

SPME References 1999 – May 2000

Record - 1

TI- Analysis of organic micropollutants in drinking water using SPME and GC-MS.

AU- Guidotti, M;Ravaioli, G

JN- Annali di Chimica (Rome)

PY- 1999

VO- 89

NO- 11-12

PG- 919-924

AB- Drinking water was subjected to SPME according to the methods of Pawliszyn et al. (Anal. Chem., 1990, 62, 2145; Ibid, 1992, 64, 1187 and J. High Resol. Chromatogr., 1 993, 16, 689). The adsorbed compounds were thermally desorbed and the analytes were determined by GC-MS on a column (30 m * 0.25 mm i.d.) coated with HP5 MS (0.25 µm) or Innowax (0.5 µm). The operating parameters of the GC-MS instrument for each type of analyte (organochlorines, PAH, chlorophenols, phthalates and pesticides) are tabulated. The results are tabulated and chromatograms are presented. The method is suitable for routine analysis of micropollutants in drinking water due to its speed of analysis, high sensitivity (detection limits in the ng/l range) and high reproducibility.

Record - 2

TI- The use of solid phase microextraction (SPME) to monitor for major organoleptic compounds produced by chrysophytes in surface waters.

AU- Watson, SB;Brownlee, B;Satchwill, T;McCauley, E

JN- Water Science and Technology

PY- 1999

VO- 40

NO- 6

PG- 251-256

AB- Pond water or culture samples were spiked with naphthalene-d8 (internal standard) and salted to 10% NaCl before a 65 µm polydimethylsiloxane/divinylbenzene coated SPME fibre was introduced. After stirring, the SPME fibre was rinsed with H₂O, manually inserted into the split/splitless injection port of a GC system and desorbed at 225degC for 1 min. The resulting compounds were analysed on a HP-5ms fused-silica column (30 m * 0.25 mm; 0.25 µm film thickness) operated with temperature programming from 40degC (held for 2 min) to 240degC at 8degC/min, He as carrier gas (1 ml/min) and EIMS detection. A detection limit of 1-2 ppb was achieved for the target unsaturated aldehydes hexenal, hexadienal, heptenal, heptadienal,

nonenal, nonadienal, decenal and decadienal. The occurrence of these compounds in various samples is discussed.

Record - 3

TI- Solid-phase microextraction gas chromatography- mass spectrometric analysis of volatile organic compounds in water.

AU- Bocchini, P;Andalo, C;Bonfiglioli, D;Galletti, GC

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 21

PG- 2133-2139

AB- Volatile organic compounds (VOC) were extracted from water (40 ml) samples under stirring conditions, with no head space volume for 30 min using a SPME fibre comprised of a 75 µm film Carbon-polydimethylsiloxane stationary phase coated onto silica fibres. The SPME desorption time was 5 min and the SPME fibre was transferred to the injection port of a Varian 3400 GC held at 250degC. The VOC were desorbed directly onto fused-silica capillary column (30 m * 0.25 mm i.d.) coated with Vocol (1.50 µm) operated with temperature programming from 35degC to 195degC (held for 1 min) at 3degC/min. A splitless step of 2 min after injection was followed by the usual split ratio of 1:50 for the remainder of the run. Mass spectra were acquired in the m/z 40-400 range at a rate of 2 scans/s. Detection limits down to 0.05 ppb were obtained for some compounds. The method was applied to the analysis of drinking waters and industrial surface waters in northern Italy. Results (listed) were comparable to purge and trap values, with better sensitivity. The development of the technique for low cost, solvent free, portable extraction of VOC in waters is discussed.

Record - 4

TI- Use of solid-phase microextraction for the analysis of bisphenol A and bisphenol A diglycidyl ether in food simulants.

AU- Salafranca, J;Batlle, R;Nerin, C

JN- Journal of Chromatography, A

PY- 1999

VO- 864

NO- 1

PG- 137-144

AB- A new method has been developed to simultaneously analyse bisphenol A (BPA) and bisphenol A diglycidyl ether (BADGE) in aqueous based food simulants. The method consists of direct immersion solid-phase microextraction (SPME) of the analytes from the liquid matrix and subsequent chromatographic analysis by gas chromatography-mass

spectrometry. Using the proposed method, a whole analysis (including chromatographic step) can be completed in less than 40 min, with minimum sample handling. The SPME method shows good analytical performance for simultaneous BPA and BADGE analysis, except for BADGE determination in the aqueous alcohol (simulant C) solution. Detection limits ranging from 0.1 to 2.0 ng/g for BPA and from 13 to 15 ng/g from BADGE were obtained, with a linear range from the low-ng/g to several-mug/g range for BPA and from 0.1 mug/g to 40 mug/g for BADGE. A possible optimization method has been also developed and introduced.

Record - 5

TI- Determination of phenols in soils by in situ acetylation headspace solid-phase microextraction.

AU- Llompart, M;Blanco, B;Cela, R

JN- Journal of Microcolumn Separations

PY- 2000

VO- 12

NO- 1

PG- 25-32

AB- A headspace solid-phase microextraction (HSSPME) method for the determination of phenols in soils has been developed. The samples were suspended in water and phenols were derivatized in situ by adding potassium bicarbonate and acetic anhydride. Afterward, the sample was stirred and HSSPME was performed exposing the PDMS fibre with a 100 µm thickness to the headspace over the sample. Finally, the fibre was inserted in the GC injector port and GC-MSD analysis was carried out. Parameters affecting the extension of the adsorption process were studied (addition of water to the soil sample, sample size, salting out effect, volume of headspace). Also the extraction kinetics at 25 and 100degC were studied and compared. The proposed HSSPME method exhibits good performance in terms of precision, sensitivity, and linearity. Detection limits were in the sub-ng/g. This method has been applied to a real contaminated soil and the concentrations of phenols found with the proposed method were in good agreement with the certified phenol values.

Record - 6

TI- Analysis of organomercuric species in soils from orchards and wheat fields by capillary gas chromatography on-line coupled with atomic absorption spectrometry after in situ hydride generation and headspace solid phase microextraction.

AU- He, B;Jiang, GB

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 365

NO- 7

PG- 615-618

AB- A convenient procedure for the determination of organomercuric compounds in soils from orchards and wheat fields is described based on the aqueous derivatization of the polar organomercuric halides in 0.1M-acetic acid-sodium acetate buffer of pH 4 into their hydrides by addition of 1 ml of 6% KBH₄ with subsequent headspace SPME of the volatile derivatives. The volatile derivatives are separated by GC with a Supelco SPB-1 capillary column and on-line detected by electric heated quartz furnace AAS. The RSD for ten replicate measurements are 2.1%, 2.8% and 3.5% for methyl-, ethyl- and phenylmercury with absolute detection limits of 16 ng, 12 ng and 7 ng, respectively. This method is applied to the analysis of organomercuric compounds in soil samples and 0.04-0.64 µg/g of organomercuric species are detected in soils from different sites. The recoveries after standard addition are between 93-106%.

Record - 7

TI- Stir bar sorptive extraction (SBSE), a novel extraction technique for aqueous samples: theory and principles.

AU- Baltussen, E;Sandra, P;David, F;Cramers, C

JN- Journal of Microcolumn Separations

PY- 1999

VO- 11

NO- 10

PG- 737-747

AB- The theory, methodology and evaluation of a novel method for sample enrichment, involving the use of polydimethylsiloxane (PDMS) coated stir bars, termed stir bar sorptive extraction (SBSE) were described. Stir bars were prepared by coating stainless steel rods (1 or 4 cm * 0.8 mm o.d.) with PDMS tubing (containing 40% v/v fumed silica) to give effective sorbent volumes of 55 µl and 219 µl, respectively. The 1 cm stir bars were shown to be best suited to sample volumes of 10-50 ml whereas the 4 cm stir bars were suitable for sample volumes of up to 250 ml. Typical stirring times for equilibrium were 30-60 min. Prior to use the PDMS-coated stir bars were conditioned at 300degC for 4 hs. After sampling the analytes were desorbed at 250degC in a Gerstel TDS-2 thermodesorption system mounted on a HP 6980/5973 GC-MS instrument equipped with a CIS-4 PTV injector operated in the cryofocusing mode. Separation was on a fused-silica column (25 m * 0.25 mm i.d.) coated with HP SMS (0.25 µm) and operated with temperature programming from 50degC (held for 2 min) to 325degC at 15degC/min, with He as carrier gas and EIMS detection operated in selected-ion monitoring mode (m/z values tabulated). The new method was evaluated by the assay of 79 compounds, ranging in

volatility from 1,1,1-trichloroethane to chrysene, as trace pollutants in water. Recoveries were generally >60%. Detection limits were found to be in the low pg/ml level; two orders of magnitude better than the SPME technique.

Record - 8

TI- In-tube SPME/semi-micro LC analysis of tricyclic antidepressants.

AU- Saito, Y;Kawazoe, M;Jinno, K;Hayashida, M

JN- Chromatography

PY- 1999

VO- 20

NO- 4

PG- 344-345

AB- Urine was injected into the in-tube SPME set-up composed of extraction capillaries (20 cm * 0.25 mm i.d.) packed with DB-1 and DB-5 connected in series, each with a removable SUS304 stainless steel wire (20 cm * 0.2 o.d.) inside. Then the solvent for desorption was introduced from a syringe pump and microfeeder for determination of desipramine, nortriptyline, imipramine and amitriptyline by LC on a Capcell Pak C18 UG80 semi-micro column (15 cm * 1.5 mm i.d.). By employing the in-tube configuration for extraction and coupling with the semi-microcolumn LC, much less sample was required when compared to that for the conventional technique.

Record - 9

TI- Identification and characterization of Fenton oxidation products of surfactants by ionspray mass spectrometry and solid-phase microextraction gas chromatography mass spectrometry 1. Lauryl sulphate.

AU- Cuzzola, A;Raffaelli, A;Saba, A;Pucci, S;Salvadori, P

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 21

PG- 2140-2145

AB- Fenton oxidation of lauryl sulphate was carried out according to the previously reported procedure of Burzio and Gorini, (Italiana delle Sostanze Grasse, 1983, 60, 681). Polar oxidation products were analysed by LC-ionspray MS analysis following dilution (1:100) of the aqueous mixture, or as acetonitrile/5mMaqueous ammonium acetate (1:1) solutions of the ether residue. Analysis was carried out by flow injection using methanol/water (1:1) mobile phase at 50 μ l/min. Ionspray mass spectra were acquired using a triple quadrupole mass spectrometer equipped with an atmospheric pressure ionization source. Mass spectra were obtained in positive and negative ion modes

(details given). The volatile Fenton oxidation products (VFOP) were sampled with a polydimethylsiloxane-coated silica SPME fibre placed into the reaction headspace for the first 30 min of the oxidation reaction. The trapped VFOP were subsequently desorbed into the injection port of a Varian 1079 GC at 250degC using splitless mode for 3 min and analysed directly on a fused-silica column (30 m * 0.25 mm i.d.) coated with VA-5MS (0.25 μ m), operated with temperature programming from 70degC to 280degC (program details given), with He as carrier gas (1 ml/min) and both CI and EIMS detection operated in full-scan mode from m/z 50-500 (other operating parameters listed). The use of the techniques described to identify degradation intermediates in the environment is discussed.

Record - 10

TI- Effect of surfactant loading on the extraction properties of C-18 bonded silica used for solid-phase extraction of phenols.

AU- Shammala, FA

JN- Analytical Letters

PY- 1999

VO- 32

NO- 15

PG- 3083-3110

AB- An SPE system was modified with cationic surfactants and evaluated for extraction and preconcentration of trace phenolic compound contaminants in water at low ppb concentrations. Cationic surfactants such as cetyl trimethyl ammonium bromide (CTAB) are steadily adsorbed on the surface of C-18 bonded silica, and the ionized functional group of the surfactant can then act as an ion-exchange site to attract the ionized phenolic compounds from water samples. The method includes enrichment of the phenolic compounds by the surfactant-loaded SPE system, followed by elution of the analyte with methylene chloride and derivatization of the phenolic compounds with acetic anhydride. Thirty-two phenolic analytes were identified and quantitatively determined by this method: identification and quantification of the compounds was performed with GC/FID using 2-bromophenol as internal standard. SPME analysis with this method was linear over 3-6 orders of magnitude, with linear correlation coefficient (R^2) greater than 0.96. Experimentally determined FID detection limits ranged from ~ 30 ppt for methyl-substituted phenols to ~ 0.1 ppb for phenol and chloro-substituted phenols. The influence of sample pH, the loading amount of surfactant on the solid phase, and the volume and matrices of the sample were studied. Absolute recoveries from H₂O spiked with 0.2 ppb phenolic compounds were 96-103%. The method was applied to analyse of various natural waters, including ground water, lake water, seawater, and wastewater. Recoveries from ground water, lake water, seawater, and wastewater

were 92-106%, 75-93%, 87-103% and 86-99%, respectively. The new technique proved to be an excellent tool for trace enrichments of phenolic compounds at low ppb concentration of these analytes, from different natural water samples.

Record - 11

TI- Application of solid-phase microextraction to the profiling of an illicit drug: manufacturing impurities in illicit 4-methoxyamphetamine.

AU- Coumbaros, JC;Kirkbride, KP;Klass, G

JN- Journal of Forensic Sciences

PY- 1999

VO- 44

NO- 6

PG- 1237-1242

AB- Illicit tablets of 4-methoxyamphetamine (PMA) were crushed and placed in a glass GC autosampler phial capped with a Teflon-backed septum cap. The septum was pierced with the SPME needle, which was a manual fibre holder equipped with a fibre coated with an 85 µm thick polyacrylate solid phase. The fibre was exposed to the headspace at 25degC for 5, 10 or 30 min and adsorption was also performed for 5 min at 45 and 65degC. The organic compound impurities adsorbed on the SPME needle were desorbed at 290degC in the injection port of a GC directly onto a fused-silica column (15 m * 0.257 mm i.d.) coated with DB-1. The column was operated with temperature programming from 50degC (held for 2 min) to 300degC at 30degC/min, with He as carrier gas (62 cm/s) and EIMS detection. The response at 65degC for 5 min exceeded that at 25degC for 30 min. Precision data are not given. Results are presented and discussed. Impurities included 4-methoxyphenol, 4-methoxybenzaldehyde, 4-methoxyphenyl-2-propanone, 4-methoxyphenyl-2-propanol and 4-methoxyphenylpropene. The presence of the compounds suggested a route for synthesis of the drug. Results were also compared with those from liquid-liquid extraction.

Record - 12

TI- Field air analysis with SPME device.

AU- Koziel, J;Jia, MY;Khaled, A;Noah, J;Pawliszyn, J

JN- Analytica Chimica Acta

PY- 1999

VO- 400

PG- 153-162

AB- Solid-phase microextraction (SPME) devices were used for a wide scope of air-monitoring including field sampling and analysis of volatile organic compounds (VOC), formaldehyde, and particulate matter (PM) in air. Grab (instantaneous) and time-weighted average (TWA) sampling

were accomplished using exposed and retracted SPME fibers, respectively. Sampling time varied from 1 to 75 min, followed by analysis with a gas chromatograph (GC). A portable GC equipped with unique, in-series detectors: photoionization (PID), flame ionization (FID), and dry electrolytic conductivity (DELCD), provided almost real-time analysis and speciation for common VOC during an indoor air quality surveys. Indoor air samples collected with SPME devices were compared with those collected using conventional National Institute for Occupational Safety and Health (NIOSH) methods. Air concentrations measured with the SPME device were as low as 700 parts-per-trillion (ppt) for semi-volatile organic compounds. SPME methodology proved to be more sensitive than conventional methods, and provided a simple approach for fast, cost-effective sampling and analysis of common VOC in indoor air. SPME technology combined with fast portable GC reduced the sampling and analysis time to less than 15 min. The configuration offered the conveniences of immediate on-site monitoring and decision making, that are not possible with conventional methods. In addition, SPME fibers were applied to sampling of particulate matter in diesel engine exhaust. Linear uptake and particulate build-up on the fiber were observed. Preliminary research suggests that SPME fibers could also be applied to sampling of airborne particulate matter.

Record - 13

TI- Determination of benzene in aqueous samples by membrane inlet, solid phase microextraction and purge and trap extraction with isotope dilution gas chromatography-mass spectrometry.

