetylmorphine were detected primarily by gas chromatography-mass spectrometry

Frisk T, Sandstrom N, Eng L, Van der Wijngaart W, Mansson P, Stemme G// Royal Inst Technol, Microsystem Technology Lab, Stockholm, Sweden

Lab Chip 2008 **8** (10) 1648

An integrated QCM-based narcotics sensing microsystem

An integrated electronic narcotics sensing system has been developed. The microsystem absorbs airborne narcotics molecules and performs a liquid assay using an integrated quartz crystal microbalance (QCM). A vertically conductive double-sided adhesive foil (VCAF) was employed and studied as a novel material for LOC and MEMS applications and facilitates easy assembly, electrical contacting and liquid containment. The system was tested for measuring cocaine and ecstasy, with successful detection of amounts as small as 100 ng and 200 ng, respectively

Fuller DC// Univ Texas SW Med Ctr Dallas, 4500 Sth Lancaster Rd, B113, Dallas, Tx 75216, USA

J Anal Toxicol 2008 **32** (8) 626

A simple gas chromatography-mass spectrometry procedure for the simultaneous determination of buprenorphine and norbuprenorphine in human urine

To monitor opiate addiction treatment, the present procedure is simple, efficient, and employs GC-MS. The specimen is hydrolyzed with β-glucuronidase prior to liquid-liquid extraction at a basic pH. The evaporated extract is derivatized to form the tertiary-butyl-dimethyl-silyl derivatives of buprenorphine and norbuprenorphine prior to analysis by GC-MS in the electron impact mode. Confirmation of the analytes is based on comparing the ion abundance ratios of the analytes to those of a contemporaneously analyzed standard

Gambelunghe C, Aroni K, Rossi R, Moretti L, Bacci M// Sezione Med Legale & Med Specialist Sport, Dipt Med Clin & Sperimentale, IT-06123 Perugia, Italy

Biomed Chromatogr 2008 **22** (10) 1056

Identification of *N,N*-dimethyltryptamine and -carbolines in psychotropic ayahuasca beverage

Gas chromatography/mass spectrometry analysis identified *N,N*-dimethyltryptamine, a potent hallucinogen, and the β-carboline alkaloids harmine and harmaline, revealing monoamine oxidase A-inhibiting properties. These substances are typical components of ayahuasca, a South American psychotropic beverage obtained by boiling the bark of the liana *Banisteriopsis caapi* together with the leaves of various admixture plants, principally *Psychotria viridis*

Garcia Boy R, Henseler J, Mattern R, Skopp G// Univ Hosp, Inst Legal Med & Forensic Med, Voss Str 2, DE-69115 Heidelberg, Germany

Ther Drug Monit 2008 **30** (6) 733

Determination of morphine and 6-acetylmorphine in blood with use of dried blood spots

Injection diacetylmorphine use is notably associated with a prevalence of infection and a risk of transmission of blood-borne viruses. The aim of the present study was to establish a method to determine morphine and 6-acetylmorphine (6-AM) as accurately and sensitively from dried blood spots as from whole blood. Analysis by LC/tandem MS was checked for carryover, ion suppression/enhancement, linearity of response, lower limits of detection and quantification, and the within-run and between-run assay imprecision for both whole blood and DBS after liquid/liquid extraction

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Talanta 2008 **77** (2) 522
Exploiting sequential injection analysis technique to automate on-line sample treatment and quantitative determination of morphine in human urine
A simple uni-stream sequential injection analysis (SIA) manifold was produced to automate the assay of morphine in human samples. The technique includes on-line sample treatment, coupling reaction and spectrophotometric measurement. Sample treatment involved solid-phase extraction (SPE) into a home-made microcolumn, installed in the SIA manifold. A coupling reaction of morphine with diazonium salt of aniline hydrochloride was adapted to SIA. The product of the reaction, an azo-morphine derivative, was spectrophotometrically detected at 390 nm. Limits of detection and quantification were 0.023 and 0.076 μg ml/l. The method is suitable for the application in forensic cases

Johansen SS, Linnet K// Frederik Vs vej 11, DK-2100 Copenhagen O, Denmark
J Anal Toxicol 2008 **32** (7) 499
Chiral analysis of methadone and its main metabolites EDDP in postmortem blood by liquid chromatography-mass spectrometry
A chiral LC-MS-MS method was developed for the measurement of methadone and 2-ethyl-1,5-dimethyl-3,3-diphenylpyrrolinium (EDDP) enantiomers in postmortem blood. Chromatographic separation was performed on a chiral-AGP analytical column with a mobile phase of acetonitrile/ammonium acetate buffer. A Quattro micro mass spectrometer was operated in the positive ion mode with an electrospray source. Multiple reaction monitoring was used with two transitions for each compound

Kacinko SL, Concheiro-Guisan M, Shakleya DM, Huestis MA*// *NIH/Natl Inst Drug Abuse, Intramural Res Program, 251 Bayview Blvd, Baltimore, Md 21224, USA
Anal Bioanal Chem 2008 **392** (5) 903
Development and validation of a liquid chromatography-tandem mass spectrometry assay for the simultaneous quantification of buprenorphine, norbuprenorphine, and metabolites in human urine
A liquid chromatography-tandem mass spectrometry method for the simultaneous quantification of buprenorphine (BUP), norbuprenorphine (NBUP), buprenorphine glucuronide (BUP-Gluc), and norbuprenorphine glucuronide (NBUP-Gluc) in human urine has been developed and fully validated. Extensive endogenous and exogenous interferences were evaluated. Analytical ranges were 5-1,000 ng/ml for BUP and BUP-Gluc and 25-1,000 ng/ml for NBUP and NBUP-Gluc. Analytes were stable at room temperature, at 4°C, and for three freeze-thaw cycles. This accurate and precise assay has sufficient sensitivity and specificity for urine analysis of specimens collected from individuals treated with BUP for opioid dependence

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J Anal Toxicol 2008 **32** (8) 586
Urinary detection times and metabolite/parent compound ratios after a single dose of buprenorphine
Urine samples were screened using cloned enzyme donor immunoassay (CEDIA) reagent and quantitation was performed with liquid chromatography-tandem mass spectrometry (LC-MS-MS) with a cut-off of 0.5 ng/ml for buprenorphine and norbuprenorphine. The mean time of continuous positive results was 9 h (range 4 to 24 h) with CEDIA, whereas for an LC-MS-MS method it was 76 h (range 23-96 h) for buprenorphine, and for norbuprenorphine all samples were positive at 96 h. Some subjects had positive CEDIA results after a negative sample, owing to differences in creatinine concentration

Lin YH, Chiang JF, Lee MR, Lee RJ, Ko WK, Wu SM*// *Kaohsiung Med Univ, Coll Pharm, 100 Shih Chung 1st Rd, Kaohsiung 80708, Taiwan
Electrophoresis 2008 **29** (11) 2340
Cation-selective exhaustive injection and sweeping micellar electrokinetic chromatography for analysis of morphine and its four metabolites in human urine
A cation-selective exhaustive injection and sweeping micellar EKC (CSEI-Sweep-MEKC) was established to analyze morphine and its four metabolites, including codeine, normorphine (NM), morphine-3-glucuronide (M3G), and morphine-6-glucuronide (M6G). After SPE, the urine samples were analyzed by this CE method. The stacking CE method could increase 2500-fold sensitivity of codeine, when comparing with CZE

Luckenbill K, Thompson J, Middleton O, Kloss J, Apple F*// *Hennepin County Med Ctr, Clin Labs P4, 701 Park Ave, Minneapolis, Mn 55415, USA
J Anal Toxicol 2008 **32** (8) 639
Fentanyl postmortem redistribution: Preliminary findings regarding the relationship among femoral blood and liver and heart tissue concentrations
Postmortem redistribution refers to the process of drugs diffusing from tissues into blood along a concentration gradient between death and time of specimen collection at autopsy. Femoral blood, liver, and heart fentanyl concentrations were compared in medical examiner cases to assist in determining which specimen most appropriately should be used for interpretation. Utilizing a published compendium of multiple postmortem drugs, liver and heart tissues to femoral blood drug ratios were compared to known volumes of distribution, solubilities, and pKa. No significant relationships were observed. In conclusion, establishing a larger evidence-based database using liver fentanyl concentrations may be more optimal than blood concentrations for interpretation of postmortem fentanyl concentrations in medical examiner and coroner cases

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J Chromatogr Sci 2008 **46** (6) 551
Gas chromatographic retention indices of fentanyl and analogues
Herein, we report retention indices of fentanyl and its eighteen analogues relative to the homologous *n*-alkane series. These values are determined on a moderately polar BP-5 capillary column under programmed temperature and isothermal chromatographic conditions. The effects of chromatographic conditions like temperature programming rate, carrier gas flow rate, and oven temperature are studied. Retention indices are also determined on a non-polar BP-1 column to study the influence of stationary phase polarity