AU- Creaser, CS;Weston, DJ;Wilkins, JPG;Yorke, CP;Irwin, J;Smith, B

JN- Analytical Communications

PY- 1999

VO- 36

NO- 11-12

PG- 383-386

AB- The determination of benzene in aqueous samples is reported using membrane inlet, solid phase microextraction and purge and trap extraction techniques combined with gas chromatography-mass spectrometry. The membrane inlet and solid phase microextraction techniques have been applied to the analysis of soft drink samples and the performance characteristics of these methods compared to the established purge and trap method. Isotope dilution quantitative procedures with hexadeuterobenzene as internal standard were used in combination with all three sampling inlets. Detection limits were at, or below the parts per billion (µg l⁻¹) level with analytical precision (%RSD) in the range 3-8%.

Record - 14

TI- Identification of organic tin compounds in contaminated lard by SPME-GC-MS.

AU- Wu, HQ;Zhang, GY;Zeng, L

JN- Fenxi Ceshi Xuebao

PY- 1999

VO- 18

NO- 6

PG- 67-69

AB- Lard (6 g) in a 10 ml headspace vial was sealed and heated in a 50degC water-bath for 30 min then an SPME needle was inserted for adsorption for 30 min. The needle was placed in the sample inlet for 2 min before GC on a flexible quartz column (30 m * 0.25 mm i.d.) coated with SPB-1 (0.25 mum), operated with temperature programming from 100-240degC (held for 15 min) at 10degC/min, with He as carrier gas (flow-rate not given), and 70 eV EIMS detection operated in full-scan mode from m/z 29-450. Six organic compounds were identified, viz. isooctyl 2-mercaptoacetate, diisooctyl 2,2'-thiobis(acetate), diisooctyl 2,2'-dithiobis(acetate), acetic acid, butanol and isooctanol as the starting materials, byproducts and hydrolytic products of the organotin compound which was postulated to be dibutyltin bis(2-isooctyl thioacetate).

Record - 15

TI- Development of in-tube solid-phase microextraction/liquid chromatography/electrospray ionization mass spectrometry for the analysis of mutagenic heterocyclic amines.

AU- Kataoka, H;Pawliszyn, J

JN- Chromatographia

PY- 1999

VO- 50

NO- 9-10

PG- 532-538

AB- In-tube solid-phase microextraction (in-tube SPME) coupled with liquid chromatography/electrospray ionization mass spectrometry (LC/ESI-MS) was developed for the analysis of mutagenic heterocyclic amines. LC/MS analyses of heterocyclic amines were initially performed by liquid injection onto a LC column. All heterocyclic amines tested in this study gave very simple ESI mass spectra and strong signals corresponding to $[M + H]^+$ were observed for all heterocyclic amines except for Glu-P-1 and Glu-P-2 ($[M + NH_4 - H_2O]^+$ ions were observed from these amines). All heterocyclic amines were well separated with a Supelcosil LC-CN column except for AalphaC and Glu-P-2 which consistently co-eluted. In order to optimize the extraction of heterocyclic amines, several in-tube SPME parameters were examined using five representative compounds. The optimum

extraction conditions were as follows: 10 aspirate/dispense steps of 30 μ l of sample in 100mM-Tris HCl (pH 8.5) at a flow rate of 100 μ l/min with Omegawax 250 capillary column. The heterocyclic amines extracted by the capillary column were easily desorbed by aspiration of 30 μ l methanol prior to injection, and carryover of heterocyclic amine was not observed. The calibration curves obtained from the ratios of heterocyclic amine area counts against that of 4,7,8-TriMeIQx as an internal standard were linear in the range of 5 to 200 ng/ml, with correlation coefficients above 0.9976 (n = 18). The detection limits (S/N = 3) were 0.2-3.1 ng/ml. This method was successfully applied to the analysis of food samples.

Record - 16

TI- Effective off-line analysis in the specialist chromatography laboratory.

AU- Spitzer, V

JN- GIT Labor-Fachzeitschrift

PY- 1999

VO- 43

NO- 11

PG- 1212-1214

AB- For a general purpose chromatographic laboratory, where a variety of matrices or concentration ranges are encountered, a highly automated system may be less attractive. Increased efficiency is then achieved by a combination of partial automation and method optimization. Some of these possibilities are reviewed. In SPE or liquid-liquid extraction, such methods include automated or accelerated Soxhlet extraction, SFE and microwave-assisted extraction. Among sample preparation methods are SPE or SPME and the elimination of sample preparation by direct injection HPLC on, e.g. LiChrospher ADS columns. In the measurement stage, automation or optimization may be achieved by direct measurement by FTNIR, increased selectivity by column or method coupling, high throughput and high-speed systems, high-performance TLC, SFC, capillary electrophoresis or electrochromatography and selective derivatization.

Record - 17

TI- Study into the equilibrium mechanism between water and poly(dimethylsiloxane) for very apolar solutes: adsorption or sorption?

AU- Baltussen, E;Sandra, P;David, F;Janssen, HG;Cramers, C

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 22

PG- 5213-5216

AB- Recently several publications appeared correlating octanol-water partitioning coefficients (K_{OW}) with solid-phase microextraction (SPME) extraction coefficients on poly(dimethylsiloxane) (PDMS) fibers. This correlation seems very good for medium-polar to polar compounds but cannot explain the observations for apolar compounds. It is shown that for polychlorinated biphenyls (PCBs) the published data are erroneous, because of system adsorption effects. PCB concentrations up to 10 times higher were measured on the stir bar compared to the SPME fiber. Using a short packed PDMS trap, it is shown that the true PDMS-water equilibrium constant is indeed proportional to literature K_{OW} data.

Record - 18

TI- SPME-HPLC analysis of Allium lacrymatory factor and thiosulfinates.

AU- Jaillais, B; Cadoux, F; Auger, J

JN- Talanta

PY- 1999

VO- 50

NO- 2

PG- 423-431

AB- Allium leaves (50 g) were rapidly cut into a flask which was fitted to another flask immersed in liquid N₂ and connected to a vacuum pump. Headspace volatiles and water were trapped during 20 min at room temperature then this frozen sample was allowed to warm until just thawing. Conditioned SPME fibers coated with polydimethylsiloxane (7, 30 or 100 μm film thickness), polyacrylate (85 μm) or Carbowax/templated resin (50 μm) were exposed to the thawed sample for 10 min then transferred onto a SPME-HPLC interface with a 1 min desorption time. A 20 μl portion of the frozen sample was analysed directly without concentration. Analysis was performed on a Adsorbosphere RP C18 column (25 cm * 4.6 mm) with aqueous 70% acetonitrile as mobile phase (0.8 ml/min) and detection at 250 nm. A linear calibration graph was obtained for both the direct injection and SPME method. However, only the Carbowax/templated resin (50 μm) showed good transfer of dimethylthiosulfinate.

Record - 19

TI- Total p-nitrophenol determination in urine samples of subjects exposed to parathion and methyl-parathion by SPME and GC-MS.

AU- Guidotti, M; Ravaioli, G; Vitali, M

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 11

PG- 628-630

AB- A method for the determination of total urinary p-nitrophenol which uses SPME and GC-MS is described. The effects of stirring, extraction time, pH and salt addition on SPME using a 85 µm polyacrylate fibre were investigated. GC was performed on a HP5MS column (25 m * 0.25 mm; film thickness = 0.25 µm) operated with temperature programming from 80degC (held for 2 min) to 280degC at 18degC/min, with He as carrier gas (1 ml/min) and MS detection operated in selected-ion monitoring mode at m/z 139 (quantitative) and, m/z 65 and 81 (qualitative). Calibration graphs were linear (r = 0.997) up to 2.5 mg/l. The method was suitable for monitoring subjects exposed to low levels of parathion or parathion-methyl. The method is less time-consuming than other GC methods (cf. Shealy et al., Environ. Int., 1996, 22, 661).

Record - 20

TI- Solid Phase Microextraction: A Practical Guide.

AU- ScheppersWercinski, SA

AB- US \$145.00; hardcover. This book covers: the theory of SPME; method development; SPME fibres and selection for specific applications; pharmaceutical applications; environmental applications; food and flavour applications; forensic and toxicology applications; and new developments in SPME.

Record - 21

TI- Rapid quantification of hexachlorobenzene in the color additives D&C Red Nos. 27 and 28 (phloxine B) using solid-phase microextraction and gas chromatography-mass spectrometry.

AU- Andrzejewski, D;Weisz, A

JN- Journal of Chromatography, A

PY- 1999

VO- 863

NO- 1

PG- 37-46

AB- The present paper describes the development of a method for the quantification of hexachlorobenzene (HCB) in the color additives D&C Red Nos. 27 (Colour Index, Solvent Red 48) and 28 (phloxine B; Colour Index, Acid Red 92) using solid-phase microextraction followed by gas chromatography-mass spectrometry (GC-MS) analysis. The method is simple and fast (1 h for each analysis), generates little solvent waste, and does not involve a solid matrix, thus permitting a more efficient extraction than does a previously developed Soxhlet extraction-GC MS method. Test portions from 30 batches of US-certified colour additives D&C Red Nos. 27 and 28 were analysed for HCB using the new method. Those batches represent domestic (five) and

foreign (one) manufacturers that requested certification for the colors during the past four years. All the samples contained HCB, ranging from 0.2 ppm to 244.3 ppm. The analyses revealed significant differences in the levels of HCB across batches from the same manufacturer as well as among different manufacturers. The range of HCB levels found in the analyzed batches (0.2-244.3 ppm) suggest that the contamination with HCB may be decreased by avoiding use of starting material (tetrachlorophthalic anhydride) heavily contaminated with HCB.

Record - 22

TI- Biosynthesis of menthofuran in *Mentha * piperita*; stereoselective and mechanistic studies.

AU- Fuchs, S;Zinn, S;Beck, T;Mosandl, A

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 10

PG- 4100-4105

AB- The biosynthesis of menthofuran (I) was studied by in vivo feeding experiments using [2H₂]- and [2H₂][18O]-labelled pulegone enantiomers as precursors. Shoot tips and first leave pairs were fed with an aqueous solution containing the respective precursor for 17-21 h in the dark. The essential oils evaporating from the glandular trichomes were concentrated by a headspace-sampling technique using a SPME fibre. The essential oils were analysed by enantiomer-selective multi-dimensional GC-MS. A fused-silica precolumn (30 m * 0.23 mm i.d.) coated with SE52 (0.23 µm) was used, operated with temperature programming from 50degC (held for 5 min) to 250degC (held for 49 min) at 5degC/min. Compounds eluting between 24.5-28.5 min were diverted onto the main analytical duran glass capillary column, (30 m * 0.23 mm i.d.) coated with coated with 30% octakis(2,3-di-O-butyl-6-O-t-butyl-dimethylsilyl)-gamma-cyclodextrin on OV-1701 (0.23 µm) operated with temperature programming from 40degC to 120degC (program details given) and 70 eV EIMS detection operated in selected-ion monitoring mode at m/z 108 for I. Labelled pulegone enantiomers were converted to the corresponding (R)- and (S)-I. The oxygen in I was introduced by enzymatic oxidation of pulegone.

Record - 23

TI- Determination of carphedon in human urine by solid-phase microextraction using capillary gas chromatography with nitrogen-phosphorus detection.

AU- Kim, S;Park, JH;Myung, SW;Lho, DS

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 11

PG- 1559-1562

AB- Urine (3 ml) was adjusted to pH 9.6 with carbonate buffer and mixed with 30% NaCl. A 65 µm Carbowax/divinylbenzene-coated SPME fibre was immersed in the resulting solution, with stirring, for 30 min at room temperature. The fibre was placed into the injection port of an HP 5890 series II GC at 250degC for 3 min and carphedon (I) was desorbed from the fibre directly onto the fused-silica analytical column (30 m * 0.25 mm i.d.), coated with DB-17 (0.25 µm) and operated with temperature programming from 100degC to 280degC (held for 8 min) at 25degC/min, with He as the carrier gas (0.9 ml/min) and N-P detection. Aniracetam (2 µg/ml) was used as an internal standard. The calibration graph was linear from 0.1- 10 µg/ml of I, with a detection limit of 0.01 µg/ml and the RSD (n not given) was 1.5-5.2% (recovery not stated).

Record - 24

TI- Monitoring toasting intensity of barrels by chromatographic analysis of volatile compounds from toasted oak wood.

AU- Chatonnet, P;Cutzach, I;Pons, M;Dubourdieu, D

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 10

PG- 4310-4318

AB- GC methods, the hydroalcoholic method and the headspace solid-phase microextraction (HSSPME) method, were developed for detecting the toasting intensity (light, medium or heavy) of oak barrels used for ageing fine wines and spirits. Wood shavings were taken from the inner walls of the barrels. The hydroalcoholic method was carried out by extracting wood shavings with aqueous ethanol containing tartaric acid. The extract was fractionated by liquid partitioning and the fractions were analysed by GC with FID. The HSSPME method was carried out by heating 1 g of dried and ground wood shavings in a sealed vial for 30 min at 80degC. The headspace gases were sampled using a microextraction fibre and analysed by GC. A Carbowax 20 M column was used with temperature programming up to 230degC and FID. The column was coupled to an MS detector for compound identification. The results produced by the two methods were processed by the multivariate techniques of principal component analysis and factorial discriminant analysis. Both analytical methods gave a clear differentiation between toasting levels. The HSSPME method was easier and quicker than the traditional liquid extraction method and provided similar information.

Record - 25

TI- Sample preparation for environmental analysis.

AU- LopezAvila, V

JN- Critical Reviews in Analytical Chemistry

PY- 1999

VO- 29

NO- 3

PG- 195-230

AB- A review is presented, of sample preparation in environmental analysis. Sample preparation continues to be an important step in environmental analysis and a lot of progress has been made in the last decade toward the development of faster, safer, and more environmentally friendly techniques for sample extraction and extract clean up before analysis. Especial emphasis is place on the state-of-the-art in sample preparation, particularly, SFE, microwave-assisted extraction, accelerated-solvent extraction for solid matrices, and both SPE and SPME for aqueous matrices. Driven by the need for faster, cheaper and more sensitive analytical methods, advances in sample preparation have included not only automation but developments in coupling sample preparation with instrumental analysis (e.g., SFE coupled with immunoassays). Examples are presented for extraction of PAH, organochlorine pesticides, PCB, phenols, triazine and chlorophenoxy-acid herbicides, and organotin and organomercury compounds from solid matrices with supercritical carbon dioxide and modified supercritical carbon dioxide as well as organic solvents under microwave irradiation. Emerging trends in sample preparation such as in situ derivatization/extraction of analytes from solid matrices and solventless extraction techniques such as SPME are also discussed. (108 references).

Record - 26

TI- Analysis of some pesticides in water samples using solid-phase microextraction-gas chromatography with different mass-spectrometric techniques.

AU- Natangelo, M;Tavazzi, S;Fanelli, R;Benfenati, E

JN- Journal of Chromatography, A

PY- 1999

VO- 859

NO- 2

PG- 193-201

AB- SPME-GC-MS methods were compared for the assay of acetochlor (I), fenoxycarb (II), myclobutanil (III) and propanil (IV) in H₂O at the ppb level. Spiked water samples (10 ml) were equilibrated with Supelco 65 µm carbowax-divinylbenzene SPME fibres at room

temperature. The extracted components were desorbed at 250degC and analysed directly by GC on a Supelco PTA5 column (30 m * 0.25 mm i.d.; film thickness = 0.5 mm) temperature programmed from 120-300degC or on a HP5-MS column (30 m * 0.25 mm i.d.; film thickness = 0.25 mm) programmed from 90-300degC, with He as carrier gas. Detection was by either EIMS on a HP 5971 quadrupole instrument operated in the selected-ion monitoring mode at m/z = 146, 162 for I, m/z = 88, 116 for II, m/z = 150, 179 for III and m/z = 161, 163 for IV or ion-trap EIMS on a Varian Saturn 2000 instrument operated in the MS-MS mode monitoring the following reactions: m/z 223 -> 146 for I, m/z 116 -> 88 for II, m/z 179 -> 125 for III and m/z 161-126. Calibration graphs for both detection limits were 2-30 pg/ml for MS detection and 2-15 pg/ml for MS-MS detection. The between-assay RSD (n = 3) within the calibration range were 3-5% for MS detection and 4-12% for MS-MS detection. The mean recoveries by SPME were 99% for I, 110% for II, 97% for III and 81% for IV.

Record - 27

TI- Solid-phase microextraction coupled with gas chromatography - ion-trap mass spectrometry for the analysis of haloacetic acids in water.

AU- Sarrion, MN;Santos, FJ;Galceran, MT

JN- Journal of Chromatography, A

PY- 1999

VO- 859

NO- 2

PG- 159-171

AB- Water (30 ml) was evaporated to sim400 mul at 50degC under vacuum and transferred to a 5 ml vial containing 0.1 g anhydrous Na₂SO₄, 30 mul concentrated H₂SO₄ and 40 mul ethanol. The mixture was vortexed and reacted at 50degC for 10 min. The resulting esters were sampled at 25degC for 10 min by headspace SPME using a 100 mum polydimethylsiloxane fibre. The analytes were desorbed at 250degC for analysis by GC on a fused-silica column (30 m * 0.25 mm i.d.) coated with DB-5MS (0.25 mum) and operated with temperature programming from 40degC (held for 1 min) to 60degC at 20degC/min, then to 120degC (held for 3 min) at 5degC/min and finally to 280degC (held for 10 min) at 25degC/min, with He as carrier gas (linear velocity 34 cm/s). Detection was by ion-trap EIMS operated in the mass range m/z 27-260. Quantitative analysis was based upon selected-ion monitoring at m/z = 77/94 for ethyl monochloroacetate (I), m/z 83/85 for ethyl dichloroacetate (II), m/z 117/82 for ethyl trichloroacetate (III), m/z 121/138 for ethyl monobromoacetate (IV), m/z 129/109 for ethyl bromochloroacetate (V) and m/z 174/120 for ethyl dibromoacetate (IV). External calibration and standard addition was used, the latter proved to be the more reliable. The linear dynamic ranges (ng/ml) were 1-125 (I), 0.5-125 (II), 0.5-125 (III), 0.85-120 (IV), 1-135 (V)

and 0.5-125 (IV); the corresponding limits of detection were 0.2, 0.04, 0.01, 0.1, 0.03 and 0.02 ng/ml, respectively. The within-day and between-day RSD (n = 5) for water spiked at the 5 or 10 ng/ml level were 6.3-7.9% and 7.9-10.3%, respectively.

Record - 28

TI- A simple method for the extraction of volatile organic compounds contained in air samples from adsorbent materials by solid-phase microextraction and their analysis by gas chromatography-mass spectrometry.

AU- Saba, A;Raffaelli, A;Pucci, S;Salvadori, P

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 19

PG- 1899-1902

AB- A common air pollutant, toluene, was used to develop a procedure for air analysis. The procedure involved SPME, followed by online GC-MS using a poly(diphenyl/dimethylsiloxane) column and a Varian Saturn 2000 ion trap mass spectrometer. The quantitation limit for toluene-d8 was 67 ng. The method was applied to an air sample from an industrial area; 34 pollutants were identified.

Record - 29

TI- Volatiles from unpasteurized and excessively heated orange juice analysed with solid-phase microextraction and GC-olfactometry.

AU- Bazemore, R;Goodner, K;Rouseff, R

JN- Journal of Food Science

PY- 1999

VO- 64

NO- 5

PG- 800-803

AB- Orange juice was either untreated or heated for 60 s at 96degC, then frozen at -20degC. A portion was thawed, further heat treated at 96degC for 180 s and refrozen. Juice (25 ml) was warmed to 40degC, equilibrated for 5 min and a 75 µm carboxen-poly(dimethylsiloxane) fibre was placed in the headspace for 30 min. Analysis of the desorbed volatiles was carried out using a column (30 m * 0.25 mm i.d.) coated with DB-5 and operated with temperature programming from 32degC (held for 3 min) to 200degC at 6degC/min, with He as carrier gas (29 cm/s) and FID. The outlet of the GC column was connected to a line of humidified, purified air flowing at 11 l/min and the aroma active components were evaluated by three trained panellists using the Osme aroma intensity scale. Some aroma peaks were common to all the juice headspace extracts, but others were only observed in the

unheated or heated extracts. The SPME fibre was more selective for terpenes than early eluting alcohols and aldehydes, compared with pentane-ether liquid-liquid extraction.

Record - 30

TI- Purge-and-trap ATR/IR spectroscopic method for the detection of semivolatile aromatic compounds in soils.

AU- Yang, J;Her, JW

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 20

PG- 4690-4696

AB- A rapid method for determination of semivolatile compounds in contaminated soil samples was developed by coupling solid-phase microextraction (SPME) with attenuated total reflectance (ATR) Fourier transform infrared (FT-IR) spectroscopy. A trapezoidal internal reflection element was mounted horizontally in a flow cell with the inlet port connected to a temperature-controlled glass extraction chamber. Soil samples were placed inside the glass tube and heated to the desired temperature. Vaporized semivolatile compounds were carried by a stream of nitrogen gas to the ATR/IR flow cell. To increase the trapping efficiency, the ATR crystal was coated with a hydrophobic polyisobutylene polymer that acted as the SPME phase. The method proved to be very sensitive in the detection of semivolatile compounds in soils. The relationship between various parameters affecting chemical quantitation, such as the film thickness, gas flow rate and water contents, was also studied. Three different compounds, 1-chloronaphthalene, nitrobenzene, and 2-chlorotoluene, were used to investigate the feasibility of this method in the analysis of organic compounds in sand and soil. Results indicated a linear relationship between concentration and IR signals can be obtained for the three analytes. The detection limit of this method was in the range of 200-300 ppb.

Record - 31

TI- An automatic analyzer for organic compounds in water based on solid-phase microextraction coupled to gas chromatography.

AU- Grote, C;Levsen, K;Wunsch, G

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 20

PG- 4513-4518

AB- An automated system for quasi-continuous and on-site monitoring of

organic compounds in water is presented. It is based on solid-phase microextraction (SPME) coupled to gas chromatography (GC) using a stopped-flow approach. The analytes are extracted either by direct or headspace SPME. All steps including sampling, sample preparation, extraction, chromatographic separation, and detection are fully automated. Using industrial wastewater as an example, the system was extensively validated both in the laboratory and in the field. Thus, relative standard deviations ranging from 1 to 7% were found for 24 compounds typically present in the wastewater of a chemical plant in Germany. Unattended operation of the system at a wastewater plant for more than one week is possible. Remote control via modem allows easy access to the system and the analytical data from any place.

Record - 32

TI- Automated in-tube solid-phase microextraction coupled with liquid chromatography-electrospray ionization mass spectrometry for the determination of beta-blockers and metabolites in urine and serum samples.

AU- Kataoka, H;Narimatsu, S;Lord, HL;Pawliszyn, J

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 19

PG- 4237-4244

AB- The technique of automated in-tube solid-phase microextraction (SPME) coupled with liquid chromatography-electrospray ionization mass spectrometry (LC-ESI MS) was evaluated for the determination of beta-blockers in urine and serum samples. In-tube SPME is an extraction technique for organic compounds in aqueous samples, in which analytes are extracted from the sample directly into an open tubular capillary by repeated draw/eject cycles of sample solution. LC-MS analyses of beta-blockers were initially performed by liquid injection onto a LC column. Nine beta-blockers tested in this study gave very simple ESI mass spectra, and strong signals corresponding to $[M + H]^+$ were observed for all beta-blockers. The beta-blockers were separated with a Hypersil BDS C18 column using acetonitrile/methanol/water/acetic acid (15:15:80:1) as mobile phase. To optimize the extraction of betablockers, several in-tube SPME parameters were examined. The optimum extraction conditions were 15 draw/eject cycles of 30 μ l of sample in 100 mM tris-HCl (pH 8.5) at a flow rate of 100 μ l/min using an Omegawax 250 capillary (Supelco, Bellefonte, PA). The beta-blockers extracted by the capillary were easily desorbed by mobile-phase flow, and carryover of beta-blockers was not observed. Using in-tube SPME-LC-ESI MS with selected ion monitoring, the calibration curves of beta-blockers were linear in the range from 2 to 100 ng/ml with correlation coefficients above 0.9982 ($n = 18$) and detection

limits (S/N = 3) of 0.1-1.2 ng/ml. This method was successfully applied to the analysis of biological samples without interference peaks. The recoveries of beta-blockers spiked into human urine and serum samples were above 84% and 71%, respectively. A serum sample from a patient administered propranolol was analysed using this method and both propranolol and its metabolites were detected.

Record - 33

TI- Analysis of methanol or formic acid in body fluids by headspace solid-phase microextraction and capillary gas chromatography.

AU- Lee, XP;Kumazawa, T;Kondo, K;Sato, K;Suzuki, O

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 734

NO- 1

PG- 155-162

AB- Methanol and its metabolite formic acid have been found extractable from human whole blood and urine by headspace solid-phase microextraction (SPME) with a Carboxen/polydimethylsiloxane fiber. The headspace SPME for formic acid was carried out after derivatization to methyl formate under acidic conditions. The determinations of both compounds were made by using acetonitrile as internal standard (IS) and capillary gas chromatography (GC) with flame ionization detection. The headspace SPME-GC gave sharp peaks for methanol, methyl formate and IS; and low background noises for whole blood and urine samples. Extraction efficiencies were 0.25-1.05% of methanol and 0.38-0.84% formic acid for whole blood and urine. The calibration curves for methanol and formic acid showed excellent linearity in the range of 1.56 to 800 and 1.56 to 500 mug/0.5 ml of whole blood or urine, respectively. The detection limits were 0.1-0.5 mug/0.5 ml for methanol and 0.6 mug/0.5 ml for formic acid for both body fluids. The within-day RSD in terms of extraction efficiency for both compounds in whole blood and urine samples were not greater than 9.8%. By using the established SPME method, methanol and formic acid were successfully separated and determined in rat blood after oral administration of methanol.

Record - 34

TI- Application of direct electrospray probe to analyse biological compounds and to couple to solid-phase microextraction to detect trace surfactants in aqueous solution.

AU- Kuo, CP;Shiea, JT

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 19

PG- 4413-4417

AB- Two novel direct electrospray probes (DEP) to generate an electrospray without using a capillary and/or syringe pump are presented. One of the DEPs is simply a copper coil connecting to a high-voltage power supply. The sample solution is deposited on the coil by a micropipette and the electrospray is subsequently generated at the tip of the copper coil after high voltage is applied to it. Another DEP is constructed by inserting two parallel optical fibres through the copper coil. The two fibres extend one end of the copper coil by 1 cm. Electrospray is generated at the tip of the fibers through the solution predeposited on the copper coil as the high voltage is applied on the copper coil. The ES mass spectra of myoglobin in liquid or solid phases can be obtained using this DEP-MS. Coupling the DEP to a solid-phase microextraction fibre is extremely easy, and a trace amount (in ppb range) of surfactants (Triton X-100) in the aqueous solution are selectively concentrated and detected.

Record - 35

TI- Application of solid-phase microextraction to analysis of organotin and organomercury compounds.

AU- Liu, JY;Jiang, GB

JN- Fenxi Huaxue

PY- 1999

VO- 27

NO- 10

PG- 1226-1130

AB- A review is presented of the use of SPME in analysing organotin and organomercury compounds including their speciation, particularly as an environmentally friendly procedure. A discussion is also given on factors influencing the extraction such as adsorbent selection, extraction time and ion strength, as well as the amount of derivatization reagent used and solvent acidity. (45 references).

Record - 36

TI- Determination of polychlorinated biphenyls in human blood serum by SPME.

AU- Poon, KF;Lam, PK;Lam, MHE

JN- Chemosphere

PY- 1999

VO- 39

NO- 6

PG- 905-912

AB- Plasma (4.3 ml) containing Aroclor 1254 was mixed with 0.2 ml

protease solution (50 mg Proteinase K in 50 ml H₂O containing 20mM-calcium chloride, 10mM-Tris HCl and 50% glycerol at pH 7.5) in a 10 ml vial. The vial was sealed and mixture was stirred for 30 min at 25degC. A SPME fibre with a 100 mum polydimethylsiloxane coating, pre-conditioned at 270degC under He overnight, was then immersed in the mixture for 60 min stirring continuously. After rinsing in 0.9% NaCl followed by H₂O, the SPME fibre was subjected to thermal desorption in the GC injector port under splitless mode at 270degC for 4.75 min. GC was performed on a 2 mum Ultra-2 column (25 m * 0.2 mm) operated with temperature programming from 80degC (held for 20 min) to 150degC at 5degC/min then to 250degC at 3degC/min and ECD. After each thermal desorption the SPME fibre was re-conditioned by keeping it in the GC injection port for 30 min at 270degC. The detection limit was 1 ppb (total PCB) with good linearity for 1-100 ppb.

Record - 37

TI- Determination of fungicides in wine by solid-phase microextraction (SPME).

AU- Friedrichs, K;Winkeler, HD

JN- GIT Labor-Fachzeitschrift

PY- 1999

VO- 19

NO- 2

PG- 69-70

AB- The fungicides iprodione, procymidone and vinclozolin (I, II and III, respectively) were determined in 1 ml wine by adding a corresponding amount of epsi-HCH as system standard. After heating for 2 h at 300degC, a 85 mum polyacrylate fibre was agitated for 120 min in the mixture. I, II and III were desorbed from the fibre during 3 min and determined on a column (30 m * 0.25 mm i.d.) coated with DB-5-MS (0.5 mum) and operated with temperature programming from 50degC (held for 1 min) to 150degC at 50degC/min and then to 280degC (held for 5 min) at 5degC/min, H₂ as carrier gas (54 cm/s) and ECD. The method was applied to white wines resulting in 1-80, 1-50 and 0.2-1 mug/l of I, II and III, respectively. The results agree with those obtained by liquid-liquid extraction and GC-MS. The advantages of SPME over other pre-treatment methods are discussed. Calibration was necessary after 10-15 analyses.

Record - 38

TI- Electrospray mass spectrometry of trimethyllead and triethyllead with in-tube solid phase microextraction sample introduction.

AU- Mester, Z;Pawliszyn, J

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 20

PG- 1999-2003

AB- The application of SPME-HPLC-electrospray mass spectrometry (SPME-HPLC-ESMS) to the analysis of organolead compounds in the aqueous environment is described. Stock organolead solutions were obtained by dissolving trimethyllead chloride and triethyllead chloride (10 mg each) in 10 ml methanol. Working standards were prepared from these stock standards. In-tube SPME was carried out using a Supel-Q-Plot gas chromatography column (60 cm * 0.25 mm i.d.) coated with coated with a porous divinylbenzene polymer with 0.1% TFA in aqueous 12% methanol as mobile phase (0.45 ml/min). The eluate was transferred to the source of the ES mass spectrometer operated in positive-ion mode with a capillary voltage of 2500 V and a nebulizer gas pressure at 40 psi. The drying gas flow was set to 10 l/min and the fragmentation voltage varied in order to investigate the effect of voltage on fragmentation spectra. Results indicated that a voltage of 150 V led to total dealkylation of the organolead compounds, while voltage in the region of 40 V resulted in an abundance of molecular ions. The application of the technique to chemical speciation and simultaneous monitoring of elemental and molecular forms of lead is discussed.

Record - 39

TI- Determination of oak lactones in barrel-aged wines and in oak extracts by stable isotope dilution analysis.

AU- Pollnitz, AP;Jones, GP;Sefton, MA

JN- Journal of Chromatography, A

PY- 1999

VO- 857

NO- 1-2

PG- 239-246

AB- Wine samples (5 ml), or extracts of wood shavings (100 g in 1 litre of simulated wine of pH 3.4 with 10% ethanol for one week) were treated with deuterium-4-labelled analogues of cis- and trans-5-butyl-4-methyl-4,5-dihydro-2(3H)-furanone (cis- and trans-I; oak lactones) and liquid-liquid extracted (LLE) with 2 ml ethyl ether/pentane (1:2). Portions (2 µl) of the extracts were analysed by GC, with the injector at 200degC and the splitter (30:1) opened after 36 s, on a fused-silica column (30 m * 0.25 mm i.d.) coated with DB-1701 (0.25 µm), operated with temperature programming from 50-250degC, (program details given), with He as carrier gas (0.72 ml/min) and 70 eV EIMS detection operated in selected-ion monitoring mode at m/z 114, 128 and 156. Calibration graphs were linear for 1-1000 µg/l of both cis- and trans-I in wine. Intra-assay RSD were 2.3-4.9 and 1-2.4%, respectively (n = 7), for 25 and 500 µg/l, of

both isomers in simulated white and red wines. The lactones could also be extracted by SPME on a 65 µm Carbowax-divinylbenzene fibre (experimental details given). The LLE method gave cleaner chromatograms, but with lower sensitivity.

Record - 40

TI- Headspace solid-phase microextraction for the determination of polychlorinated biphenyls in soils and sediments.