Moros J, Galipienso N, Vilches R, Garrigues S*, De la Guardia M// *Univ Valencia, Dept Anal Chem, 50th Dr Moliner, ES-46100 Valencia, Spain
Anal Chem 2008 **80** (19) 7257
Nondestructive direct determination of heroin in seized illicit street drugs by diffuse reflectance near-infrared spectroscopy
A new fast and nondestructive direct determination of heroin in seized street illicit drugs using partial least-squares regression analysis of diffuse reflectance near-infrared spectra is presented. Results were obtained from untreated samples placed in standard glass chromatography vials. A heterogeneous population of 31 samples, previously analyzed by a reference method, was employed to build the calibration model and to have a separated validation set

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Anal Chim Acta 2008 **630** (2) 150
New cut-off criterion for uninformative variable elimination in multivariate calibration of near-infrared spectra for the determination of heroin in illicit street drugs
Uninformative variable elimination-partial least squares (UVE)-PLS and PLS were applied to diffuse reflectance near-infrared spectra of heroin samples. The application of a proposed new cut-off criterion, based on the t-Students distribution, provided similar predictive capabilities of the PLS models than those obtained using the original criteria based on quantile value. However, the repeatability of the number of selected variables was improved significantly

Nikolaou PD, Papoutsis II, Maravelias CP, Spiliopoulou CA, Pistos CM, Calokerinos AC, Atta-Politou J// Univ Athens Sch Med, Dept Forensic Med & Toxicol, GR-11527 Athens, Greece
J Anal Toxicol 2008 **32** (7) 478
Development and validation of an EI-GC-MS method for the determination of methadone and its major metabolites (EDDP and EMDP) in human breast milk
Methadone and its two major metabolites, 2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine (EDDP) and 2-ethyl-5-methyl-3,3-diphenylpyrroline (EMDP) appear in breast milk and so a sensitive and specific GC-MS method has been developed, optimized, and validated for their quantitative determination. The procedure combined protein precipitation with acetonitrile and solid-phase extraction, using Isolute Confirm HCX mixed-mode SPE columns, with minimal matrix effect. The optimum extraction conditions for all three analytes were evaluated using spiked human breast milk, and the recovery exceeded 93.0%. Analytes were found to be stable in breast milk at room temperature for at least 4 h and at -20°C for at least one month

Parkin MC, Turfus SC, Smith NW, Halket JM, Braitwaite RA, Elliott SP, Osselton MD, Cowan DA, Kicman AT*// *KCL, Dept Forensic Sci & Drug Monitoring, 150 Stamford St, London SE1 9NH, England
J Chromatogr B 2008 **876** (1) 137
Detection of ketamine and its metabolites in urine by ultra high pressure liquid chromatography-tandem mass spectrometry
Analysis of ketamine and its metabolites was performed using UPLC-MS/MS following extraction of urine using mixed-mode (cation and C8) solid-phase cartridges. As ketamine and norketamine (including their stable isotopes) are available as reference standards, the assay was additionally validated for quantification purposes to study elimination of the drug and primary metabolite following a small oral dose of ketamine to volunteers. Dehydronorketamine, a secondary metabolite, was also analyzed qualitatively to determine whether monitoring could improve retrospective detection of administration. The detection limit for ketamine and norketamine could be confirmed in urine for up to 5 and 6 days, respectively whereas dehydronorketamine was confirmed up to 10 days, providing a very broad window of detection

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J Electroanal Chem 2008 **624** (1-2) 293
Simultaneous voltammetric and amperometric determination of morphine and codeine using a chemically modified-palladized aluminum electrode
The use of Prussian blue film modified-palladized aluminum electrode (PB/Pd-Al) prepared by a simple and rapid electroless method for the determination of codeine (CO) and morphine (MO) is presented. The oxidation of CO and MO at the modified electrode was investigated by cyclic voltammetry. The electro-oxidation pathway for the both two compounds in the neutral medium (pH 6) was proposed. Hydrodynamic amperometry was used for the determination of CO and MO at the μM concentration level

Thompson JG, Vanderwerf S, Seningen J, Carr M, Kloss J, Apple FS*// *Hennepin County Med Ctr, Clin Labs P4, 701 Park Ave, Minneaplois, Mn 55415, USA
J Anal Toxicol 2008 **32** (8) 673
Free oxycodone concentrations in 67 postmortem cases from the Hennepin County Medical Examiner's Office
Heart blood free oxycodone concentrations in oxycodone-related and mixed drug overdose deaths were compared with those found incidentally at autopsy in medical examiner cases. The findings substantiate the considerable overlap that exists with blood oxycodone concentrations in cases where oxycodone alone was determined to be the cause of death when compared with mixed drug overdoses and incidental findings. Free oxycodone concentrations in postmortem cases should be interpreted in the context of the deceased's past medical history and autopsy findings

VandenBoer TC, Grummett SA, Watterson JH*// *Laurentian Univ, Dept Forensic Sci, Forensic Toxicology Res Lab, 935 Ramsey Lake Rd, Sudbury, Ontario, Canada P3E 2C6
J Forensic Sci 2008 **53** (6) 1474
Utility of immunoassay in drug screening in skeletal tissues: Sampling considerations in detection of ketamine exposure in femoral bone and bone marrow following acute administration using ELISA
Detection of ketamine exposure in skeletal tissues of rats by automated enzyme-linked immunosorbent assay (ELISA) and gas chromatography with electron capture detection (GC-ECD) is described. Ketamine was extracted from ground femoral bone by methanolic incubation followed by liquid-liquid extraction (LLE), while marrow was homogenized in alkaline solution, and then underwent LLE. Extracts were analyzed by ELISA, and subsequently by GC-ECD following derivatization with trifluoroacetic acid anhydride. Results indicate that the type of skeletal tissue sampled and position sampled within a given bone (diaphyses *vs*. epiphyses) are important parameters in drug screening of skeletal tissues

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Rev Roum Chim 2008 **53** (12) 1157
Bioanalysis of methadone in human plasma and urine by LC/MS/MS
Methadone in human plasma and urine was analysed by a rapid LC/MS/MS technique. Separation was performed on a Zorbax SB-C18 column under isocratic conditions using a mixture of acetonitrile and formic acid in water. Detection was in the MRM mode. Human plasma samples were deproteinisated with methanol and the urine samples were diluted with bidistilled water. The method showed a good linearity, precision and accuracy over the range of 10-1000 ng/ml in plasma and 20-2000 ng/ml in urine

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J Anal Toxicol 2008 **32** (8) 631
Effects of tissue type and the dose-death interval on the detection of acute ketamine exposure in bone and marrow with solid-phase extraction and ELISA with liquid chromatography-tandem mass spectrometry confirmation
Ketamine exposure was detected in skeletal tissues of rats by ELISA and LC-MS-MS. Extracted femora were separated into epiphyseal and diaphyseal fragments, with marrow isolated from the medullary cavity. Bone was ground and incubated in methanol. Extracts were dried and reconstituted in phosphate buffer (0.1 M, pH 7.3), and marrow was homogenized in alkaline solution. Both then underwent solid-phase extraction. Results suggest that the tissue type sampled and dose-death interval may influence the sensitivity of detection of ketamine exposure in skeletal tissues

12 Forensics

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J Anal Toxicol 2008 **32** (8) 680
A validated method for the quantitation of 1,1-difluoroethane using a gas in equilibrium method of calibration
Abuse of 1,1-difluoroethane (DFE), also known as Freon 152A has necessitated development of methods for its detection and quantitation in postmortem and human performance specimens. This paper describes a method for the quantitation of DFE using a gas chromatography-flame-ionization headspace technique that uses solventless standards for calibration. The method is suitable for use in forensic laboratories. In addition, it facilitates research oriented studies because the removal of solvent from standard preparation eliminates the possibility for solvent induced changes to the gas/liquid partitioning of DFE or chromatographic interference due to the presence of solvent in specimens

Caboni P, Sarais G, Vargiu S, De Luca MA, Garau VL, Ibba A, Cabras P// Univ Cagliari, Dept Toxicol, Via Ospedale 72, IT-09124 Cagliari, Italy
Chromatographia 2008 **68** (9-10) 739
LC-MS-MS determination of rotenone, deguelin, and rotenolone in human serum
A new rapid and sensitive LC–MS–MS method for quantification of rotenone, deguelin, and rotenolone in human serum is reported. The analytical procedure involves extraction with ethyl acetate without further clean-up. Active ingredients were separated on a C_8 reversed-phase column by isocratic elution. Eleven simultaneous transitions of precursor ions were monitored

Copper CL, Newman CID, Collins GE*// *Naval Res Lab, Chem Div, 4555 Overlook Ave SW, Code 6112, Washington, DC 20375, USA
J Sep Sci 2008 **31** (21) 3727
Simple and rapid extraction, separation, and detection of alkaloids in beverages
An uncomplicated SPE technique has been developed for the rapid extraction and preconcentration of the alkaloids, colchicine, strychnine, aconitine, and nicotine, from water, apple juice, and nonfat milk samples. When coupled to analysis via micellar EKC (MEKC), the total analysis time per sample was less than 15 min for the water and juice samples and less than 20 min for the milk. SPE resulted in between a three and a fourteen-fold improvement in the LOD for each alkaloid when compared to detecting the alkaloids in a nontreated water sample matrix. After SPE, the LODs for colchicine, strychnine, and nicotine were sufficient to meet levels from 150 to 5000 times more dilute than the LD_{50} for a 50 kg individual drinking 12 oz of a contaminated beverage. Aconitine, on the other hand, was detected at approximately the LD_{50} level

Daundkar BB, Malve MK, Krishnamurthy R*// *Home Dept State Maharashtra, Santa Cruz (E) Directorate Forensic Sci Labs, Kalina, Vidyanagari, Hans Bhugra Marg, IN-400098 Mumbai, India
J Planar Chromatogr Mod TLC 2008 **21** (4) 249
A specific chromogenic reagent for detection of diazepam among other benzodiazepines from biological and nonbiological samples after HPTLC

The detection of the benzodiazepine drug diazepam in biological tissues, blood, urine, vomit, and tablets by a new sensitive chromogenic TLC spray reagent is described. Diazepam standard and extracts of these samples in alkaline chloroform-isopropyl alcohol 9:1 were applied to TLC plates coated with 0.25 mm layers of silica gel G. Plates were developed with chloroform-methanol 9:1 as mobile phase, dried, and then sprayed with 5% sodium hydroxide solution followed by 1% *m*-dinitrobenzene in dimethyl sulfoxide (DMSO). Diazepam produced violet bands but other benzodiazepines, for example oxazepam, nitrazepan, lorazepam, chlordiazepoxide, and flurazepam, did not react

De Castro A, Concheiro M, Quintela O, Cruz A, Lopez-Rivadulla M*// *Univ Santiago de Compostela, Inst Med Legale Forensic Toxicol Serv, C/ San Francisco s/n, ES-15782 Santiago de Compostela, Spain

J Pharm Biomed Anal 2008 **48** (1) 183

LC-MS/MS method for the determination of nine antidepressants and some of their main metabolites in oral fluid and plasma - Study of correlation between venlafaxine concentrations in both matrices

A fast, sensitive and selective LC-MS/MS method is described for the simultaneous determination of amitriptyline, imipramine, clomipramine, fluoxetine, paroxetine, sertraline, fluvoxamine, citalopram and venlafaxine, as well as some of their main metabolites (nortriptyline, desipramine, norclomipramine and norfluoxetine), in oral fluid and plasma. The sample was extracted with an automated solid-phase extraction system (ASPEC XL), using mixed mode OASIS MCX cartridges. Chromatographic separation was performed in a Sunfire C18 IS column using a gradient of acetonitrile and ammonium formate as the mobile phase which allowed the elution of all the compounds in less than 5 min. The method has been fully validated in both body fluids

DeRienz RT, Holler JA, Manos ME, Jemionek J, Past MR// Armed Forces Inst Pathol, Armed Forces Med Examiner System, Div Forensic Toxicology, Rockville, Md 20850, USA

J Anal Toxicol 2008 **32** (6) 433

Evaluation of four immunoassay screening kits for the detection of benzodiazepines in urine

The performance of four commercially available immunoassay urine screening kits were evaluated for use in a forensic urine analysis testing program of benzodiazepines (BZD). The four kits included the Roche Benzodiazepine Plus KIMS assay, Microgenics CEDIA Benzodiazepine assay, Microgenics CEDIA high sensitivity assay with β-glucuronidase, and Microgenics DRI reagent ready Benzodiazepine assay. Each kit was evaluated for linearity, precision, accuracy, carryover, reagent specificity, and confirmation rates

El-Haj B, Al-Amri A, Ali H// SHJ Police Forensic Sci Lab, Sharjah, United Arab Emirates

J Anal Toxicol 2008 **32** (9) 790

Cross-reactivity of nefopam and its metabolites with benzodiazepine EMIT immunoassay (Letter)

No abstract available

ElSohly MA, Gul W, Avula B, Murphy TP, Khan IA// ElSohly Labs Inc, 5 Ind Park Rd, Oxford, Ms 38655, USA

J Anal Toxicol 2008 **32** (8) 547

Simultaneous analysis of thirty-five benzodiazepines in urine using liquid chromatography-mass spectrometry-time of flight

Using LC-MS-ToF procedure, the limit of quantitation for all benzodiazepines tested ranged from 0.1 to 10 ng/ml, and the limit of determination range was 0.03 to 3.0 ng/ml. The method was used for the analysis of 111 forensic/clinical samples previously tested for benzodiazepines by immunoassays (81 positives and 30 negatives). All immunoassay positive specimens were confirmed by this procedure for one or more analytes. However, only 12 out of the 35 benzodiazepines analyzed were detected in these specimens

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J Anal Toxicol 2008 **32** (8) 621

Comparison of drug concentrations taken from clamped and unclamped fermoral vessels

Postmortem drug concentrations may vary depending on sampling site, volume of blood collected, and method of sampling. Drug concentrations of three selective serotonin reuptake inhibitors, or SSRIs (sertraline, paroxetine, citalopram), two benzodiazepines (diazepam and alprazolam), two antihistamines (diphenhydramine and promethazine), and one opiate (hydrocodone) were evaluated in clamped femoral blood, blind stick femoral blood, and heart blood and compared using concentration ratios and linear regression analysis. Clamped femoral blood concentrations and blind stick femoral blood concentrations were found to have good predictability across all drug classes with ratios around 1.0, indicating good correlation between blind stick femoral and clamped femoral samples

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J Mass Spectrom Soc Jpn 2008 **56** (3) 131

Application of new mass spectrometric techniques to legal medicine (Japanese, English Abstract)

The application of surface ionization organic MS, a combination of MS and cryogenic oven trapping (COT), and laser spray ionization (LSI) to legal medicine is described. GC/surface ionization organic MS is very sensitive in detecting drugs containing tertiary amine residues and for phencyclidine (PCP), its sensitivity is 20 to 1,000 times higher than that of the conventional EI method. Surface ionization organic MS is suitable for compounds containing cyclic tertiary amino side chains. COT in combination with GC has facilitated detection of many volatile organic compounds (VOCs) with high sensitivity. Several VOCs, such as chloroform, cyanide, thinner components, ethanol, and general anesthetics were quantified in whole blood by COT; COT affords a sensitivity that is 10 to 50 times higher than that of the conventional headspace GC method. In addition, 15 VOCs were identified and quantified in human whole blood with the combination of COT and MS. LSI is a new ionization method for LC/MS. Aconitine alkaloids and psychopharmaceuticals were detected using LSI. In the infusion mode, aconitine and haloperidol were ionized with higher efficiency compared with that in the conventional electrospray mode

Legrand T, Roy S, Monchaud C, Grondin C, Duval M, Jacqz-Aigrain E*// *Hop Robert Debre, AP-HP, Serv Pharmacol Pediat & Pharmacogenet, FR-75019 Paris, France

J Pharm Biomed Anal 2008 **48** (1) 171

Determination of ketamine and norketamine in plasma by micro-liquid chromatography-mass spectrometry

A sensitive method of determination of ketamine and norketamine by micro-liquid chromatography/mass spectrometry has been developed. Compounds were extracted from 100 µl plasma samples using solid-phase extraction on Oasis MCX cartridges. Separation was achieved on a C18 micro-column with a mobile phase composed of formic acid 0.1% and acetonitrile (90/10, v/v). Detection of ketamine, norketamine and their internal standard norketamine D4 was performed using selected ion monitoring. No loss of signal due to matrix effect was observed and time of analysis did not exceed 10 min. Extracted calibration curves were linear from 5 to 500 ng/ml for each analyte. Intra- and inter-day validation studies showed mean recoveries between 98.1 and 101.7%. Extraction recoveries ranged from 84.8 to 89.8% for both ketamine and norketamine. Limit of quantification was 4 ng/ml for each analyte

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Anal Lett 2008 **41** (14) 2564

Analysis of cyanide in blood by headspace-isotope-dilution-GC-MS

Analysis of low cyanide concentrations in whole blood by an uncomplicated, rapid, automated procedure is reported. The analysis was performed by headspace GC and JMS. Carryover from cyanide adsorption onto the surface of the needle was prevented by developing a new method that enabled automated flushing of the needle in between each cyanide analysis. Results were compared of ordinary calibrations and those of isotope dilutions. Analysis time was 18 min for a single cyanide sample

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J Anal Toxicol 2008 **32** (8) 715

Distribution of etomidate in a fatal intoxication

Whereas etomidate is often found during a postmortem drug screen conducted for medicolegal reasons, concentrations of in fluids/tissues have not been reported previously. A case of suicide by etomidate with concentrations of 0.40 mg/l in the femoral blood, 0.46 mg/l in the bile, and 0.30 mg/l in the vitreous with a blood alcohol content of 0.119 g/dl is reported. As a comparison, two cases are reported where etomidate was administered during resuscitation after trauma with levels of 0.05 mg/l and < 0.026 mg/l, respectively

Nakamae T, Shinozuka T, Sasaki C, Ogamo A, Murakami-Hashimoto C, Irie W, Terada M, Nakamura S, Furukawa M, Kurihara K// Int Univ Hlth & Welfare, Dept Pharmaceut Sci, 2600-1 Kitakanemaru, Ohtawara, Tochigi 324 8501, Japan