AU- Llompart, M;Li, K;Fingas, M

JN- Journal of Microcolumn Separations

PY- 1999

VO- 11

NO- 6

PG- 397-402

AB- Subsurface sandy loam soil was collected, dried at for 4 h at 100-105degC, crushed and screened to 300 µm. The soil (0.1-2 g) was mixed with 0.1-10 ml H₂O, the holding vial was sealed and placed in a bath at 100degC, and a 100 µm polydimethylsiloxane fibre was exposed to the headspace for 30 min. The analytes were desorbed from the fibre at 260deg and separated by GC on a HP-1 column (0.2 mm i.d.) operated with temperature programming from 120degC (held for 1 min) to 310degC (held for 10 min) at 10degC/min and MS detection. The calibration curve for PCB was linear and the within- and between-day RSD were 4.7% and 7.2%, respectively. The method was applied to sediments as well.

Record - 41

TI- Coupling device for desorption of drugs from solid-phase extraction-pipette tips and online gas-chromatographic analysis.

AU- vanHout, MWJ;deZeeuw, RA;deJong, GJ

JN- Journal of Chromatography, A

PY- 1999

VO- 858

NO- 1

PG- 117-122

AB- The micro-extraction of lidocaine (lignocaine; I) and diazepam (II) from aqueous solution was performed using C18 SPE discs mounted in SPEC.PLUS.PT pipette tips connected to a 10 ml Omnifix gas-tight syringe (details given). The SPE discs were activated with 200 µl methanol followed by 100 µl 0.1M-K₂HPO₄ of pH 8 prior to loading with 200 µl phosphate buffer spiked with I and II. The discs were washed with 100 µl H₂O and dried in a stream of air. The test compounds were eluted with 100 µl ethyl acetate and transferred directly to a gas chromatograph equipped with a PTV injector operated between 40-290degC at 5degC/s. Separation was performed on a fused-

silica column (30 m * 0.32 mm i.d.) coated with HP-5 (0.25 µm) operated with temperature programming from 40degC (held for 3 min) to 215degC at 20degC/min then to 230degC at 5degC/min and finally to 290degC (held for 10 min) at 25degC/min, with He as carrier gas (1.1 ml/min) and FID. The system was shown to be suitable for in-vial and online analyte desorption (details given). The performance of the system was illustrated by the assay of samples containing 1 µg/ml of I and II, respectively, with recoveries of sim75%.

Record - 42

TI- Determination of lead in blood and urine by SPME-GC.

AU- Yu, XM;Yuan, HD;Gorecki, T;Pawliszyn, J

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 15

PG- 2998-3002

AB- Lead is the most frequently quantitated toxic metal in biological matrixes. In this paper, a method is described for lead determination in whole blood and urine using solid-phase microextraction (SPME)-gas chromatography. Lead ion is first derivatized with sodium tetraethylborate to form tetraethyl-lead, which is then extracted from the headspace over the sample by SPME. The analytical procedure was optimized for coating selection, pH, extraction time, and effect of salt. The relative standard deviation was less than 10% for both urine and blood samples. The limit of detection was 3 and 4 ppb; the limit of quantification is 5 and 10 ppb for urine and blood samples, respectively. Good linearity was found for both urine and blood samples when PDMS coating was used. The standard addition method was used for quantitation. Certified urine and blood samples were analysed and good accuracy was obtained.

Record - 43

TI- Solid-phase microextraction of alkenylbenzenes and other flavour-related compounds from tobacco for analysis by selected-ion monitoring gas chromatography-mass spectrometry.

AU- Stanfill, SB;Ashley, DL

JN- Journal of Chromatography, A

PY- 1999

VO- 858

NO- 1

PG- 79-89

AB- Tobacco from a single cigarette was transferred to a 10 ml headspace SPME vial and 250 µl ethanol and 125 µl ethanolic 3',4'-methyleneedioxyacetophenone (internal standard) added. After mixing, 2

ml 3M-KCl was introduced via the vial septum and the mixture equilibrated at 95degC for 5 min. The headspace vapours were sampled using Supelco Carbowax divinylbenzene fibres. Analytes were desorbed at 230degC in the inlet port of the GC. Separation was performed on a 30 m DB-5MS column operated with temperature programming from 55degC (held for 1 min) to 110degC at 30degC/min, then to 155degC at 3degC/min and finally to 270degC at 30degC/min. He was used as carrier gas (1.2 ml/min). Detection was by EIMS operated in the selected-ion monitoring mode, characteristic m/z values are tabulated. Coumarin, piperonal, pulegone and nine alkenylbenzenes, including trans-anethole, methyleugenol, myristicin and safrole, were detected, identified and quantified in the concentration range from 1.8 ng to 43 mug/g. Calibration data, limits of detection and mean recoveries obtained using spiked tobacco samples are tabulated.

Record - 44

TI- Ion-exchange voltammetry as a solid-phase microextraction analytical method: factors influencing the mass transfer to perfluorosulfonated ionomer film-coated electrodes and some of their consequences on the current responses.

AU- Bagel, O;Degrand, C;Limoges, B

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 15

PG- 3192-3199

AB- A simple theoretical model of mass transfer kinetics at an electrode coated with an ion-exchange film polymer is proposed. The model takes into account the analyte depletion in solution and gives the relationship between the ion-exchange voltammetric (IEV) peak current and the initial analyte concentration in the sample matrix. The verification of the model is investigated at disposable Nafion film-coated screen-printed electrodes, using the redox cationic (ferrocenylmethyl)trimethylammonium salt. It is shown that the theoretical model and the experimental data fit satisfactorily insofar as the variation of the extraction and apparent diffusion coefficients of the salt with the film thickness are taken into account. Indeed, the film thickness plays a crucial role for the optimization of the IEV sensitivity, because the physicochemical properties of the recast Nafion polymer are dependent on the amount per unit area of Nafion deposited on the electrode surface.

Record - 45

TI- Biogenetic studies in *Mentha piperita*. 2. Stereoselectivity in the bioconversion of pulegone into menthone and isomenthone.

AU- Fuchs, S;Beck, T;Sandvoss, M;Mosandl, A

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 8

PG- 3058-3062

AB- *Mentha piperita* (peppermint) plants were fed with aqueous solutions of different deuterium-labelled pulegone and various enantiomeric distributions. The essential oil was extracted by SPME onto a fibre coated with 100 µm poly(dimethylsiloxane) using headspace sampling and analysed by enantioselective multidimensional GC-MS as previously described (Fuchs et al., J. Agric. Food Chem., 1999, 47, 3053). Results show that *Mentha piperita* was able to convert both enantiomers of deuterium-labelled precursor pulegone into menthone and isomenthone and a pathway is proposed.

Record - 46

TI- Alternative trihalomethane (THM) determination in swimming bath water.

AU- Masson, R;Speckle, W

JN- LaborPraxis

PY- 1999

VO- 23

NO- 9

PG- 42-44

AB- Trihalomethanes were extracted from a mixture of 1.2 ml sample and 1.3 ml H₂O containing sodium thiosulfate by SPME on a 75 µm Carboxen/PDMS fibre for 30 min with stirring. The THM were desorbed for 4 min at 30degC and determined by GC on a VOCOL column (10 m * 0.2 mm i.d.; film thickness 1.2 µm) with deactivated nonpolar tubing pre- and post-columns (i.d. 0.2 mm) operated with temperature programming from 30degC (held for 1.5 min) to 160degC (held for 4 min) at 20degC/min and FID (no carrier gas details given). Calibration graphs were linear for 2-300 µg/l of THM. The method was applied to water from an indoor swimming baths giving results which are lower than those obtained by headspace GC during which THM were formed by decomposition of their precursors. When the method was applied to fibres which had been stored at -4degC for 3 days after SPME the change in the CHBrCl₂ concentration was 6.5% of the original value.

Record - 47

TI- Development of a solid-phase microextraction GC-nitrogen-phosphorus detection procedure for the determination of free volatile amines in waste water and sewage-polluted waters.

AU- Abalos, M;Bayona, JM;Ventura, F

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 16

PG- 3531-3537

AB- Water containing C1-C6 alkylamines as their hydrochlorides was extracted by SPME using fibres with four types of coating. Optimum results were obtained with 100 µm poly(dimethylsiloxane) coatings, equilibration for 10 min and a desorption time of 3 min at 200degC. Headspace sampling was followed by GC on a PoraPLOT amine column (30 m * 0.32 mm i.d.) coated with Chrompack (10 µm) and operated with temperature programming from 60degC (held for 5 min) to 220degC (held for 15 min) at 10degC/min and N-P detection using cyclopropylamine as internal standard. The detection limits were 3-56 µg/l of volatile amine, which was close to or below the odour threshold concentration, and the calibration graph was linear in the range 50-600 ng/ml. The method was applied to the influent and effluent from a waste water treatment plant, and to sewage-polluted surface water. The RSD were 13-33% (n = 5).

Record - 48

TI- Coupling of biological sample handling and capillary electrophoresis.

AU- Veraart, JR;Lingeman, H;Brinkman, UAT

JN- Journal of Chromatography, A

PY- 1999

VO- 856

NO- 1-2

PG- 483-514

AB- A review is presented of the coupling of sample preparation techniques with capillary electrophoresis in the analysis of biological samples. Chromatography-based methods (online, offline, in-line and at-line SPE, SPME and LC), electrophoresis-based methods (isotachopheresis, sample stacking, field-amplified injection and electro-extraction) and membrane-based methods (dialysis, microdialysis, supported-liquid membranes and electro-dialysis) for sample preparation are discussed. The combination of automated sample preparation and capillary electrophoresis is considered and the potential of the coupled methods are discussed. (162 references).

Record - 49

TI- Monoterpene composition of essential oil from peppermint (*Mentha * piperita* L.) with regard to leaf position using solid-phase microextraction and gas chromatography-mass spectrometry analysis.

AU- Rohloff, J

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 9

PG- 3782-3786

AB- Portions (1 g) of leaf pairs and flowers from peppermint were sealed into 10 ml vials and maintained at 25degC for 24 h. A PDMS-coated solid-phase microextraction fibre was then inserted into each vial. After an adsorption period of 1 min, each fibre was transferred to the the injection port of a GC maintained at 250degC and the volatiles on the fibre were desorbed for 2 min. Volatiles were analysed directly onto on a fused-silica column (60 m * 0.25 mm i.d.) coated with Supelcowax (0.25 mum) operated with temperature programming from 35degC (held for 2 min) to 250degC (held for 10 min) at 5degC/min, with He carrier gas (100 ml/min) and 70 eV EIMS detection operated in full-scan mode from m/z 25-300. The essential oil compounds were identified from their mass spectrum and relative retention index. Nineteen essential oil compounds (listed) were detected: older parts of the plants contained higher concentrations of menthol, menthyl acetate and neomenthol than the younger plant parts whereas younger plant parts contained higher concentrations of menthone and isomenthone. Higher levels of menthofuran were found in the flowers than in the leaves. Results were compared with those obtained by a steam-distillation extraction (SDE) method. SPME sampling resulted in relatively higher amounts of high-volatile monoterpenes and lower detection of less volatile compounds (e.g., menthol and menthone).

Record - 50

TI- Determination of equilibrium constant of alkylbenzenes binding to bovine serum albumin by solid-phase microextraction.

AU- Yuan, H;Ranatunga, R;Carr, PW;Pawliszyn, J

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 10

PG- 1443-1448

AB- The use of SPME coupled with GC to study the binding properties between BSA and several alkylbenzenes in order to determine their equilibrium constants was investigated. SPME gave good results for the equilibrium constants, provided that the amount of analyte (alkylbenzene) partitioned into the headspace was negligible. In cases where this condition was not met, considerable errors were found in the equilibrium constant measurements. The means by which such errors may be avoided are discussed.

Record - 51

TI- Online combination of aqueous-sample preparation and capillary gas chromatography.

AU- Vreuls, JJ;Louter, AJH;Brinkman, UAT

JN- Journal of Chromatography, A

PY- 1999

VO- 856

NO- 1-2

PG- 279-314

AB- A review is presented of several methods for the online combination of aqueous-sample preparation and capillary GC. Online reversed-phase LC-GC is described with direct and indirect introduction of water to the system. The direct introduction of water is discussed in terms of micro-LC, special interfaces and special retention gaps or stationary phases. The indirect introduction of water is discussed in terms of reversed-phase LC-liquid liquid extraction-GC and reversed-phase LC-trapping column GC. Online analyte extraction-GC is described in terms of liquid-liquid extraction and SPE methods. SPE-thermal desorption GC, open-tubular trap GC and SPME-GC are also considered as online aqueous-sample preparation methods. (179 references).

Record - 52

TI- Sol-gel method for the preparation of solid-phase microextraction fibres.

AU- Zhou, ZP;Wang, ZY;Wu, CY;Zhan, W;Xu, Y

JN- Analytical Letters

PY- 1999

VO- 32

NO- 8

PG- 1675-1681

AB- A cleaned fused-silica fibre was dipped in a sol solution [consisting of 300 μ l methyltrimethoxysilane, 150 mg hydroxy-terminated poly(dimethylsiloxane), 30 mg poly(methylhydrosiloxane) and 200 μ l 95% TFA] for 30 min at 70degC. The coated fibre was then dipped for 1 min in trimethoxysilane/methanol (4:1) and conditioned at 310degC under N for 1-2 h. The performance of the resulting SPME fibre was compared with that of a commercial polyacrylate fibre (details given). The prepared SPME fibre exhibited faster mass transfer, higher thermal stability and more efficient extraction rate than the commercial fibre.

Record - 53

TI- Study of carbonaceous adsorbent for solid-phase microextraction and comparison with commercial device.

AU- Fang, RB;Zhang, WH;Wang, J;Zhang, KL;Nai, Z

JN- Sepu

PY- 1999

VO- 17

NO- 5

PG- 453-455

AB- The cited SPME device containing carbonaceous adsorbent was prepared.

Its properties are also presented, including shorter adsorption time (35 min only) and influence of ion strength (enhancement by 25% NaCl), and compared with those of commercially-available products. After physical and chemical treatments, its detection limits for e.g. agrochemicals are lower than those of polyacrylate and polydimethylsiloxane. The SPME is applicable in water analysis and determination of organic pesticides by GC.

Record - 54

TI- Rapid analysis of tetracycline antibiotics by combined solid-phase microextraction/high-performance liquid chromatography/mass spectrometry.

AU- Lock, CM;Chen, L;Volmer, DA

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 17

PG- 1744-1754

AB- Four SPME fibres were evaluated: 60µm

polydimethylsiloxane/divinylbenzene, 85µm polyacrylate, 50µm carbowax/templated resin and 65µm carbowax/divinylbenzene. Aqueous sample (3.5ml) was placed in a vial, heated to 65degC, saturated with potassium chloride and the SPME fibre immersed. The fibre was transferred to a desorption chamber containing acetonitrile/H₂O (3:17) and desorbed at 40degC for 5 min. The chamber was washed with mobile phase (details given). HPLC separation was effected on a 3 µm RP-18e PuroSphere column (5.0 cm * 4.0 mm i.d.) with gradient elution (1 ml/min) with acetonitrile containing 0.2% formic acid (mobile phase A) and H₂O containing 0.2% formic acid (mobile phase B) [program details given]. The mobile phase was introduced into the API 300 triple quadrupole instrument (Sciex, Concord, ON, Canada) for ionspray [MS detection (spray voltage 4.5 kV, ring voltage 300 V and orifice skimmer potential 25 V) with pure N₂ gas as nebuliser gas (1.0 l/min) and skimmer curtain (1.1 l/min) gas. Results indicate that the Carbowax/templated resin fibre SPME was most efficient for the extraction of tetracycline antibiotics from aqueous solutions.

Record - 55

TI- Determination of 1-naphthol in water by solid-phase microextraction-capillary gas chromatography.

AU- Miao, XS;Tian, H

JN- Fenxi Shiyanshi

PY- 1999

VO- 18

NO- 5

PG- 69-71

AB- Sample (4 ml), was saturated with NaCl and adjusted with to pH 2 with HCl and subjected to SPME with a polyacrylate fibre PA-85 membrane (85 µm thick) for 10 min. The fibre was placed in the injection port of a GC held at 220-310degC for 2 min. Desorbed 1-naphthol (I) was analysed directly on a quartz capillary column (30 m * 0.25 mm i.d.) coated with SE-54 (0.25 µm), operated with temperature programming from 40degC (held for 1 min) to 280degC (held for 5 min) at 10degC/min, with N₂ as carrier gas (26 cm/s) and FID. The detection limit for I was 0.5 µg/l. RSD were <4%.

Record - 56

TI- Solid-matrix fluorescence and phosphorescence and solid-phase microextraction of polycyclic aromatic hydrocarbons with hydrophobic paper.

AU- Ackerman, AH;Hurtubise, RJ

JN- Applied Spectroscopy

PY- 1999

VO- 53

NO- 7

PG- 770-775

AB- Standard solutions of polycyclic aromatic hydrocarbons (PAHs) in H₂O were analyzed by extracting 10 ml samples with a circle (6.4 mm diameter) of filter paper (Whatman 1PS) for 15 min with stirring. Prior to extraction, the filter paper was developed 4 times in distilled ethanol to remove impurities and then dried at 110degC for 30 min. After extraction, the exposed paper was again dried at 110degC for 30 min, before cooling to room temperature and placing in the sample compartment of the spectrofluorometer. Samples were then exposed to nitrogen gas for 15 min before measuring the solid-phase phosphorescence and fluorescence. Limits of detection for the PAHs phenanthrene, benzo[a]pyrene, benzo[e]pyrene and 1,2-benzanthracene ranged from 19 pg/ml to 1.9 ng/ml (fluorescence) and 6.2 pg/ml to 1.3 ng/ml (phosphorescence), although it was not possible to detect the phosphorescence of benzo[a]pyrene. Linear dynamic ranges for the same compounds were also determined. Two-, three- and four-component mixtures were studied. The identification of individual components was relatively easy when using both the fluorescence and phosphorescence data.

Record - 57

TI- Estimation of the octanol-water partition coefficients of PAH by solid-phase microextraction.

AU- Wang, YR;Wang, XR;Lee, FSC

JN- Sepu

PY- 1999

VO- 17

NO- 5

PG- 424-426

AB- Sample was stirred and SPME was performed with a fibre coated with 100 µm polydimethylsiloxane for 11 h and the adsorbed PAH were desorbed in the injection port of a GC at 270degC. The desorbed PAH were analysed on a fused-silica column (30 m * 0.25 mm i.d.) coated with DB-5 (0.25 µm), operated with temperature programming from 60degC (held for 3 min) to 120degC (held for 10 min) at 30degC/min, and then to 220degC at 4degC/min, and finally to 280degC (held for 10 min) at 30degC/min, and ion-trap MS detection. Eleven PAH (viz. naphthalene, biphenyl, 2,3-dimethylnaphthalene, 2,6-dimethylnaphthalene, fluorene, phenanthrene, anthracene, 9,10-dihydroanthracene, 1-methylphenanthrene, pyrene and fluoranthrene) were thus analysed and their partition coefficients were obtained at equilibrium. The linear free energy relationship between the polydimethylsiloxane-H₂O partition coefficient and octanol-H₂O partition coefficient was established by the extraction, with good correlation coefficient ($r = 0.9504$). Such linear equation is applicable to other PAH, positional isomers, and similar H acceptors. Results were compared with those obtained by the Leo method.

Record - 58

TI- Solid-phase micro-extraction gas chromatographic-mass spectrometric method for the determination of inhalation anaesthetics in urine.

AU- Poli, D;Bergamaschi, E;Manini, P;Andreoli, R;Mutti, A

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 732

NO- 1

PG- 115-125

AB- Samples (10 ml) were transferred to 20 ml phials containing 1 g NaCl and 0.2 ml 9N-H₂SO₄ and 20 µl CH₂Cl₂ (5.343 µg/ml, internal standard, IS). SPME fibres were introduced at 2 cm above the unstirred solution and immediately after the extraction period they were inserted into a GC injection port at 240degC for 16 min for desorption. The desorbed anaesthetics were separated on an RT-QPLOT column (30 m * 0.32 mm i.d.) coated with a porous divinylbenzene

polymer (Restek), operated with temperature programming from 40-180degC, (program details given) and EIMS detection operated in selected-ion monitoring mode at m/z 30, 84, 51 and 117, respectively, for N2O, IS, isoflurane and halothane. Two SPME fibres, one coated with divinylbenzene/Carboxen/polydimethylsiloxane and one with Carboxen/polydimethylsiloxane, were compared; they needed 20 and 15 min extraction times, respectively, but otherwise behaved similarly. Calibration graphs were linear from 0.37-145, 0.032-61.2 and 0.037-75.5 mug/l of N2O, isoflurane and halothane respectively, with detection limits of 15-30 ng/l for the halogen compounds and 75-100 ng/l for N2O. Absolute recoveries of nmol amounts at equilibrium were 22.3-82.2% for the halogen compounds, but only 0.1-0.3% for N2O.

Record - 59

TI- Automated in-tube solid-phase microextraction-liquid chromatography - electrospray-ionization mass spectrometry for the determination of ranitidine.

AU- Kataoka, H;Lord, HL;Pawliszyn, J

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 731

NO- 2

PG- 353-359

AB- Ground tablets were dissolved (2 mug/ml) in H2O, whereas urine was diluted tenfold with H2O. Either solution was filtered (0.45 mum), and a 0.1 ml portion was treated with 0.05 ml 0.5M-Tris hydrochloride buffer of pH 8.5, diluted to 1 ml with H2O and placed in the autosampler of an automated in-tube SPME system (cf., Eisert and Pawliszyn, Anal. Chem., 1997, 69, 3140; schematic shown) in which organic analytes are extracted from aqueous solutions directly on to a WCOT column from which they are desorbed for transport to the analytical column. A 30 mul portion of the solution containing ranitidine (I) was dispensed on to a capillary column (60 cm * 0.25 mm i.d.) coated with Omegawax 250 (0.25 mum) from which the ranitidine was desorbed with 40 mul methanol on to a 3 mum Supelcosil LC-CN analytical column (3.3 cm * 4.6 mm i.d.) with methanol/propan-2-ol/5M-ammonium acetate (50:50:1) as mobile phase (0.5 ml/min) and electrospray MS detection operated in selected-ion monitoring mode at m/z 270, 315 and 337. Calibration was effected with aqueous standard solutions; graphs of the sum of ion-peak areas were linear from 5-1000 ng/ml of I with a detection limit of 1 ng/ml (10 ng/ml in urine). Recoveries from tablets and urine were >92 and 58%, respectively.

Record - 60

TI- Fishing for a drug: solid-phase microextraction for the assay of clozapine in human plasma.

AU- Ulrich, S;Kruggel, S;Weigmann, H;Hiemke, C

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 731

NO- 2

PG- 231-240

AB- Plasma (0.25 ml) was treated with 50 µl 0.0008% loxapine (internal standard) solution, 1.7 ml H₂O and 0.5 ml 6% NaCl solution in 1M-NaOH with vortex-mixing for 10 s after each addition. A similar 1.5 ml portion of the mixture was placed in a 1.5 ml vial, a 0.1 mm poly(dimethylsiloxane) SPME fibre was mounted in the vial, and the vial was shaken for 30 min at 30°C. The fibre was subsequently washed with H₂O and H₂O/methanol (1:1), dried in air for 2 min, and inserted into a GC injection port at 260°C for desorption in the splitless mode for 1 min, after which the split was opened on to a fused-silica column (30 m * 0.53 mm i.d.) coated with BPX-5 (1 µm), operated with temperature programming from 160°C to 260°C at 40°C/min and then to 288°C at 4°C/min and N-P detection. Calibration graphs of peak-area ratios were linear for 100-1000 ng/ml of clozapine in aqueous standard solutions, and the detection limit was 30 ng/ml. The within-day RSD (n = 4) at 100, 200, 500 and 1000 ng/ml were 10.0, 7.9, 9.7 and 14.5%, and the between-day RSD (n = 8) at these concentrations were 22.6, 12.7, 7.9 and 10.6%, respectively. Results agreed with those obtained by GC with N-P detection after liquid-liquid extraction, and by HPLC.

Record - 61

TI- Solid-phase microextraction for the determination of pethidine and methadone in human urine using gas chromatography with nitrogen-phosphorus detection.

AU- Myung, SW;Kim, S;Park, JH;Kim, M;Lee, JC;Kim, TJ

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 9

PG- 1283-1286

AB- SPME of pethidine (I) and methadone (II) was performed by immersing a 100 µm polydimethylsiloxane fibre for 30 min in 3 ml urine, which had been adjusted to pH 11 and to which 15% NaCl had been added. Diphenylamine solution (15 µl) was used as an internal standard (100 µg/ml in methanol). Following extraction, I and II were thermally desorbed from the fibre at 240°C for quantification by GC, with temperature programming from 100°C (held for 0.5 min) to 300°C (held for 3 min) at 20°C/min, N₂ as the carrier gas (1 ml/min) and

N-P detection (column used not specified). The calibration graphs were linear for 1-12 mug/ml I and for 0.05-0.8 mug/ml II, the detection limit was <1 ng/ml for both drugs and the within-day RSD (n = 5) were <9%.

Record - 62

TI- Gas-chromatographic - mass-spectrometric analysis of dichlorobenzene isomers in human blood with headspace solid-phase microextraction.

AU- Liu, JT;Hara, K;Kashimura, S;Hamanaka, T;Tomojiri, S;Tanaka, K

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 731

NO- 2

PG- 217-221

AB- Blood (0.5 g) was mixed with 0.5 ml H₂O in a 12 ml phial and the phial was sealed. A 1 mul portion of 0.05% [2H10]p-xylene (internal standard, IS) solution in tetraoxyethylene glycol dimethyl ether (I) was injected into the mixture, which was heated at 30degC for 2 min. A 0.1 mm poly(dimethylsiloxane) SPME fibre was exposed to the headspace vapour at 30degC for 15 min and subsequently inserted for 3 min into a GC injection port at 250degC (He flow rate 2.1 ml/min) for splitless injection (1 min) on to a fused-silica column (30 m * 0.25 mm i.d.) coated with Restek XTI (0.25 mum), operated with temperature programming from 20degC (held for 1 min) to 290degC (held for 6 min) at 30degC/min and 70 eV MS detection operated in selected-ion monitoring mode at m/z 116 and 117, and 146, respectively, for IS and m-, p- and o-dichlorobenzene, which were all separated in the order cited. Calibration was effected by adding 0.01-2 mug of each analyte in I to 0.5 g of blood; the graphs were linear, and the limits of determination were all 0.02 mug/g. The within-day (n = 5) and day-to-day RSD (n = 4) at 1 mug/g were <=2.58 and 10.70% and at 10 mug/g were <=3.96 and 6.12%, respectively.

Record - 63

TI- Simple analysis of arylamide herbicides in serum using headspace-solid phase microextraction and GC-MS.

AU- Namera, A;Watanabe, T;Yashiki, M;Iwasaki, Y;Kojima, T

JN- Forensic Science International

PY- 1999

VO- 103

NO- 3

PG- 217-226

AB- Serum (200 mul), spiked with 10 mul propyzamide (0.1 mg/ml; internal standard, IS) was mixed with 500 mg NaCl and 800 mul H₂O and heated in a sealed phial with silicone septum and aluminium cap at 90degC.

The needle of an SPME device was inserted through the septum and the polydimethylsiloxane-coated silica fibre was exposed to the headspace. After 45 min, the fibre was withdrawn and inserted into a GC injection port (Shimadzu QP-500 GC-MS) at 250degC. The 3 herbicides, butachlor (I), diphenamide (diphenamid; II) and propanil (III) adsorbed on the fibre were desorbed onto a fused-silica column (30 m * 0.32 mm i.d.) coated with Supelco SPB-1 (0.25 µm). The column was temperature-programmed from 100degC (held for 3 min) to 250degC at 20degC/min, with He as carrier gas (0.8 ml/min) and EIMS detection operated in selected-ion monitoring mode at m/z 57, 160, 176 and 188; 72, 167 and 239 and 57, 161, 163 and 217, respectively, for I, II and III. Calibration graphs were linear from 0.25-10 µg/ml for each analytes with detection limits of 0.10, 0.05 and 0.25 µg/ml, respectively, for I, II and III. Intra- and inter-day RSD (n = 5) were 4.9, 5.6 and 6.0 and 5.2, 6.0 and 6.7%, respectively, for 1 µg/ml of I, II and III. Results (tabulated) from an attempted suicide case involving the herbicide Kusanon A (a mixture of III and carbaryl) showed that the initial concentration of III in serum was 26.7 µg/ml falling to 17.2 µg/ml after 6 h.

Record - 64

TI- Rapid analysis of parathion in biological samples using headspace solid-phase micro-extraction (HS-SPME) and gas chromatography/mass spectrometry (GC/MS).

AU- Musshoff, F; Junker, H; Madea, B

JN- Clinical Chemistry and Laboratory Medicine

PY- 1999

VO- 37

NO- 6

PG- 639-642

AB- Blood (0.5 g) was mixed with 10 µg malathion (internal standard), 0.5 g ammonium sulfate and 2 ml 0.1M-H₂SO₄ in a headspace vial which was sealed and heated at 90degC for 15 min. A SPME fibre coated with 100 µm polydimethylsiloxane was then exposed to the headspace for 15 min before being placed in the injection port of the GC system set at 250degC. The desorbed compounds were analysed on a HP-5MS column (30 m * 0.25 mm i.d.; 0.25 µm film thickness) operated with temperature programming from 80degC (held for 5 min) to 250degC (held for 5 min) at 10degC/min, He as carrier gas (0.8 ml/min) and MS detection. Linearity was established for 0.1-5 µg/g parathion-methyl and -ethyl with minimum detectable levels of 0.02-0.05 µg/g.

Record - 65

TI- Determination of polynuclear aromatic hydrocarbons in human blood serum by proteolytic digestion - direct immersion SPME.

AU- Poon, KF;Lam, PKS;Lam, MHW

JN- Analytica Chimica Acta

PY- 1999

VO- 396

NO- 2-3

PG- 303-308

AB- Blood serum samples were subjected to proteolytic digestion using a non-specific serine protease, Proteinase K, prior to the extraction of polynuclear aromatic hydrocarbons (PNAH) by SPME. The proteolytic digestion prevented lipoproteins from fouling the SPME fibre. A mixture of 3.8 ml serum and 0.4 ml protease solution was incubated at 25degC in a sealed vessel for 1 h. The optimum protease concentration was 190 mug/ml serum. A pre-conditioned SPME fibre (100 mum, polydimethylsiloxane-coated silica) was immersed in the reaction mixture for 60 min while stirring was maintained. The SPME fibre was then removed and rinsed with 0.9% NaCl. The adsorbed PAH were determined by GC following thermal desorption at 270degC for 5 min. The chromatography was carried out on a HP-5 column (0.33 mum, 50 m * 0.2 mm i.d.) with temperature programming from 40degC (held for 20 min) to 290degC and FID. The detection limits for 16 prioritized PNAH (tabulated) were 2.1-30.4 ppb. The recoveries of 88-177 ppb PNAH from spiked blood plasma samples were 81.8-98.5% and RSD were about 5.6%. Recoveries were 24.6-61.9% without proteolytic digestion.

Record - 66

TI- Development of a solid-phase microextraction method for detection of the use of banned azo dyes in coloured textiles and leather.

AU- Cioni, F;Bartolucci, G;Pieraccini, G;Meloni, S;Moneti, G

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 18

PG- 1833-1837

AB- Approximately 10 g of textile material was cut into 25-mm² pieces and thoroughly mixed. Then a 1-g sub-sample of material was treated with 17 ml of 0.06M-citrate buffer solution (pH 6; preheated to 70 +- 2degC). The reaction vessel was sealed and the mixture was vigorously shaken for 30 min. Then 3 ml of freshly-prepared sodium dithionite (200 mg/ml) was added and the vessel was re-sealed and shaken for a further 30 min at 70 +- 2degC, in order to reduce the extracted azo dyes to their constituent amines. The reaction vessel was finally cooled to room temperature by immersion in a bath of iced water. A 1-ml portion of the reaction mixture was mixed with 15 ml of internal standard solution (containing 30 mug/ml of 2,4,5-trichloroaniline for monocyclic amines, and 50 mug/ml of 2-methyl-1-naphthylamine for amines containing two aromatic rings) and a Carbowax-divinylbenzene

SPME fibre (65 µm film thickness) was inserted in the sample for 10 min with agitation of the solution. The fibre was then inserted in the injection port for 2 min and the adsorbed amines were desorbed and injected in split mode (with a split ratio of 1:10) at an injector temperature of 250degC. Analyses were performed on a Bio-Rad SUPEROX II column (30 m * 0.18 mm i.d.; 0.25 µm film thickness) with temperature programming from 65 to 270degC (held for 4.5 min) at 5degC/min, with He as carrier gas at a head pressure of 1.2 bar. The analytes were detected by 70 eV EI MS with scanning in the range m/z 50 to 450. Calibration graphs were linear for 0.75 to 12 µg/ml (corresponding to 15 to 240 µg/g of amine in the original textile material) for 18 aromatic amines investigated. At the 30 µg/g level (150 µg/g for 2,4-diaminotoluene or 2,4-diaminoanisole) the RSD were in the range 2.5 to 12.5% (n = 12). Recoveries of amines at the 30, 45, 60 and 90 µg/g levels were generally >85%.