Forensic Sci Int 2008 **182** (1-3) e1

Case report: Etizolam and its major metabolites in two unnatural death cases

Simultaneous analysis of etizolam and its main metabolites (α-hydroxyetizolam and 8-hydroxyetizolam) was achieved in whole bloodusing solid-phase extraction, TMS derivatization and ion trap GC-MS/MS. Separation of etizolam, TMS derivatives of alpha-hydroxyetizolam and 8-hydroxyetizolam and fludiazepam as internal standard was performed. This method is satisfactory for clinical and forensic purposes

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J Agric Food Chem 2008 **56** (23) 11139

Quantitation of abrine, an indole alkaloid marker of the toxic glycoproteins abrin, by liquid chromatography/tandem mass spectrometry when spiked into various beverages

Abrine is an alkaloid chemical marker and surrogate analyte of abrin, a group of highly toxic glycoproteins which can be easily isolated from the seed of the rosary pea plant and distributed in a variety of matrices, including food. A procedure for the cleanup of abrine from various beverages, including milk, cola, juice drink, tea, and water, by C18 Strata-X solid-phase extraction (SPE) cartridges is described with comparison to a previously developed liquid-liquid extraction protocol utilizing acetonitrile and water. Analysis was by LC/MS. The method detection limit was 0.025 µg/mL, and the quantitation limit was 0.05 µg/ml

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J Anal Toxicol 2008 **32** (8) 709

Direct injection mass spectrometric confirmation of multiple drugs in overdose cases from postmortem blood using electrospray ionization-tandem mass spectrometry and MS^3

Whole blood was analyzed by a direct injection multi-stage mass spectrometric (MS^n) method to confirm the identity of 26 drug analytes at or above 10 ng/ml using 16 different deuterium-labeled internal standards. Samples were spiked with internal standards, precipitated with acetonitrile, and centrifuged. Samples were further diluted with either 0.1% formic acid or 0.1% ammonium hydroxide in methanol prior to injection into an electrospray ionization ion trap mass spectrometer (MS). Ions were monitored as MS-MS or MS^3 product ions. In all cases, analysis by MS-MS confirmed the presence of the drugs and metabolites when the internal standards were detected. Detection of characteristic MS^3 ions was used for further confirmation of the presence of parent drugs in all but three instances. Total analysis time was less than 1 h. The method was only useful for qualitative or confirmatory purposes

Pos Pok PR, Haddouche D, Mauras M, Kuhlmann E, Burle J, Salmon T, Berland E, Coiffait PE, Viala A// INPS, Lab Police Sci Marseille, Sect Toxicol, 97 Blvd Camille Flammarion, BP30, FR-13245 Marseille 4, France

J Anal Toxicol 2008 **32** (9) 782

Cardiac and peripheral blood similarities in the comparison of nordiazepam and bromazepam blood concentrations

Concomitant heart and peripheral blood determinations were performed on 40 fatal cases involving nordiazepam (20 cases) and bromazepam (20 cases). The heart blood concentration for the two drugs did not differ from the corresponding peripheral blood concentration. No postmortem redistribution was observed for these two benzodiazepines. It is suggested that corresponding heart blood can be proposed in the quantitative analysis of these drugs when peripheral blood is unavailable. The present study also shows the stability of the two drugs after a year of storage

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J Anal Toxicol 2008 **32** (8) 688

The incidence of zolpidem use in suspected DUI drivers in Miami-Dade Florida: A comparative study using immunalysis zolpidem ELISA KIT and gas chromatography-mass spectrometry screening

The objective of this study was to evaluate, and retrospectively compare, the use of the Immunalysis ELISA kit and GC-MS to screen blood/urine specimens for zolpidem. Urine and blood samples were screened *via* the Immunalysis Zolpidem ELISA kit and on GC-MS in full EI scan mode following an alkaline liquid-liquid extraction. Results show 5% of the urine and blood samples screened positive for zolpidem using the ELISA kits, and all 5% confirmed positive for zolpidem using GC-MS. The ELISA kit demonstrated no cross-reactivity to zaleplon or zopiclone at a spiked urine concentration of 1000 ng/ml

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Anal Bioanal Chem 2008 **392** (7-8) 1299

Unraveling the metabolic transformation of tetrazepam to diazepam with mass spectrometric methods

The metabolic transformation pathways of the 1,4-benzodiazepine tetrazepam were investigated with capillary LC-QqTOF-MS and -MS/MS by analyzing human plasma and urine samples collected from healthy volunteers. Full-scan fragment ion mass spectra were collected in subsequent LC/MS/MS experiments. Each spectrum was matched to a spectral library containing 3759 MS/MS-spectra of 402 compounds, including eighteen different benzodiazepines, to prove the structural relatedness of a tentative metabolite to tetrazepam

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J Anal Toxicol 2008 **32** (8) 644

Quantitation of benzodiazepines in whole blood by electron impact-gas chromatography-mass spectrometry

Benzodiazepines are frequently encountered in forensic toxicology. A literature search was conducted to find a simple method using EI-GC-MS. The procedure was then developed and validated as a rapid and efficient method for the screening and quantitation of benzodiazepines in blood using liquid-liquid extraction and EI-GC-MS in selective ion monitoring mode. Target compounds included diazepam, desalkylflurazepam, nordiazepam, midazolam, oxazepam, temazepam, lorazepam, clonazepam, and alprazolam

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J Sep Sci 2008 **31** (21) 3704

Validation of SPE-HPLC determination of 1,4-benzodiazepines and metabolites in blood plasma, urine and saliva

A simple, sensitive, selective, and reproducible RP-HPLC method with DAD detection at 240 nm was developed for the determination of six 1,4-benzodiazepines: bromazepam (BRZ), clonazepam (CLZ), diazepam (DZP), flunitrazepam (FNZ), lorazepam (LRZ), alprazolam (APZ); and two metabolites: α-hydroxyalprazolam (HALZ) and α-hydroxytriazolam (HTZL) in human plasma, urine, and saliva, using colchicine as internal standard, after SPE using Nexus Varian cartridges. Separation was achieved with a Kromasil C_8 (250 mm x 5 mm, 5 µm) analytical column with a gradient mobile phase containing methanol, ACN and 0.05 M ammonium acetate

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J Anal Toxicol 2008 **32** (7) 522

Detection of clenbuterol in heroin users in twelve postmortem cases at the Philadelphia Medical Examiner's Office

The presence of clenbuterol is reported in a series of 12 postmortem cases in which the cause of death was attributed to illicit drug use. Confirmation of clenbuterol was performed using solid-phase extraction, derivatization with trimethylboroxine, and analysis utilizing a GC-MS operated in the full-scan mode. Its detection was an unexpected finding. Its presence in these cases serves as a caution to emergency room physicians and toxicologists to consider and test for clenbuterol when treating a suspected heroin user who presents atypically

Xu XM, Song GL, Zhu Y, Zhang J, Zhao YX, Shen HT, Cai ZX, Han JL, Ren YP*// *Zhejiang Province Ctr Dis Control & Prevention, CN-310051 Hangzhou, Zhejiang, Peoples Rep China

J Chromatogr B 2008 **876** (1) 103

Simultaneous determination of two acute poisoning rodenticides tetramine and fluoroacetamide with a coupled column in poisoning cases

A coupled column system was developed for the simultaneous determination of both rodenticides fluoroacetamide and tetramine in this paper by GC/MS. The precisions of the coupled column were analyzed with peak area and retention time. Good linear correlations were found for both. Typical samples were discussed for each rodenticide and some poisoning cases presented

13 Alcohol

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Int J Legal Med 2008 **122** (5) 429

Was a child poisoned by ethanol? Discrimination between ante-mortem consumption and post-mortem formation

Since its distribution in the different fluids and tissues remains contentious, the determination of specific metabolites of ethanol such as ethyl glucuronide (EtG) may be performed to discriminate between exogenous (ante-mortem) and endogenous (post-mortem) before drawing conclusions on the origin of the ethanol. In order to discriminate between ante-mortem alcohol administration and post-mortem formation, the presence of microorganisms capable of ethanol production was checked by fermentation tests and the liver was tested for the presence of EtG and compared with a positive control. Fermentation tests illustrated the presence of *Lactococcus garvieae*, a bacterium capable of producing ethanol from glucose. The absence of EtG in the liver compared to the high level detected in that of a control is a further indication that the ethanol detected in the body of the deceased is of post-mortem origin

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Ther Drug Monit 2008 **30** (6) 725

Liquid chromatography with tandem mass spectrometric detection for the measurement of ethyl glucuronide and ethyl sulfate in meconium: New biomarkers of gestational ethanol exposure?