Record - 67

TI- Development of online in-tube solid-phase microextraction LC-MS system.

AU- Kataoka, H;Narimatsu, S;Lord, HL;Pawliszyn, J

JN- Chromatography

PY- 1999

VO- 20

NO- 3

PG- 237-246

AB- A review is presented. The development of the system (including construction and instrumentation) is discussed. Its characteristics, the optimization of in-tube SPME, and its application in the determination of pharmaceuticals and environmental pollutants are described. (33 references).

Record - 68

TI- Simultaneous analysis of thiols, sulfides and disulfides in wine aroma by headspace solid-phase microextraction-gas chromatography.

AU- Mestres, M;Marti, MP;Busto, O;Guasch, J

JN- Journal of Chromatography, A

PY- 1999

VO- 849

NO- 1

PG- 293-297

AB- Thiols, sulfides and disulfides in wine volatiles were extracted by headspace-SPME using a Carboxen-polydimethylsiloxane fibre and cryogenic trap to focus the analytes. The fibre was inserted into the injection port of a GC at 300degC for 1 min, and the desorbed analytes were analysed on a fused-silica column (30 m * 0.32 mm i.d.)

coated with SPB-1 Sulfur (4 µm), operated with temperature programming from 35degC (held for 8 min) to 150degC at 15degC/min, then to 150degC at 40degC/min and to 250degC (held for 5 min), with He as carrier gas (1.2 ml/min) and flame photometric detection. Detection limits were in the range 0.05-3 µg/l for sulfides and disulfides and 0.5-1.0 µg/l for thiols.

Record - 69

TI- A systematic approach to optimize solid-phase microextraction.

Determination of pesticides in ethanol-water mixtures used as food simulants.

AU- Batlle, R;Sanchez, C;Nerin, C

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 13

PG- 2417-2422

AB- An optimization method based on a composite design using four quantitative and two qualitative experimental variables was applied to the SPME of organochlorine and organophosphorus pesticides in aqueous 15-95% ethanol. Matrix evaluation was carried out by comparing each variable with the mean of the whole set of experimental results. Separations were performed using poly(dimethylsiloxane)-, polyacrylate- and Carbowax/divinylbenzene-coated fibres, with analysis on a column (30 m * 0.25 mm i.d.) coated with HP-5MS (0.25 µm) and operated with temperature programming from 50degC (held for 1 min) to 215degC (held for 2 min) at 25degC/min, then to 236degC at 2degC/min, He as carrier gas (1.1 ml/min) and mass-selective detection. Detection limits were from 0.02-0.4 ng/g for H₂O to 38.7-205.5 ng/g for aqueous 95% ethanol food simulants. The RSD (n = 6) were <20%. The optimized SPME method was compared with liquid-liquid extraction. The effect of the ethanol matrix on the SPME performance is discussed.

Record - 70

TI- Heterogenic catalytic hydrolysis and analysis of natural pyrethrins in subcritical water coupled with solid-phase microextraction (SPME) and GC-MS.

AU- Krappe, M;Hawthorne, SB;Wenclawiak, BW

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 7

PG- 625-630

AB- The determination of natural pyrethrins as their hydrolysis product,

chrysanthemic acid, is described. The hydrolysis occurred during a water extraction process in a 64 * 7 mm i.d. steel tube in the presence of basic alumina. The SPME was performed by immersing the absorption fibre in a sample of the aqueous hydrolysate in a septum capped vial for the equilibration period. The fibre was then removed and placed into the split/splitless injection port of a Hewlett-Packard GC 5890 instrument. The GC operating conditions, using a 30 m Supelco capillary column are presented. The maximum, ramped oven temperature was 270degC with FID for the optimization process. Quantitation was performed with a Hewlett-Packard MSD 5972 in selected-ion monitoring mode set at m/z 123. The method was applied to some commercial products containing pyrethrum and the results were comparable to those obtained by SFC-FID and corresponded to the values stated by the manufacturer.

Record - 71

TI- Determination of VOC contamination in borehole sediments by headspace-SPME-GC analysis.

AU- Dermietzel, J;Streng, G

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 7

PG- 645-647

AB- Sediment (5-20 g) was shaken with 50-500 ml Ar purged H₂O for at least 24 h. After separation, an aqueous portion was transferred to a 15 ml headspace vial and sealed. After equilibration, VOC in the headspace vapour were absorbed onto a SPME fibre coated with 85 µm polyacrylate for 25 min. The VOC were analysed using a Varian 3400GC instrument, after desorption in the hot splitless GC injector at 250degC for 5 min. The capillary column was a HP5 MS, (25 m * 0.25 mm i.d.) using a maximum oven temperature of 190degC. The results of VOC concentrations in some borehole sediments are presented.

Record - 72

TI- Simultaneous filtration and liquid-chromatographic microextraction with subsequent GC-MS analysis to study absorption equilibria of pesticides in soil.

AU- Ramos, L;Vreuls, JJ;Brinkman, UAT;Sojo, LE

JN- Environmental Science and Technology

PY- 1999

VO- 33

NO- 18

PG- 3254-3259

AB- Pesticides ranging from relatively polar triazines to non-polar

compounds such as hexachlorobenzene or bromophos-ethyl in an organic soil were subjected to simultaneous filtration and LC microextraction (schematic given). The SPE cartridge was packed with 15-25 µm PLRP-5 conditioned with 5 ml each of methanol and H₂O. The microfilter used was a 5 µm stainless-steel screen or a 0.5 µm frit. After separation and preconcentration, the filter and SPE cartridge fractions were independently dried under N₂ and extracted with methyl acetate. Analysis was by GC on a column (30 m * 0.25 mm i.d.) coated with XTI-5 (0.25 µm) operated with temperature programming from 69degC (held for 3.5 min) to 280degC (held for 1 min) at 15degC/min, with He as carrier gas (97 kPa) and 70 eV EIMS detection. RSD were <6.2%. The soil-water partition coefficients were calculated and were well correlated ($r^2 = 0.973$) with published octanol-H₂O partition coefficients demonstrating the applicability of the method for adsorption equilibrium studies.

Record - 73

TI- Improved extraction of glycol ethers from water by solid-phase micro extraction by carboxen polydimethylsiloxane-coated fiber.

AU- Bensoam, J;Cicolella, A;Dujardin, R

JN- Chromatographia

PY- 1999

VO- 50

NO- 3-4

PG- 155-160

AB- Fifteen glycol ethers (GE; listed) in water were extracted by SPE on 75 µm Carboxen SPME fibre (specific surface area 1200m²/g) coated with polydimethylsiloxane. The fibre was placed in the injection port of a GC and the GE were desorbed by heating at 280degC for 15 min. The desorbed GE were analysed on a fused-silica column (25 m * 0.53 mm i.d.) coated with CPWAX 52CB, operated with temperature programming from 50degC (held for 10 min) to 250degC (held for 15 min) at 5degC/min, with He as carrier gas (4.5 ml/min) and FID. The calibration graphs were linear ($r^2 = 0.99$) over the range 0.1-10 mg/l for all GE in water with detection limits (signal-to-noise ratio = 3) of 50-500 µg/l. Repeatability RSD were <5%.

Record - 74

TI- Extraction and analysis of polycyclic aromatic hydrocarbons (PAH) by solid-phase micro-extraction-supercritical fluid chromatography (SPME-SFC).

AU- Lesellier, E

JN- Analisis

PY- 1999

VO- 27

NO- 4

PG- 363-368

AB- The SPME-SFC of the 16 EPA 610 PAH from water using the SPME-SFC interface of Boyd-Boland and Pawliszyn (J. Anal. Chem., 1996, 68, 1521) was studied. Good separation was achieved by immersing a 100 μm polydimethylsiloxane fibre for 45 min in aqueous sample solution stirred at 1000 rpm. The PAH were desorbed dynamically from the fibre and determined by SFE on 5 μm Kromasil C18 and Hypersil Green PAH columns (25 cm * 4.6 mm i.d.) at 32degC and outlet pressure 10 MPa with a gradient of 0-50% of acetonitrile in CO₂ over 25 min holding at 50% acetonitrile for 5 min as mobile phase (3 ml/min) and detection at 210 nm. Before SPME, the fibre was cleaned by the desorption procedure. Recoveries of 10 ppb to 0.2 ppm of the PAH added to water were up to 30% and decreased with increasing PAH mol. wt. RSD were 3-15% (n = 3). With detection at λ_{max} of each PAH the detection limit was 2 ppb. Recoveries of lower and higher PAH were increased and decreased, respectively, by the presence of NaCl. The effects of some other experimental conditions on the SPME of PAH were also studied.

Record - 75

TI- Investigation of membrane dryers and evaluation of a new ozone scrubbing material for the sampling of organosulfur compounds in air.

AU- HaberhauerTroyer, C;Rosenberg, E;Grasserbauer, M

JN- Journal of Chromatography, A

PY- 1999

VO- 852

NO- 2

PG- 589-595

AB- Gaseous mixtures of sulfur compounds were generated in air (schematic of apparatus shown), homogenized in a mixing chamber, pumped through a dryer and/or ozone scrubber at 100 ml/min, sampled by SPME (75 μm Carboxen-polydimethylsiloxane-coated fused-silica fibres) for 15 min, and analysed (100 s desorption) on a fused-silica column (60 m * 0.32 mm i.d.) coated with HP1 (1 μm), operated with temperature programming from -20degC (held for 2 min) to 120degC at 15degC/min, then to 180degC at 25degC/min, then to 280degC (held for 2 min) at 40degC/min, with He as carrier gas (2.5 ml/min) and AES detection. Nafion membrane dryers based on counter-current flow (27.2 cm * 2.5 mm i.d.) and desiccant drying (1 m * 1.1 mm i.d.; embedded in dry molecular-sieve), and a new ozone scrubbing material (70 or 140 mg polyphenylene sulfide wool) were investigated. Efficiency was highest for desiccant-based drying, but no analyte losses were observed with either dryer at r.h. \leq 50%. Losses (ppb levels) at higher r.h. depended on the condition of individual membranes. Using conditioning times of 5-10 min, the polyphenylene sulfide removed ozone

efficiently without analyte loss, and did not cause artefacts.
Recoveries of four organosulfur compounds at different conditions are given (table and graph).

Record - 76

TI- Solid-phase microextraction and gas chromatography-mass spectrometry for determination of monoaromatic hydrocarbons in blood and urine: application to people exposed to air pollutants.

AU- Andreoli, R;Manini, P;Bergamaschi, E;Brustolin, A;Mutti, A

JN- Chromatographia

PY- 1999

VO- 50

NO- 3-4

PG- 167-172

AB- Benzene, toluene, ethylbenzene and xylenes (BTEX; as test compounds) were extracted from 2 ml blood containing 20 µl heparin (or urine), spiked with 1 µl 13C6-benzene (20 mg/l in methanol; internal standard) by SPME on 75 µm Carboxen-PDMS fibres. The fibres were inserted into the injection port of a GC held at 280degC for 3 min and the desorbed BTEX compounds were separated on a fused-silica column (30 m * 0.25 mm i.d.) coated with HP-5MS (0.25 µm), operated with temperature programming from 45degC (held for 3 min) to 120degC (held for 1 min) at 5degC/min, with H₂ as carrier gas and EIMS detection operated in selected-ion monitoring mode (m/z listed for each analyte). Detection limits in blood and urine ranged from 5-10 ng/l and RSD were in the range 6.5-9.2%.

Record - 77

TI- Trends in solid-phase microextraction for determining organic pollutants in environmental samples.

AU- Penalver, A;Pocurull, E;Borrull, F;Marce, RM

JN- Trends in Analytical Chemistry

PY- 1999

VO- 18

NO- 8

PG- 557-568

AB- A review is presented of recent developments and applications of SPME in the determination of organic pollutants in environmental samples. Various matrices such as soils, water and air can be analysed successfully using SPME, both directly and with headspace extraction. SPME can be coupled easily to GC and, with the use of interfaces, to HPLC; full automation of these processes is now possible. New SPME fibres have been introduced which extend the range of application of SPME to other classes of analytes, such as inorganic compounds. (52 references).

Record - 78

TI- Optimization of the SPME device design for field applications.

AU- Mueller, L;Gorecki, T;Pawliszyn, J

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 7

PG- 610-616

AB- The design, construction and optimization of a SPME device is described. This is used for sampling VOC in the field where the analyte vapours are absorbed onto a porous polymeric coating on a fibre inserted into a modified micro syringe used for GC sampling. These are shown diagrammatically. Storage investigations on some prototype SPME were carried out at different temperatures and the results are presented. The VOC were determined with a Varian 3400 GC-FID system with a column (30 m *0.25 mm i.d.) coated with SPB-5 (1 µm). H₂ was used as the carrier gas at 30 psi with a ramped oven temperature from 40-100degC.

Record - 79

TI- Solid-phase microextraction/capillary gas chromatography for the profiling of confiscated ecstasy and amphetamine.

AU- Kongshaug, KE;PedersenBjergaard, S;Rasmussen, KE;Krogh, M

JN- Chromatographia

PY- 1999

VO- 50

NO- 3-4

PG- 247-252

AB- Impurities in ecstasy tablets and amphetamines dissolved in 0.1M-acetate buffer of pH 5 were extracted by SPME on polydimethylsiloxane-divinylbenzene fibers or by head-space SPME and analysed by GC on a fused silica column (30 m * 0.25 mm i.d.) coated with SPB-1 (0.25 µm), operated with temperature programming from 80degC (held for 3 min) to 150degC at 10degC/min and to 175degC at 2degC/min and to 300degC (held for 10 min) at 10degC/min, with He as carrier gas (1 ml/min for FID and NPD, and 0.7 ml/min for MSD), For ecstasy samples, and 60degC (held for 3 min) to 275degC (held for 2 min) at 10degC/min for amphetamines. Relative peak areas were repeatable with RSD of 2.2-12.6% and 2-10.9%, respectively, for ecstasy and amphetamines.

Record - 80

TI- Solid-phase microextraction for the assay of clozapine in human

plasma.

AU- Kruggel, S;Ulrich, S

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 7

PG- 654-655

AB- Plasma (0.25 ml) was vortexed with 1.7 ml H₂O, 0.5 ml 1M-NaOH containing 6% NaCl and 0.05 ml of 8 mg/l loxapine (internal standard). SPME was effected with a 0.1 mm-polydimethylsiloxane fibre for 30 min. After washing and drying, it was injected into a Hewlett 5890 series II GC equipped with a N-P detector and a split/splitless injection port. Separation was carried out on a BPX-5 capillary column at a N₂ carrier gas flow rate of 20 ml/min and a ramped oven temperature programme with a maximum temperature of 288degC. A linear calibration was obtained for clozapine up to 1000 ng/ml with a detection limit of 30 ng/ml. No interfering drug was found. The method was found to be suitable for the monitoring of clozapine in plasma.

Record - 81

TI- Identification of proteins in complexes by solid-phase microextraction/multistep elution/capillary electrophoresis-tandem mass spectrometry.

AU- Tong, W;Link, A;Eng, JK;Yates, JR_I

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 13

PG- 2270-2278

AB- Proteins in complex mixtures were loaded on a SPME cartridge containing 10-µm beads of Poros 10 R2 reversed-phase material, and the cartridge was connected to the capillary of a capillary electrophoresis (CE) apparatus. The system was flushed with sim50 µl 0.5% acetic acid and multistep gradient elution was performed with 10-80% methanol in 0.5% acetic acid. For each elution step, a CE-tandem MS run was performed with 2% methanol/0.5% acetic acid as running buffer and a two-step operating voltage in the range -25 to -8 kV. A sheathless electrospray interface was employed for CE-tandem MS, and sub-fmol detection limits were achieved by database searching. The method improved the concentration detection limit for CE and allowed more proteins in complex mixtures to be identified. In the analysis of a tryptic digest of yeast ribosomal complex, 66 of the 75 proteins were identified from 136 unique peptides.

Record - 82

TI- Analysis of volatile varnishes of coated wires by SPME.

AU- Hinz, DC;KwartengAcheampong, W;Wenclawiak, BW

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 7

PG- 641-642

AB- Wire (1 g) was heated in a headspace vial at 150degC for 1 h then an SPME fibre was immediately placed in the headspace zone for 30 s and then into the injector of a Hewlett-Packard HP6890 gas chromatograph, with a mass ion detector, at 250degC for 1 min. Analysis was performed on a column (25 m * 0.25 mm i.d.; 0.25 µm) operated up to 280degC (held for 11 min) with He as carrier gas (1 ml/min). The results were compared to those obtained with a conventional GC/headspace/FID system. The headspace/SPME-GC-MS method was more sensitive for the determination of the pyrolysis products evolved from the heated wire varnish.

Record - 83

TI- New gas chromatographic approach to polar compounds by derivatization/SPME.

AU- Takeuchi, M

JN- Bunseki

PY- 1999

NO- 7

PG- 595-597

AB- A review is presented on methods for derivatizing polar compounds and coupling these methods with SPME for GC analysis. This includes derivatization in the sample matrix, on SPME fibres, and in GC injection ports, e.g. in the analysis of air, water, tobacco, urine and serum. (9 references).

Record - 84

TI- Solid-phase microextraction turned inside out.

AU- Tan, D;Marriott, P;Lee, HK;Morrison, P

JN- Chemistry in Australia

PY- 1999

VO- 66

NO- 6

PG- 9-11

AB- SPME using a GC column (100 cm * 0.25 mm i.d.) packed with polydimethylsiloxane beads is illustrated by the measurement of partition coefficients by equilibrium extraction. Large volumes of aqueous standard solution were passed through the column and the

coefficients were calculated from the total amount of sorbed solute on the column phase once equilibrium had been reached. The sorbed solute was measured by re-extraction with organic solvent and the extract was analysed by GC. Equilibrium extraction occurred with as little as 200-1000 µl of aqueous solution for concentrations in the low mg/l range. The factors affecting partition coefficients (e.g. temperature, salt concentration and pH), and the application of in-tube SPME to trace analyses are discussed. Example chromatograms are presented.

Record - 85

TI- Headspace solid-phase microextraction for the analysis of dimethyl sulfide in beer.

AU- Scarlata, CJ;Ebeler, SE

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 7

PG- 2505-2508

AB- Beer (5 ml) was pipetted into vial and headspace sampling carried out by exposure of carboxen-polydimethylsiloxane SPME fibre into the headspace for 15 min. Dimethyl sulfide (I) was desorbed from the fibres for 3 min at 250degC in the injection port of a Hewlett-Packard 5890 Series II GC (Hewlett-Packard Inc., Avondale, PA, USA). I was analysed on a fused-silica column (30 m * 0.53 mm i.d.) coated with DB-1 (1 µm), operated with temperature programming from 40degC (held for 2 min) to 200degC (held for 4 min) at 20degC/min, with He as carrier gas (4 ml/min) and flame-photometric detection. The detection limit for the determination of dimethyl sulfide (I) by this method was 1 µg/ml. RSD (between assay) was 5%, between day was 3.9%. Results obtained by SPME were compared to those obtained by static headspace analysis and gave similar results (tabulated). The average recovery of I was >96%. Results for a range of commercial beers are presented.

Record - 86

TI- Examination of aroma production kinetics of different commercial wine yeasts in fermenting Muscat Ottonel wines with the help of SPME headspace sampling and fast GC analysis.

AU- Vas, G;Blehschmidt, I;Kovacs, T;Vekey, K

JN- Acta Alimentaria

PY- 1999

VO- 28

NO- 2

PG- 133-140

AB- A study of the aroma compounds formed in grape musts fermented with four different yeast varieties by SPME-GC is presented. Wines from the Muscat Ottonel grape variety (Eger region, Hungary) were fermented with 40 g/l of yeast at 12degC. Portions (2 ml) of each sample were placed in a sampling bottle and a 100 µm poly(dimethylsiloxane) coated fibre (Supelco. Inc., Bellefonte, PA, USA) was inserted into the headspace for 10 min. The fibre was removed and placed into the injector of a GC for 5 min at 250degC. The desorbed aroma compounds were transferred to the fused-silica analytical column (10 m * 0.1 mm i.d.) coated with CP-WAX 52CB (0.2 µm) with carrier gas. The column was temperature programmed from 30degC to 220degC (held for 1 min) at 10degC/min, with H₂ as carrier gas (45 cm/s) and FID. The concentrations of the dominating monoterpene alcohols (viz. linalool, terpineol, citronellol and geraniol), the fatty acid esters and the acetates of higher alcohols (considered to be the most important compounds in the aroma profile) were monitored over 400 h. Results (tabulated) showed that the aroma contents were practically unchanged until 48 h after fermentation commenced. The concentrations of acetate esters (listed) increased dramatically after this, particularly with the yeast varieties Uvaferm CM and 228. The CM and 228 yeasts contained the most aroma. Uvaferm ALB yeast and spontaneous fermentation with natural yeast flora gave 20-25 and 30-35% less, respectively, aroma compounds than CM and 228 varieties.

Record - 87

TI- Chemical characterization and screening of hydrocarbon pollution in industrial soils by headspace solid-phase microextraction.

AU- Havenga, WJ;Rohwer, ER

JN- Journal of Chromatography, A

PY- 1999

VO- 848

NO- 1-2

PG- 279-295

AB- Ground and dried soil (0.1 g) was introduced into a 1.8 ml vial. The vial was sealed and brought of thermal equilibrium for at least 2 h. A 100 µm polydimethylsiloxane micro-extraction fibre was then inserted into the headspace. After 40 min the micro-extraction fibre was removed and transferred to the GC-MS system. A fused silica DB5 MS column (30 m * 0.25 mm i.d., 0.25 µm film thickness) was used with He as carrier gas, an injection temperature of 280degC, column temperature programming from 60-130degC at 7degC/min, to 200degC at 5degC/min, to 260degC at 6degC/min and to 320degC (held for 4 min) at 20degC/min. The mass spectrometer was operated in the mass range 45-450 u with a scan rate of 0.81 s/scan. Linear calibration graphs were obtained for up to 17-1924 mg/kg PAH (naphthalene, 2-

methylnaphthalene, dibenzofuran, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene and pyrene). Precision was tested using the EPA-certified BNA contaminated soil, CRM-105-100. RSD (n = 6) were 2.78% (for naphthalene) to 8.4% (for phenanthrene). The experimental data was utilized for chemical characterization, source discrimination, the measurement of individual PAH distribution and calculation of weathering ratios.

Record - 88

TI- Determination of organophosphorus pesticides in water by solid-phase microextraction.

AU- Su, PG;Huang, SD

JN- Talanta

PY- 1999

VO- 49

NO- 2

PG- 393-402

AB- A 85 µm polyacrylate SPME fibre was immersed in the stirred sample (3 ml) contained in a 4 ml vial. After 30 min, the extracted organophosphorus pesticides (OPP) were thermally desorbed from the fibre at 290degC for 5 min and were analysed by GC on a fused-silica column (25 m * 0.2 mm i.d.), coated with HP Ultra 2 (0.33 µm), operated with temperature programming from 50degC (held for 5 min) to 180degC (held for 1 min) at 40degC/min, and to 220degC (held for 5 min) at 5degC/min, with N₂ as carrier gas (flow rate not given) and flame-photometric detection. The detection limits were 0.013-0.29 µg/l for all 5 OPP studied, except for mevinphos (420 µg/l). RSD (n = 7) were 2-9%. The method was applied to lake water.

Record - 89

TI- Determination of trichothecene (T2 mycotoxin) in aqueous sample with solid-phase microextraction technique followed by gas chromatography with flame ionization detection.

AU- Lee, PK;Kee, SYK;Ng, W;Gopalakrishnakone, P

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 7

PG- 424-426

AB- Water sample (2.5 ml) was saturated with NaCl and transferred to a 4 ml phial. An 85 µm polyacrylate SPME fibre was introduced into the liquid, which was stirred at 780 rpm for 75 min. The fibre was transferred to the injection port of a GC heated at 280degC for 8 min. Desorbed T-2 toxin (I) was desorbed directly onto the fused-silica analytical column (30 m * 0.25 mm i.d.) coated with DB5 (0.25

mum), operated with temperature programming from 70degC (held for 2 min) to 270degC (held for 18 min) at 25degC/min, with He as carrier gas (flow rate not stated) and FID. The calibration graph was linear from 10 (detection limit) to 1000 ng/ml of I. The RSD (n = 6) was 7.8% for 20 ng/ml of I. Recoveries of 20 and 100 ng/ml of I from sea water, reservoir water and drain water were in the range 93-108%. For low levels of I, the results were confirmed by GC-MS under similar conditions.

Record - 90

TI- Selective determination of selenite and selenate using SPME and GC-MS.

AU- Guidotti, M;Ravaioli, G;Vitali, M

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 7

PG- 414-416

AB- For determination of selenite, 5 ml samples were stirred at 1000 rpm with 0.15 ml 10% HCl and 0.2 ml 1% 4,5-dichloro-1,2-phenylenediamine in ethanolic 0.1M-HCl in a sealed phial for 30 min, to form its piaszelenole derivative. The SPME fibre (no details given) was introduced into the solution and stirring was continued for 35 min. The fibre was transferred into the injection port of a GC at 240degC for 3 min. The desorbed selenite derivative was analysed on a fused-silica column (30 m * 0.25 mm) coated with HP5-MS (0.25 mum), operated with temperature programming from 80degC (held for 2 min) to 280degC at 10degC/min, with He as carrier gas (1 ml/min) and EIMS detection operated in selected-ion monitoring mode at m/z 252. For total selenium (selenite + selenate), 10 ml sample were mixed with 12 ml 30% HCl in a 40 ml phial and heated at 600 W microwave power for 2 min. The solution was cooled. treated with 12 ml 30% NaOH and 5 ml portions were analysed as before. The calibration graph was linear from 0.05-100 mug/l of Se(IV), with a detection limit of 6 ng/l. RSD (n = 7) were 11.2, 9 and 6.6%, respectively, for 0.5, 5 and 50 mug/l of Se(IV). Average recoveries of 10 and 2 mug/l of Se(IV) from river and tap water, were 103 and 97%, respectively.

Record - 91

TI- Optimization of solid-phase microextraction conditions using a response surface methodology to determine organochlorine pesticides in water by gas chromatography and electron-capture detection.

AU- Aguilar, C;Penalver, A;Pocurull, E;Ferre, J;Borrull, F;Marce, RM

JN- Journal of Chromatography, A

PY- 1999

VO- 844

NO- 1-2

PG- 425-432

AB- The conditions for the SPME were optimized with the Doehlert design of the response surface method. Immersion SPME gave generally higher recoveries than the headspace method. Water was treated with 2 mug/l of endosulfan sulfate (internal standard) and 3 ml portions were transferred to 4 ml phials. The fibre, coated with 85 mum of poly(acrylate), was introduced into the sample and extraction was carried out at 60degC for 45 min, with magnetic stirring. The adsorbed compounds were desorbed for 5 min at 250degC in the injection port, which was fitted with a liner of 0.75 mm i.d. and were analysed on a column (30 m * 0.25 mm i.d.) coated with HP-1 (0.25 mum) with He (1.5 ml/min) as carrier gas, temperature programming (60-210degC, details given) and ⁶³Ni ECD. Thirteen organochlorine pesticides were separated in 32 min. Calibration graphs for most compounds were linear from 1 ng/l to 2.5 mug/l, with detection limits of 0.15-0.3 ng/l. Recoveries of 7 mug/l were 75.7-117.3%, except for heptachlor (53.6%) and p',p'-DDT (32.3%) both of which gave better recoveries by the headspace method. The RSD were 4-26% (n = 3). The method was applied to river and tap water.

Record - 92

TI- Analysis of organochlorine compounds in water by solid-phase microextraction and gas chromatography.

AU- Zhang, DN;Zhou, ZP;Tang, YZ;Wu, CY;Zhan, W;Xu, Y

JN- Fenxi Huaxue

PY- 1999

VO- 27

NO- 7

PG- 768-772

AB- In a 4-ml bottle, a 0.25 g portion of NaCl was mixed with the sample solution containing 100 mug/l each of twelve organochlorine pesticides (OCP, listed), closed with a stopper, analytes were preconcentrated ultrasonically for 7 min by head-space SPME using polydimethylsiloxane-coated silica fibres at 70degC. The SPME apparatus was inserted into a GC injector at 250degC and the desorbed OCP were analysed on a fused-silica column (30 cm * 0.25 mm i.d.) coated with HP-5 polyacrylate (0.25 mum), operated with temperature programming from 100degC to 190degC at 4degC/min and ECD. An external standard method was used to quantitation. Results were satisfactory.

Record - 93

TI- Evaluation of solid-phase microextraction for sampling of volatile organic sulfur compounds in air for subsequent gas-chromatographic

analysis with atomic-emission detection.

AU- HaberhauerTroyer, C;Rosenberg, E;Grasserbauer, M

JN- Journal of Chromatography, A

PY- 1999

VO- 848

NO- 1-2

PG- 305-315

AB- Sulfur VOC were determined in spiked air samples by SPME coupled with GC-AED. The spiked air samples were generated by means of a permeation apparatus and transferred to the sampling chamber (500 ml volume). SPME was carried out manually using 100 µl polydimethylsiloxane (PDMS) or 75 µm Carboxen-PDMS-coated fused-silica fibres. The loaded fibres were transferred to the injection port of the gas chromatograph. Desorption was carried out at 250degC (for carboxen-PDMS fibres) or 150degC (for PDMS fibres) for at least 100 s. The analysis was performed using a HP column (60 m * 0.32 mm i.d., 1 µm film thickness) with He carrier gas (2.5 ml/min), temperature programming from -20degC to 280degC and detection at 181 and 193 nm for S and C, respectively. When compared to the PDMS fibre, the Carboxen-PDMS fibre gave improved sensitivity and repeatability. The detection limits with the Carboxen-PDMS fibre were 0.04-0.06 ppb methanethiol, 0.003-0.004 ppb dimethyl sulfide, 0.005-0.007 ppb isopropanethiol and 0.003-0.004 ppb isobutylthiol. Sensitivity decreased with analyte volatility. The proposed method was limited to quantitative on-site analysis due to (i) the low storage stability of the loaded fibres, (ii) the dependence of extraction efficiencies on relative humidity and (iii) the pronounced difference in sensitivity between fibres.

Record - 94

TI- Artificial receptor-facilitated solid-phase microextraction of barbiturates.

AU- Li, S;Sun, LF;Chung, Y;Weber, SG

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 11

PG- 2146-2151

AB- Barbiturates were extracted from urine or serum by SPME using receptor-doped films of PVC. The receptor NN'-bis-[6-(2-ethylhexanoylamino)-pyridin-2-yl]-isophthalamide (synthesis described) was designed to dissolve in the PVC plasticizer, and extractions were performed as a function of receptor concentration. A stainless-steel rod was dip-coated with primer and the PVC solution to form membranes, and SPME was carried out by immersing the rod for 5 min in standard solution or sample containing the standard. Back-

extraction was performed for 15 min with 20mM-phosphate buffer of pH 11.5 and the extract was analysed by capillary electrophoresis. Receptor concentration affected the extraction yield of compounds that bound to the receptor but had negligible effect on weakly binding molecules, indicating that it did not act as a cosolvent. In addition, NMR data showed that the receptor self-associates. Signal-to-noise ratios were improved when the receptor-doped membranes were used for SPME of barbiturates, compared with extractions using a phosphate ester solvent.

Record - 95

TI- Total 4-chlorophenol determination in urine samples of subjects exposed to chlorobenzene, using SPME and GC-MS.

AU- Guidotti, M;Ravaioli, G;Vitali, M

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 7

PG- 427-428

AB- Samples (2 ml) were heated with 1 ml 37% HCl at 50degC for 1 h.

Portions (0.1 ml) of hydrolysates were mixed with 9 ml H₂O and 2.7 g NaCl in 10 ml phials. An 85 µm polyacrylate SPME fibre was introduced into the headspace above the solution, which was stirred at 1000 rpm for 30 min. The fibre was transferred to the injection port of a GC and the port was heated at 240degC for 3 min. The desorbed p-chlorophenol (4-chlorophenol; I) was analysed directly on a fused-silica column (15 m * 0.25 mm i.d.) coated with HP50+ (0.25 µm), operated with temperature programming from 55degC (held for 2 min) to 280degC at 15degC/min, with He as carrier gas (1 ml/min) and EIMS detection operated in selected-ion monitoring mode at m/z 128 for I (m/z 99 and 130 for confirmation). The calibration graph was linear from 0.2-50 mg/l of I in urine, with a detection limit of 8 µg/l. RSD (n = 7) were 14.2, 11.9 and 8.7%, respectively, for 3 quality control urines, containing 0.2, 5 and 25 mg/l of I. The mean recovery of 3 mg/l of I was 95% (n = 5).