A LC-MS/MS method with postcolumn addition of acetonitrile for the determination of ethyl glucuronide and ethyl sulfate in meconium was developed and validated using pentadeuterated ethyl glucuronide and pentadeuterated ethyl sulfate as internal standards. The analytes were extracted from the matrix by acetonitrile, concentrated by solid phase extraction, separated using a reversed-phase chromatographic column, and quantified within 9 minutes. Lower limits of quantification were 5 and 1 ng/g meconium for ethyl glucuronide and ethyl sulfate, respectively

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J Anal Toxicol 2008 **32** (7) 505

Comparison among plasma, serum, and whole blood ethanol concentrations: Impact of storage conditions and collection tubes

This paper explores systematic differences in ethanol levels among several methods of processing and storing blood samples. Samples for plasma and whole blood were drawn into Vacutainers containing either an anticoagulant or an anticoagulant plus preservative. Samples for serum were drawn into Vacutainers containing no additives or a preservative only. Neither processing condition (i.e., type of additive) nor storage condition significantly affected ethanol levels

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J Pharm Biomed Anal 2008 **48** (3) 927

Liquid chromatography-tandem mass spectrometry for fatty acid ethyl esters in meconium: Assessment of prenatal exposure to alcohol in two European cohorts

Fatty acid ethyl esters (FAEEs) in meconium emerged as a reliable, direct biological marker for establishing fetal exposure to ethanol. An LC-MS/MS method for FAEES was developed using ethyl heptadecanoate as the internal standard. The analytes were extracted from meconium with hexane, followed by solid-phase extraction with aminopropyl-silica columns. Chromatography was performed on a C_8 reversed-phase column using water/isopropanol/acetonitrile (20:40:40, v/v/v) as a mobile phase. A triple quadrupole mass spectrometer that monitored the transitions in multiple reaction-monitoring mode was used for the detection of the analytes

Schulz K, Schlenz K, Malt S, Metasch R, Romhild W, Dressler J, Lachenmeier DW// Tech Univ Dresden, Inst Rechts Med, Fetscherstr 74, DE-01307 Dresden, Germany

J Chromatogr A 2008 **1211** (1-2) 113

Headspace solid-phase microextraction-gas chromatography-mass spectrometry for the quantitative determination of the characteristic flavouring agent eugenol in serum samples after enzymatic cleavage to validate post-offence alcohol drinking claims

A rapid headspace solid-phase microextraction-gas chromatography-mass spectrometry method has been developed for the determination of eugenol in serum samples after enzymatic cleavage. Eugenol is a characteristic marker for the consumption of certain alcoholic beverages including some digestif bitters and herbal liqueurs as well as wood-cask-aged spirits. Results confirm that eugenol undergoes a rapid phase II metabolism as it occurs completely conjugated as eugenol glucuronide in serum. Free eugenol was not detectable in any samples, which necessitated enzymatic cleavage with β-glucuronidase prior to HS-SPME sampling. These test results, in particular, confirm that the analysis of volatile compounds can be useful in forensic toxicology for the verification of post-offence alcohol consumption claims

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Forensic Sci Int 2008 **182** (1-3) 41

Influence of preservatives on the stability of ethyl glucuronide and ethyl sulphate in urine

Bacterial decomposition as well as in vitro and post-mortem formation of EtG has been reported. Glucuronidase-positive *Escherichia coli* were added to urine samples after sterile filtration. Preservatives tested were thymol, chlorhexidine, boric acid and the combination of chlorhexidine, ethylparabene and sodium propionate. EtG and EtS analyses were performed by liquid chromatography-electrospray ionization-mass spectrometry. Chlorhexidine on its own as well as in the above combination, and boric acid proved useful preservatives, while EtG degraded in samples doped with thymol. Addition of these preservatives did not interfere with the liquid chromatography-electrospray ionization-mass spectrometry analysis

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Talanta 2008 **77** (2) 494

Sequential injection-LOV format for peak height and kinetic measurement modes in the spectrophotometric enzymatic determination of ethanol: Application to different alcoholic beverages

Sequential injection-lab-on-valve (SI-LOV) format for the miniaturization of enzymatic assays, by using different measurement modes (peak height and initialrate-based measurement) and applied to several alcoholic beverages, including a certified sample material. The LOV system was developed for the enzymatic assay of ethanol in beverages, based on the conversion of ethanol to acetaldehyde by alcohol dehydrogenase, using spectrophotometric detection. The kinetic-based approach facilitates the applicability of the enzymatic determination to samples with intrinsic absorption, with a higher determination throughput

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J Anal Toxicol 2008 **32** (9) 778

Solid-phase extraction procedure for ethyl glucuronide in urine

A solid-phase extraction (SPE) procedure employing a HyperSep SAX strong anion exchanger was developed for sample cleanup of urinary EtG prior to LC-MS analysis. The EtG content in a 50-100-µl urine sample was finally reconstituted in the same volume as the original aliquot. The cleaner SPE extracts, without sample dilution, allowed for improved quantification of urinary EtG in the low concentration range

14 Tobacco

Fan Z, Xie FW, Xia QL, Wang S, Ding L, Liu HM*// *Zhengzhou Tobacco Res Inst CNTC, Chem Dept, CN-450001 Zhengzhou, Peoples Rep China

Chromatographia 2008 **68** (7-8) 623

Simultaneous determination of nicotine and its nine metabolites in human urine by LC-MS-MS

The direct determination of nicotine, cotinine, *trans*-3'-hydroxycotinine, their corresponding glucuronide conjugates as well as nornicotine, norcotinine,

cotinine-*N*-oxide and nicotine-*N'*-oxide was determined in the urine of smokers by LC-MS-MS. The assay only involves centrifugation and filtration of diluted urine. Analysis was performed on a C18 reversed-phase column using a gradient of ammonium acetate and methanol as the mobile phase. Nicotine-methyl-d_3, Cotinine-methyl-d_3 and *trans*-3'-hydroxycotinine-methyl-d_3 were used as internal standards. Recoveries for nicotine and nine nicotine metabolites ranged from 78.4 to 115.6%

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Clin Chem 2008 **54** (12) 2018

Meconium nicotine and metabolites by liquid chromatography-tandem mass spectrometry: Differentiation of passive and nonexposure and correlation with neonatal outcome measures

Concentrations of nicotine and 4 metabolites were measured with and without hydrolysis simultaneously in meconium from tobacco-exposed and nonexposed neonates by LC-MS. Unconjugated nicotine, cotinine, and OH-cotinine should be analyzed in meconium to detect *in utero* tobacco exposure because approximately 25% of positive specimens did not contain cotinine. Immunoassay monitoring of cotinine only would underestimate the prevalence of prenatal tobacco exposure

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J Sep Sci 2008 **31** (19) 3366

Application of comprehensive two-dimensional gas chromatography mass spectrometry and different types of data analysis for the investigation of cigarette particulate matter

Two-dimensional gas chromatography offers different ways for data analysis, namely, compound target analysis, automated peak-based compound classification and comprehensive pixel-based data analysis. Automated peak-based compound classification including mass spectrometric pattern recognition is used for the classification of tobacco particulate matter samples and the puff-dependent investigation of different compound classes. This compound group specific analysis is further reinforced by applying an even more comprehensive pixel-based analysis

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J Anal Toxicol 2008 **32** (8) 577

Complete automation of solid-phase extraction with subsequent liquid chromatography-tandem mass spectrometry for the quantification of benzoylecgonine, *m*-hydroxybenzoylecgonine, *p*-hydroxybenzoylecgonine, and norbenzoylecgonine in urine-application to a high-throughput urine analysis laboratory

A fully automated system utilizing a liquid handler and an online solid-phase extraction (SPE) device coupled with liquid chromatography-tandem mass spectrometry (LC-MS-MS) was designed to process, detect, and quantify benzoylecgonine (BZE), *meta*-hydroxybenzoylecgonine (*m*-OH BZE), *para*-hydroxybenzoylecgonine (*p*-OH BZE), and norbenzoylecgonine (nor-BZE) metabolites in human urine. No assay interference was noted from controls containing cocaine, cocaethylene, and ecgonine methyl ester. The automated specimen handling and SPE procedure, when compared to the traditional extraction schema, eliminates the human factors of specimen handling, processing, extraction, and derivatization, thereby reducing labor costs and rework resulting from batch handling issues, and may reduce the number of fume hoods required in the laboratory

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J Chromatogr A 2008 **1213** (2) 239

Development of solid-phase microextraction followed by gas chromatography-mass spectrometry for rapid analysis of volatile organic chemicals in mainstream cigarette smoke

In this work, a novel, simple and efficient method based on solid-phase microextraction followed by gas chromatography-mass spectrometry was developed to the analysis of VOCs in mainstream cigarette smoke (MCS). Using a simple home-made smoking machine device, extraction and concentration of VOCs in MCS were performed by solid-phase microextraction fiber, and the VOCs adsorbed on fiber were desorbed, and analyzed by gas chromatography-mass spectrometry

15 Homeland Security

Frawley DA, Samaan MN, Bull RL, Robertson JM, Mateczun AJ, Turnbull PCB*// *Arjemptur Technology Ltd, Science Park, Salisbury SP4 0JQ, England

J Forensic Sci 2008 **53** (5) 1102

Recovery efficiencies of anthrax spores and ricin from nonporous or nonabsorbent and porous or absorbent surfaces by a variety of sampling methods

The 2001 anthrax letter cases brought into focus the need to establish the most effective environmental sampling procedures. Results are presented from two studies aimed at establishing the best procedures for everyday surfaces likely to be contaminated after the release of environmentally stable bioaggressive agents, as exemplified by anthrax spores and ricin. With anthrax spores, contact plates, with mean retrieval rates of 28-54%, performed better than other methods by a wide margin for flat nonporous, nonabsorbent surfaces. They also proved best on flat porous, absorbent materials, although recoveries were low (<7%). For both agents, dry devices (swabs, wipes, Trace Evidence Collection Filters) had universally poor retrieval efficiencies with no significant differences between them. Among moistened devices (wipes, swabs, and Sample Collection and Recovery Devices), wipes were generally best, albeit with considerable cross-over among individual readings (highest mean recoveries for anthrax spores and ricin 5.5% and 2.5%, respectively, off plastic)

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Lab Chip 2008 **8** (11) 1793

Bead-based microfluidic toxin sensor integrating evaporative signal amplification

A microfluidic platform that incorporates substrate-laden silica beads for sensing the proteolytic activity of botulinum neurotoxin type A (BoNT/A) is described. The sensor employs toxin-mediated cleavage of a fluorophore-tagged peptide substrate specific for only BoNT/A. Peptide immobilized on beads is recognized and cleaved by the toxin, releasing fluorescent fragments into solution that can be concentrated at an isolated port *via* evaporation and detected using microscopy

Hagan NA, Cornish TJ, Pilato RS, Van Houten KA, Antoine MD, Lippa TP, Becknell AF, Demirev PA// Johns Hopkins Univ, Appl Phys Lab, 11100 Johns Hopkins Rd, Laurel, Md 20723, USA

Int J Mass Spectrom 2008 **278** (2-3) 158

Detection and identification of immobilized low-volatility organophosphates by desorption ionization mass spectrometry

Insecticides malathion and dicrotophos were selected as simulants of low vapor pressure chemical warfare agents which are inherently difficult to detect directly by traditional methods. Both liquid and powdered forms of either insecticide were readily detected by laser desorption/ionization mass spectrometry or desorption electrospray ionization mass spectrometry. Laser desorption/ionization mass spectrometry was performed on a miniaturized home-built time-of-flight (TOF) mass spectrometer and a commercial TOF/TOF instrument. For desorption electrospray ionization mass spectrometry, a home-built ion source was interfaced to a commercial quadrupole ion trap. In laser desorption/ionization, intact molecular ion signatures could be acquired by using an appropriate cationizing agent and powder additive in positive ion mode. MS^2 was used to confirm the identity of each analyte based on the observed characteristic fragmentation pattern. Effects of sample surface, salt additives, nanoparticle admixtures, and analyte solubility on the laser desorption/ionization and desorption electrospray ionization mass spectrometry sensitivity were also investigated

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Electroanalysis 2008 **20** (24) 2629

Application of impedimetric and voltammetric genosensor for detection of a biological warfare: Anthrax

The use of an electrochemical genosensing technology for the detection of anthrax is described. An alkanathiol-linked or unlabeled capture probe related to B. anthracis is immobilized onto gold or graphite electrode surface. A 101-mer anthrax target is used for hybridization. The extent of hybridization between probe and target sequences is determined by using differential pulse voltammetry (DPV) and electrochemical impedance spectrometry (EIS). EIS analysis are based on electron transfer resistance (Rct) in the presence of

$[Fe(CN)_6]^{3-/4-}$ and DPV measurements are based on transduction of both guanine oxidation and Meldola's blue (MDB) reduction signal as hybridization indicator. The response of the probe-modified electrodes which was interacted with a noncomplementary sequence was the same as the responses of probe-modified surface and proved the specifity of the hybridization with the target

Liu GD, Wang J, Barry R, Petersen C, Timchalk C, Gassman PL, Lin YH*// *Pacific NW Natl Lab, Richland, Wa 99352, USA

Chem Eur J 2008 **14** (32) 9951

Nanoparticle-based electrochemical immunosensor for the detection of phosphorylated acetylcholinesterase: An exposure biomarker of organophosphate pesticides and nerve agents

A nanoparticle-based electrochemical immunosensor has been developed for the detection of phosphorylated acetylcholinesterase (AChE), which is a potential biomarker of exposure to organophosphate (OP) pesticides and chemical warfare nerve agents. Zirconia nanoparticles (ZrO_2 NPs) were used as selective sorbents to capture the phosphorylated AChE adduct, and quantum dots (ZnS@CdS, QDs) were used as tags to label monoclonal anti-AChE antibody to quantify the immunorecognition events. Paraoxon was used as the model OP insecticide to prepare the phosphorylated AChE adducts to demonstrate proof of principle for the sensor. The phosphorylated AChE adduct was characterized by Fourier transform infrared spectroscopy (FTIR) and mass spectroscopy. The binding affinity of anti-AChE to the phosphorylated AChE was validated with an enzyme-linked immunosorbent assay. The parameters (e.g., amount of ZrO_2 NP, QD-anti-AChE concentration,) that govern the electrochemical response of immunosensors were optimized

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Lab Chip 2008 **8** (12) 2046

An integrated microfluidic platform for sensitive and rapid detection of biological toxins

A microfluidic chip-based immunoassay have been developed where the it is integrated with miniaturized electronics, optical elements, fluid-handling components, and data acquisition software to develop a portable, self-contained device for testing in case of an accidental or intentional exposure/intoxication to biotoxins. Preconcentration is enabled by photopolymerizing a thin, nanoporous membrane with a MW cut-off of approximately 10 kDa in the sample loading region of the chip

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Talanta 2008 **77** (1) 451

Improvement of acetylcholinesterase-based assay for organophosphates in way of identification by reactivators

Detection of organophosphates is frequently based on following acetylcholinesterase (AChE) inhibition. Although limit of detection and sensitivity for AChE-based assays seem to be intriguing, the identification of organophosphates is not currently efficient in this way. An improvement of AChE-based assay is described using reactivators with a selective return of AChE activity after previous inhibition. Four organophosphates: paraoxon-ethyl, paraoxon-methyl, trichlorfon, methamidophos were selected as representative pesticides and the three most available reactivators: HI-6, obidoxime, pralidoxime. Reactivation was accomplished in 96-well photometric microplates and activity of human recombinant AChE was followed by reaction of Ellman's reagent with one of enzyme digestion product: thiocholine

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J Chromatogr B 2008 **873** (1) 86

Chromatographic resolution, characterisation and quantification of VX enantiomers in hemolysed swine blood samples

VX (*O*-ethyl *S*-[2(diisopropylamino)ethyl] methylphosphonothioate) in blood samples was carried out with gas and liquid chromatography. The GC chiral stationary phase was HYDRODEX-β-TBDAc (β cyclodextrin). On the chiral HPLC phase CHIRALCEL OD-H, the enantiomers of VX were isolated with enantiomeric excess >99.99%. Quantitative chiral detection of VX the enantioresolution was realized on the HPLC chiral phase CHIRAL AGP. The limit of detection was 200 fg per enantiomer on column. The absolute recovery of the overall sample preparation procedure was 75%. After an intravenous and percutaneous administration of a supralethal dose of VX in anesthetised swine (+)-VX and (-)-VX could be quantified up to 720 min

Swaim LL, Johnson RC*, Zhou YT, Sandlin C, Barr JR// *Ctr Dis Control & Prevention, Natl Ctr Environm Hlth, Div Sci Lab, Atlanta, Ga 30341, USA

J Anal Toxicol 2008 **32** (9) 774

Quantification of organophosphorus nerve agent metabolites using a reduced-volume, high-throughput sample processing format and liquid chromatography-tandem mass spectrometry

A reduced-volume, high-throughput analytical method has been developed for the quantification of organophosphorus (OP) nerve agent metabolites in human urine. In human urine, metabolites of soman, sarin, cyclohexyl-sarin, VX, and Russian-VX were quantified down to a lowest reportable limit of 1 ng/ml. One hundred microliter urine samples were preconcentrated using normal-phase 96-well solid-phase extraction silica sorbent beds. Dual-column hydrophilic interaction liquid chromatography was applied in a 2.5-min isocratic separation followed by negative electrospray ionization-dilution multiple-reaction-monitoring MS

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Biotechniques 2008 **45** (5) 543

Development of isothermal TaqMan assays for detection of biothreat organisms

Rapid real-time isothermal assays for biodefense targets that include *Vibrio cholerae* and *Bacillus anthracis* have been developed. A novel isothermal real-time detection method (HDA/helicase-dependent amplification-TaqMan) has been developed that combines the advantages of both HDA and a TaqMan assay. In this assay, the reactions of DNA unwinding, primer annealing, polymerization, probe hybridization, and subsequent hydrolysis by the polymerase are coordinated and synchronized to perform at a single temperature

Uzawa H, Ohga K, Shinozaki Y, Ohsawa I, Nagatsuka T, Seto Y, Nishida Y// National Institute Advanced Industrial Science & Technology, Research Ctr Advanced Bionics, 1-1-1 Higashi, Tsukuba, Ibaraki 305 8565, Japan

Biosens Bioelectron 2008 **24** (4) 923

A novel sugar-probe biosensor for the deadly plant proteinous toxin, ricin

Highly toxic ricin in has potential in bioterrorism. A facile and sensitive detection method using synthetic analogues of β-lactosyl- and β-D-galactosyl ceramides as the ligands based on the fact that ricin binds cell-surface oligosaccharides. Sugar-probes having lipoic acids as anchor functions were synthesized via either a chemical or chemoenzymatic way and were immobilized on the sensor chips by a self-assembled monolayer technique. In addition, a visual monitoring method was developed, in which sugar-coated Au nanoparticles were utilized for discriminating ricin from other proteins in a facile manner, taking 10-30 min for judgment

Winemiller MD, Bae SY*// *US Army Edgewood Res, Res & Technol Directorate, Edgewood Chem Biol Ctr, Aberdeen Proving Ground, Md 21010, USA

J Chromatogr A 2008 **1210** (2) 185

Liquid chromatography-time-of-flight mass spectrometry analysis of 1-(2-chloroethoxy)-2-[(2-chloroethyl)thio] ethane and related compounds: Separation of an eleven component mixture

A method of separation for an eleven component mixture comprised of 1-(2-chloroethoxy)-2-[(2-chloroethyl)thio] ethane and its derivatives has been developed using LC-time-of-flight-MS. Substrate extraction and hydrolysis results where compared with sulfur mustard

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J Chromatogr A 2008 **1213** (1) 8

Selective preconcentration of chemical warfare agent degradation products using a zirconia preconcentration column

Zirconia has strong Lewis acid sites which have an affinity for the strongly electronegative phosphonate group of organo-phosphates. To investigate whether this affinity can be used for selective preconcentration, the retention of chemical warfare agent degradation products, methyl, ethyl, and propylphosphonic acid (MPA, EPA and PPA) and inorganic anion matrix components on Zirconia was investigated. Only organo-phosphates and sulfate exhibited retention on zirconia. After preconcentration detection limits for a 10 ml sample were 0.16, 0.19 and 0.16 μg/l for methyl, ethyl, and propylphosphonic acid, respectively

16 Workplace

Blackburn GR, Bleicher WT, Glidden S, Reinke G// PetroLabs Inc, 133 Industrial Dr, Ivyland, Pa 18974, USA

Polycycl Aromat Compound 2008 **28** (4-5) 533

Monitoring polycyclic aromatic compounds in environmental and workplace samples using conventional methods and the Ames mutagenicity assay of their nitrated derivatives

Techniques for workplace monitoring of airborne polycyclic aromatic compounds (PAC) levels in the hot-mix asphalt paving industry are described. Standard industrial hygiene methods and a newer, biologically based assay called the Nitration Assay to measure relative ambient levels of fumes and/or PACs in various paving workplace settings were employed. The latter assay was also used to test bitumen fumes generated in the laboratory by a new "microfuming" technique and to determine specific activities of the 16 PAHs designated by the US EPA as priority pollutants. The Nitration Assay makes use of two properties of 3-7-ring PACs: the ease with which they can be chemically nitrated and the high mutagenic potency of the nitrated products in the Ames Salmonella mutagenicity assay

Bobeldijk I, Karlsson D, Pronk A, Gonsalves J, Hekman M, Van de Lagemaat D, Preller L, Heederik D, Skarping G// TNO Qual Life Qual & Safety, Utrechtseweg 48, NL-3700 AJ Zeist, The Netherlands

Ann Occup Hyg 2008 **52** (8) 757

Validation of transferability of DBA derivatization and LC-MS/MS determination method for isocyanates *via* an interlaboratory comparison

A method for the quantitative determination of isocyanates (isophorone diisocyanate, isocyanic acid (ICA), methyl isocyanate, ethyl isocyanate, propyl isocyanate, hexamethylene diisocyanate (HDI), 2,6- and 2,4-toluene diisocyanate, 4,4'-methylene diphenyl diisocyanate (MDI), phenyl isocyanate (PhI), MDI oligomers and different HDI adducts) in air based on air sampling using an impinger flask containing di-*n*-butylamine (DBA) in toluene and a glass fibre filter in series. The DBA derivatives were analysed using liquid chromatography and tandem mass spectrometry. The method was applied in a large field study on exposure of workers in car repair shops and industrial painters

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J Chromatogr B 2008 **875** (2) 531

Development of a gas chromatography/mass spectrometry method to quantify several urinary monohydroxy metabolites of polycyclic aromatic hydrocarbons in occupationally exposed subjects

A method is described for the determination of 12 urinary monohydroxy metabolites of PAHs, namely 1-hydroxynaphthalene, 2-hydroxynaphthalene, 2-hydroxyfluorene, 9-hydroxyfluorene, 1-hydroxyphenanthrene, 2-hydroxyphenanthrene, 3-hydroxyphenanthrene, 4-hydroxyphenanthrene, 9-hydroxyphenanthrene, 1-hydroxypyrene, 6-hydroxychrysene, and 3-hydroxybenzo[*a*]pyrene. Analytes were determined in the presence of deuterated analogues as internal standards, by GC/MS operating in the electron impact mode. Urine samples from coke-oven workers shows that 1-hydroxynaphthalene and 2-hydroxyfluorene were the most abundant compounds (median 61.4 and 69.7 μg/l, respectively), while 6-hydroxychrysene, and 3-hydroxybenzo[*a*]pyrene were always below the quantification limit

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J Chromatogr B 2008 **875** (2) 419

Application of ion chromatography for the determination of inorganic ions, especially thiocyanates in human saliva samples as biomarkers of environmental tobacco smoke exposure

Environmental tobacco smoke is a major factor influencing the indoor air quality. Various toxic compounds emitted during tobacco smoking into the environment have a significant influence on the chemical composition of human biological fluids. The thiocyanate concentration in saliva is a biochemical measure, often used as an indicator of tobacco consumption. The goal of this study was to find significant relationships between salivary thiocyanates and other inorganic ions, which are constituents of natural saliva

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J Environ Monit 2008 **10** (11) 1297

A novel approach to evaluation of adsorbents for sampling indoor volatile organic compounds associated with symptom reports

A study was undertaken of chemicals which may contribute to 'sick building syndrome' (SBS) utilizing three adsorbents (Carbopack B, Chromosorb 106 and Tenax TA) for detecting differences in personal chemical exposure to volatile organic compounds in indoor air, between persons with and without SBS symptoms. The acquired samples were analysed by gas chromatography/mass spectrometry (GC/MS). Tenax TA gave the best partial least square discriminant analysis (PLS-DA) models for separating cases and controls, but a combination of measurements with Tenax TA and Carbopack B gave better PLS-DA models than models based on measurements from either adsorbent alone

Karnauhov YA, Kuz'mina NV, Hizbullin FF, Alekhina IE, Maistrenko VN// Res Inst Life Safety Republic Bashkortostan, ul 8 Marta 12/1, RU-450005 Ufa, Bashkortostan, Russia

J Anal Chem Engl Tr 2008 **63** (9) 867

Gas-chromatographic determination of alkylphenols in atmospheric air and the air of the working area

Industrial alkylphenols (agidols) in atmospheric and work area air are determined under optimized sampling conditions using solid-phase preconcentration on Amberlite KhAD-7 and absorption by toluene. The procedure employs GC for determination of alkylphenols

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J Environ Monit 2008 **10** (12) 1454

Production of test gases in the ppb range for round-robin tests and quality assurance measures during the measurement of VOCs

Quality assurance measures such as round-robin tests for the measurement of VOCs in indoor areas or at workplaces have not so far been available. A particular challenge is the production of test gases in the necessary concentrations. The BGIA test gas facility has been modified for the production of test gases in the $\mu g/m^3$ range. A two-stage primary gas purifier, a continuous test gas generator with multi-stage dilution, a capillary evaporator for low-volatility compounds and an online thermodesorber have been installed specifically for this purpose

Pacolay BD, Ham JE, Slaven JE, Wells JR*// *NIOSH, Exposure Assessment Branch, Hlth Effects Lab Div, 1095 Willowdale Rd, MS-3030, Morgantown, WV 26505, USA

J Environ Monit 2008 **10** (7) 853

Feasibility of detection and quantification of gas-phase carbonyls in indoor environments using PFBHA derivatization and solid-phase microextraction (SPME)

Solid-phase microextraction (SPME) was evaluated for the detection and quantification of the gas-phase carbonyls: citronellal, glyoxal, methylglyoxal, and β-ionone.

Schettgen T, Musiol A, Alt A, Ochsmann E, Kraus T// Univ Aachen, Rhein Westfal TH Aachen, Fac Med, Inst Occupational & Social Med, Paulwelsstr 30, DE-52074 Aachen, Germany

Anal Bioanal Chem 2008 **393** (3) 969

A method for the quantification of biomarkers of exposure to acrylonitrile and 1,3-butadiene in human urine by column-switching liquid chromatography-tandem mass spectrometry

Urinary mercapturic acids of 1,3-butadiene and acrylonitrile-*N*-acetyl-*S*-(3,4-dihydroxybutyl)cysteine (DHBMA) and MHBMA (an isomeric mixture of *N*-acetyl-*S*-((1-hydroxymethyl)-2-propenyl)cysteine and *N*-acetyl-*S*-((2-hydroxymethyl)-3-propenyl)cysteine) for the former and *N*-acetyl-*S*-2-cyanoethylcysteine (CEMA) for the latter) are specific biomarkers for the determination of individual internal exposure to these chemicals. A fast, specific, and very sensitive method for the simultaneous determination of DHBMA, MHBMA, and CEMA in human urine has been developed and validated using automated multidimensional LC/MS/MS which requires no additional sample preparation. Owing to its automation, our method is well suited for application in occupational or environmental studies

17 Product Authenticity

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J Pharm Biomed Anal 2008 **48** (3) 1011

Detecting counterfeit antimalarial tablets by near-infrared spectroscopy

NIRS was able to identify genuine or counterfeit tablets with high accuracy. Multivariate classification models indicated that this discriminatory ability was based, at least partly, on the presence or absence of spectral signatures related to artesunate. This technique can be field-portable and requires little training after calibrations are developed, thus showing great promise for rapid and accurate fake detection

Gaudiano MC, Di Maggio A, Antoniella E, Valvo L, Bertocchi P, Manna L, Bartolomei M, Alimonti S, Rodomonte AL// Ist Superiore Sanita, Dipt Farmaco, Viale Regina Elena 299, IT-00161 Rome, Italy
J Pharm Biomed Anal 2008 **48** (2) 303
An LC method for the simultaneous screening of some common counterfeit and sub-standard antibiotics. Validation and uncertainty estimation
Pharmaceutical counterfeiting is a worldwide public health problem and frequently under-recognised, particularly in developing countries where the percentage of counterfeit and sub-standard medicines is dramatically high. Antibiotics, among the most widespread drugs, have been particularly targeted by counterfeiters. WHO emphasizes the need for development and distribution of screening methods explicitly targeted to counterfeit drugs. In this paper is presented a single method for the simultaneous analysis of some of the most common and counterfeited essential antibiotics. Full validation was performed in terms of linearity, precision, robustness and trueness. A wide linearity range was investigated considering the specific nature of counterfeit and sub-standard drugs, whose content in active substance may be rather far from the declared amount. Robustness parameters were considered and a complete intermediate precision assessment was conducted, envisaging the possibility of transferring the method to quality control laboratories, hopefully in developing countries. The technique was successfully applied to the analysis of antibiotics purchased on the informal market in Chad, among which counterfeit and sub-standard samples were detected

18 Techniques

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Anal Sci 2008 **24** (11) 1409
Polymeric membrane sensors for the selective determination of dextromethorphan in pharmaceutical preparations
The construction and electrochemical response characteristics of poly(vinyl chloride) matrix ion-selective electrodes (ISEs) for dextromethorphan (DXM) hydrobromide are described. The membranes incorporate ion-association complexes of DXM with reineckate salt or phosphomolybdic acid, as electroactive materials and dioctylphthalate or dibutylsebacate as a plasticizing solvent mediator. The sensors exhibit very good selectivity for DXM over opiate alkaloids

Hawryl A, Cichocki D, Waksmundzka-Hajnos M*// *Med Univ, Fac Pharm, Dept Inorgan Chem, 6 Staszica, PL-20081 Lublin, Poland
J Planar Chromatogr Mod TLC 2008 **21** (5) 343
Determination of the lipophilicity of some psychotropic drugs by RP-TLC
RP-18-HPTLC plates with mobile phases containing water, an organic modifier (methanol, dioxane, acetone, acetonitrile or tetrahydrofuran), and ion-pair reagents or ammonia were employed to separate psychotropic drugs. R_F was measured for different concentrations of organic modifier. Relationships between solute retention and modifier concentration were described by the Soczewinski-Wachtmeister and Schoenmaker equations. Equations were employed to determine R_{MW} by the extrapolation method. Calculated values of R_{MW} or C were correlated with log P values for the drugs, estimated by use of software. Significant correlations between the intercept (R_{MW}) and slope (S) of the linear equations were also calculated. Chromatographic systems for which the R MW values obtained were close to calculated log P values can be used for determination of the lipophilicity of basic drugs

Langel K, Engblom C, Pehrsson A, Gunnar T, Ariniemi K, Lillsunde P// Natl Publ Hlth Inst, Drug Res Unit, Mannerheimintie 166, FI-00300 Helsinki, Finland
J Anal Toxicol 2008 **32** (6) 393
Drug testing in oral fluid - Evaluation of sample collection devices
Nine different oral fluid (OF) collection devices were studied to evaluate their suitability for collecting samples for drug analysis. The devices were Greiner Bio-One, Orasure Intercept, Immunalysis Quantisal, StatSure Saliva.Sampler, Cozart, Sarstedt Salivette, Malvern Medical OraCol, Acro Biotech Salicule, and Varian OraTube. For comparison, OF was also collected into plastic tubes. Drug recovery was studied by collecting OF fortified at 1000 ng/ml with amphetamine, 3,4-methylenedioxymethamphetamine, cocaine, Δ^9-tetrahydrocannabinol, morphine, codeine, diazepam, and alprazolam with the devices in vitro and analyzing the samples with GC-MS. Recovery of ethanol was measured OF by headspace GC-flame-ionization detection. The study shows that there are substantial differences between the OF collection devices on the market

Lin SC, Whang CW// Tunghai Univ, Dept Chem, Taichung 40704, Taiwan
J Sep Sci 2008 **31** (22) 3921
Capillary electrophoretic separation of tricyclic antidepressants using a polymer-coated capillary and -cyclodextrin as an electrolyte additive
A new method for the CE separation of nine tricyclic antidepressants (TCAs), viz. amitriptyline, clomipramine, desipramine, doxepin, fluphenazine, imipramine, nortriptyline, promazine, and thioridazine, is described. The capillary was statically coated with a layer of poly(*N,N*-dimethylacrylamide) (PDMA) to suppress the EOF, and beta-CD was used as an additive in the BGE solution

Marin SJ, Coles R, Merrell RCM, McMillin GA// ARUP Labs Inc, ARUP Inst Clin & Expt Pathol, 500 Chipeta Way, Salt Lake City, Ut 84108, USA
J Anal Toxicol 2008 **32** (7) 491
Quantitation of benzodiazepine in urine, serum, plasma, and meconium by LC-MS-MS
A single method for confirmation and quantitation of a panel of commonly prescribed benzodiazepines and metabolites, α-hydroxyalprazolam, α-hydroxyethylflurazepam, α-hydroxytriazolam, alprazolam, desalkylflurazepam, diazepam, lorazepam, midazolam, nordiazepam, oxazepam, temazepam, clonazepam, and 7-aminoclonazepam, was developed for three specimen types, urine, serum/plasma, and meconium was performed using LC-MS-MS on a Waters Alliance-Quattro Micro system. The instrument was operated in multiple reaction monitoring mode with an EI source in positive ionization mode

Sauvage FL, Gaulier JM, Lachatre G, Marquet P*// *Univ Hosp, Dept Pharmacol Toxicol, 2 Ave Martin Luther King, FR-87042 Limoges, France
Clin Chem 2008 **54** (9) 1519
Pitfalls and prevention strategies for liquid chromatography-tandem mass spectrometry in the selected reaction-monitoring mode for drug analysis
False-positive results with the use of liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) have been observed. Different LC-MS/MS techniques that use the selected reaction-monitoring mode, routinely employed for the analysis and quantification of drugs and toxic compounds in biological matrices, were employed when the false-positive and potentially false-positive results were observed. We sought to analyze the causes of and solutions to this problem. A previously reported LC-MS/MS general unknown screening method, as well as manual spectral investigation in 1 case, were utilized to perform verification and identification of interfering compounds. False-positive results included a metabolite of zolpidem that might have been mistaken for lysergic acid diethylamide, benzoylecgonine mistaken for atropine, and clomipramine and 3 phenothiazines that share several common ion transitions. It is recommended the use of stable-isotope internal standards whenever possible, relative retention times, 2 transitions or more per compound if possible, and acceptable relative abundance ratios between transitions

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Anal Chem 2008 **80** (18) 6870
Automation of solid-phase microextraction in high-throughput format and applications to drug analysis
The automation of SPME coupled to LC-MS/MS was accomplished using a 96 multiwell plate format, a SPME multifiber device, two orbital shakers, and a three-arm robotic system. Extensive optimization of the proposed setup was performed including coating selection, optimization of the fiber coating procedure, confirmation of uniform agitation in all wells, and the selection of the optimal calibration method. The system allows the use of pre-equilibrium extraction times with no deterioration in method precision due to reproducible timing of extraction and desorption steps and reproducible positioning of all fibers within the wells. The optimized multifiber SPME-LC-MS/MS was subsequently fully validated for the high-throughput analysis of diazepam, lorazepam, nordiazepam, and oxazepam in human whole blood