Record - 96

TI- Improvement of the chemometric variety characterization of wines by improving the detection limit for aroma compounds.

AU- Weber, J;Beeg, M;Bartsch, C;Feller, KH;DelaCalleGarcia, D; Reichenbaecher, M;Danzer, M

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 6

PG- 322-326

AB- Wine samples (10 ml) were mixed with 3 g NaCl and 10 µl each of methanolic 3-decanol (internal standard) and 3- octanol (surrogate standard), both at 10 µg/ml were added. Portions (3 ml) were transferred to 5 ml headspace vials, an SPME fibre coated with 0.85 µm of polyacrylate was introduced into the headspace and the solution was stirred at 1 500 rpm for 3 h. The fibre was transferred to the GC injection port for 5 min at 270degC and the desorbed aroma compounds were analysed on a fused-silica column (30 m * 0.25 mm i.d.) coated with DB-Wax (0.25 µm), operated with temperature programming from 30degC (held for 5 min), to 200degC at 1degC/min, to 210degC at 20degC/min and EIMS detection operated in selected-ion monitoring (SIM) mode for the determination of 35 aroma and flavour compounds. Detection limits were in the range 1-700 ng/l, up to 200 times lower than in full-scan mode (FSM) and the linearity of calibration graphs was also better. The RSD of relative peak areas for 1 µg/l standards and for 90 samples were lower in SIM (generally 1.5-15.8%) were lower than in FSM mode (2.7-26.1%). The results were used to classify the 90 wines by grape variety, vintage and area by discrimination analysis, with cross-validation.

Record - 97

TI- Determination of volatiles from red wines made by carbonic maceration using solid-phase microextraction (SPME) technique.

AU- Vas, G;Loerincz, G

JN- Acta Alimentaria

PY- 1999

VO- 28

NO- 1

PG- 95-101

AB- Wine (125 ml) was placed in a 130 ml sampling bottle and the headspace was sampled for 10 min by inserting a 100 µm polydimethylsiloxane coated SPME fibre. The analytes were desorbed for 5 min from the SPME fibre into a GC injector at 250degC. Quantitation was performed by GC on a fused-silica column (30 m * 0.25 mm i.d.) coated with a poly(alkylene-glycol) operated with temperature programming from 35degC (held for 5 min) to 100degC at 5degC/min, then to 200degC (held for 1 min) at 3degC/min, and finally to 220degC at 10degC/min, with H₂ as carrier gas (1.8 ml/min) and FID. GC-70 eV EIMS (same GC conditions as above) was used to confirm the identity of the volatiles. The calibration mixture contained 20 aliphatic hydrocarbons (C₈-C₂₇). Wines made by carbonic maceration contained higher concentrations of diethyl succinate and ethyl acetate but lower amounts of methanol and hexanol compared to skin-fermented wine.

Record - 98

TI- Determination of trace xylene in contaminated palm oil by solid-phase micro-extraction and capillary gas chromatography.

AU- Chen, WR;Guo, CC;Hu, GC

JN- Fenxi Huaxue

PY- 1999

VO- 27

NO- 6

PG- 676-678

AB- Sample (10-20 g), contaminated with xylene (I) was mixed with 200 mug/g benzene (internal standard) to a final concentration of 2-5 mug/g. The mixture was transferred into a flask, covered with a rubber stopper, heated at 60degC on a water bath. A SPME apparatus was inserted in head-space of the flask for 1 min, and the SPME fibre was introduced into a GC injector directly. The fibre was heated and the desorbed I was analysed on a fused-silica column (50 m * 0.25 mm i.d.), coated with CBP20 (0.22 mum) operated isothermally at 80degC with H2 as carrier gas (1.6 ml/min) and FID. The calibration graph was linear from 0.2-20 mug/g of I with a detection limit of 0.06 mug/g. RSD were <5.3%.

Record - 99

TI- Determination of menthol and menthone in food and pharmaceutical products by solid-phase microextraction-gas chromatography.

AU- Ligor, M;Buszewski, B

JN- Journal of Chromatography, A

PY- 1999

VO- 847

NO- 1-2

PG- 161-169

AB- Samples of peppermint tea (18.11 mg), menthol sweets (46.6 mg), peppermint chewing gum (49.7 mg) or gastric peppermint drops (22.74 mg) were mixed with 10 ml methanol and 2 ml H2O in 5 ml headspace vials. The VOC were adsorbed on ethoxypoly(dimethylsiloxane)-coated fibres (10 mum) for 15 min at 30degC, without stirring. The fibres were transferred to the injection port of a GC and desorbed by heating at 200degC for 2 min. VOC were analysed on a fused-silica column (30 m * 0.53 mm i.d.) coated with RTX 200 (0.25 mum), operated with temperature programming from 40degC (held for 2 min) to 150degC (held for 4 min) at 10degC/min and to 225degC (held for 2 min) at 20degC/min, with He as carrier gas (flow rate not stated) and FID. Calibration graphs were linear for 25.86-85.28 mug/ml of menthol in the headspace solution; for the determination of menthone, standard additions of 1 mug/ml to the solution were made. Detection limits were 0.05-0.06 mug/ml. RSD (n = 3) on samples were 2.2-8.34%. The

SPME fibre tested gave better selectivity, lower detection limits and higher recoveries than commercial poly(dimethylsiloxane)-coated fibres.

Record - 100

TI- Volatiles from *Fusarium verticillioides* (Sacc.) Nirenb. and their attractiveness to Nitidulid beetles.

AU- Bartelt, RJ; Wicklow, DT

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 6

PG- 2447-2454

AB- Volatiles from *Fusarium verticillioides* (Sacc.) Nirenb were sampled using SPME sampling onto a 100 µm poly(dimethylsiloxane) fibre. After 30 min, the sample was injected onto a fused-silica column (30 m * 0.32 mm i.d.) coated with DB-1 (5 µm), or onto a fused-silica column (30 m * 0.25 mm i.d.) coated with DB-5MS (1 µm), with He as carrier gas and operated with temperature programming from 50degC (held for 1 min) to 250degC (held for 3 min) at 10degC/min and FID or MS. Volatiles were a blend of five alcohols (ethanol, 1-propanol, 2-methyl-1-propanol, 3-methyl-1-butanol and 2-methyl-1-butanol), acetaldehyde and ethyl acetate. Attraction to Nitidulid beetles was confirmed through wind tunnel bioassays.

Record - 101

TI- Quantitative analysis of pesticides in water by solid-phase micro-extraction (SPME) coupled with mass spectrometry.

AU- Massat, F; Laurent, A

JN- Spectra Analyse

PY- 1999

VO- 28

NO- 208

PG- 23-29

AB- Water (1.2 ml) was agitated for 30 min with a 100 µm polydimethylsiloxane or a 65 µm polydimethylsiloxane/divinylbenzene fibre from which the pesticides were desorbed by heating for 5 min at 260degC in a GC injector. The pesticides were determined on a DB5-MS column (30 m * 0.25 mm i.d.; film thickness 0.25 µm) operated with temperature programming from 80degC (held for 1.5 min) to 160degC (held for 1 min) at 25degC/min and then to 260degC (held for 21 min) at 3degC/min, He as carrier gas (12 psi) and 70 eV EIMS detection. The latter fibre gave the lower detection limits for 60 pesticides. The effects of experimental conditions on the SPME were studied. In some cases, e.g., atrazine (I), the detection limit was lowered by

the presence of NaCl. Recoveries of 4-8.6 µg/l alachlor, I, pirimiphos, lindane and trifluralin were 28.76-74.1%. Linear calibration ranges for 18 pesticides are tabulated. In the determination of 4 µg/l of I the RSD was 9% (n = 10). The method should be useful for monitoring drinking water. The results are compared with those obtained by extraction with CH₂Cl₂. The advantages and limitations of the method are discussed.

Record - 102

TI- Influence of extraction parameters and medium on efficiency of solid-phase microextraction sampling in analysis of aliphatic aldehydes.

AU- Keszler, A;Heberger, K

JN- Journal of Chromatography, A

PY- 1999

VO- 845

NO- 1-2

PG- 337-347

AB- The 12 aldehydes tested were added to sunflower oil and to water (20 ng/µl). Portions were transferred to 6 ml headspace phials, with variable liquid-air ratios and aldehyde samples were prepared with 7 or 100 µm poly(dimethylsiloxane) fibres at a range of temperatures and times. Samples were also prepared by total immersion of the fibres in phials completely filled with the aqueous solution. The analytes were desorbed in the injector at 220degC for 1 min; the injector also contained a narrow inlet liner (0.75 mm i.d.) to reduce tailing. After each analysis, the fibre was heated for 30 min in the injector. The aldehydes were separated on a column (30 m * 0.25 mm i.d.) coated with CP WAX 52CB (0.25 µm), with He (35 cm/s) as carrier gas, temperature programming (40-210degC, details given) and 70 eV EIMS detection, both in full scan (m/z = 10-650 in 0.5 s) for identification and selected-ion mode for determination. For headspace analysis the best results were given with a volume ratio of 1:1, the 100 µm fibre and an exposure of 30 min at 40degC. The detection limits were 50-500 pg/µl in the oil. The ion counts were about one order of magnitude greater from aqueous solution (headspace or immersion) than from the oil, giving detection limits of 5-50 pg/µl. A good separation of all 12 aldehydes was obtained in 27 min.

Record - 103

TI- Anaerobic biotransformation of trichlorofluoroethene in groundwater microcosms.

AU- Vancheeswaran, S;Hyman, MR;Semprini, L

JN- Environmental Science and Technology

PY- 1999

VO- 33

NO- 12

PG- 2040-2045

AB- Microcosms were constructed using 310 ml glass serum bottles containing 225 ml groundwater (supplemented with perchloroethene, trichlorofluoroethene and trichloroethene) with 85 ml headspace. Gas phase perchloroethene, trichlorofluoroethene, cis-1,2-dichlorofluoroethene, trans-1,2-dichlorofluoroethene, trichloroethene and cis-1,2-dichloroethene were determined in 50 µl portions of headspace by GC on a 30 m megabore GSQ-PLOT column with photoionization detection and FID. Headspace H₂ and CO₂ were determined using a Carboxen 1000 packed column (15 ft * 0.125 in) with thermal conductivity detection. Qualitative analysis of trichlorofluoroethene and its reduction products was performed after SPME (Zhang et al., Anal. Chem., 1994, 66, 844A) of 1 ml water from the microcosms by GC-MS with an Rtx-20 column (30 m * 0.25 mm i.d.) of 1 µm film thickness and electron ionization MS.

Record - 104

TI- Study on solventless sample preparation of pesticides with SPME-SFE technique.

AU- Selleh, SH;Saito, Y;Jinno, K

JN- Chromatography

PY- 1999

VO- 20

NO- 2

PG- 126-127

AB- Sample was subjected to SPME with a 85 µm polyacrylate coating as adsorbent, at 60degC for 3 h, then SFE was carried out in a Supelco model SFE-400 supercritical-fluid extractor equipped with a fused-silica capillary tubing (1 m * 50 µm i.d.) as restrictor, at 70degC, by bubbling vented CO₂ at pressure between 1500-4500 psi through 5 ml CH₂Cl₂ for static and dynamic extractions (all for 10 min). The eluate was evaporated to dryness and the residue was dissolved in 3 ml mobile phase and phenyl n-propyl ketone (internal standard) was added. Portions (20 µl) were analysed by HPLC on a 3 µm Phenomenex Luna-ODS column (15 cm * 4.6 mm i.d.), operated at 35degC, with aqueous 60% acetonitrile as mobile phase at 1 ml/min. Simazine (I), thiram, chlorothalonil, bensulide, thiobencarb and EPN in water were determined by this method. The recovery of very polar pesticides, e.g. I in SPME was low (1.16%). Other results are also discussed.

Record - 105

TI- Interaction between natural organic matter (NOM) and polycyclic aromatic compounds (PAC) - comparison of fluorescence quenching and solid phase microextraction (SPME).

AU- Doll, TE;Frimmel, FH;Kumke, MU;Ohlenbusch, G

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 364

NO- 4

PG- 313-319

AB- The interaction of phenanthrene (I) and 9-aminophenanthrene (II) with samples of brown lake water (III) and humic material (IV) was investigated by (i) fluorescence quenching, and (ii) SPME. Levels of dissolved organic carbon (DOC) were initially 210 mg/l III (by preconcentration) and 1732 mg/l IV; experiments were performed at 20degC using sample solutions (phosphate buffered to pH 7.3) of <30 mg/l DOC (i, no other details given) and (ii) <300 mg/l DOC, <0.07 mg/l I and <0.6 mg/l II. For i Steady-state and time-resolved fluorescence measurements were carried out (details given). Binding constants were determined from experimental data according to Stern-Volmer (Lakowicz, "Principles of fluorescence spectroscopy", Plenum Press, New York, 1983). For ii, sorption experiments were performed with a fused silica fibre coated with polydimethylsiloxane (7 µm) introduced into a 30 ml sample solution and after 20 min, the fibre was injected straight into the GC-MS system for analysis (conditions given). Sorption on to the fibre was monitored over 50 min for kinetic studies. The effect of reaction time and sample concentrations upon binding was studied. Generally, natural organic matter from III bound less polycyclic aromatics than that from IV. Specific interactions between amino groups and natural organic matter were demonstrated. Methods were compared, and results are discussed.

Record - 106

TI- In-tube SPME-LC analysis of phthalate esters in water samples.

AU- Saito, Y;Nakao, Y;Jinno, K

JN- Chromatography

PY- 1999

VO- 20

NO- 2

PG- 124-125

AB- An aqueous sample was introduced with a syringe pump into an extraction fused-silica capillary column (40 m * 0.25 mm) coated with DB-1, DB-17 or DB-WAX (0.25 µm) before percolation with H₂O if necessary and the phthalic acid esters (PAE) were eluted with a little methanol by using the syringe pump. The PAE were directly sampled into the injector of a liquid chromatograph for LC separation and analysis on a 5 µm Inertsil ODS-2 column (25 cm * 4.6 mm i.d.) [no other experimental conditions listed].

Record - 107

TI- An attempt by solid-phase microextraction with on-column silylation for a rapid and highly sensitive determination of bisphenol A.

AU- Takao, Y;Lee, HC;Arizono, K

JN- Bunseki Kagaku

PY- 1999

VO- 48

NO- 6

PG- 589-593

AB- Bisphenol A (an endocrine disruptor) was extracted from a water sample using SPME fibre, the fibre was placed in the injection port of a GC instrument and bisphenol A was thermally desorbed. The analyte was subjected to on-column derivatization by the subsequent injection of bis(trimethylsilyl)trifluoroacetamide. The derivatized analyte was detected by MS. The derivatization resulted in a 20-fold increase in single response; the detection limit was sim1 ppb.

Record - 108

TI- Efficiency of direct solid-phase microextraction from water: comparison of different fibre types including a new C8-coating.

AU- Popp, P;Paschke, A

JN- Chromatographia

PY- 1999

VO- 49

NO- 11-12

PG- 686-690

AB- The extraction efficiencies of 7 µm polydimethylsiloxane (PDMS), 30 µm PDMS, 100 µm PDMS, 65 µm PDMS-divinylbenzene, 85 µm polyacrylate, 65 µm Carbowax-divinylbenzene, 75 µm Carboxen-PDMS and a new 65 µm C8 fibre (SPME) were evaluated using 9 halogenated benzenes, 7 organochlorine pesticides (OCP) and 6 PCB (as test compounds) in aqueous solution (0.5-1 ng/ml). In each case, SPME sampling was carried out in 8 ml vials sealed with Teflon-lined septa loaded with 5 ml aliquots of aqueous phase. Extraction was carried out with strong stirring (ca 1000 rpm) with glass-coated mini-impellers for 1 h. The extracted analytes were thermally desorbed and determined by GC on CP SIL 5 or 8 CB capillary columns operated with temperature programming and either FID or ECD (details tabulated). Extraction efficiencies were calculated using single mid-point standard injections or 12-point calibrations of the ECD signals (results tabulated). The 75 µm Carboxen-PDMS fibre was shown to be the most efficient extractants for low boiling solutes. Whereas the 65 µm C8 fibre enabled the extraction of all of the compounds studied, in no one case was it the most efficient coating. 100 µm PDMS and the more polar coatings were shown to be applicable for the recovery of as broad range of compounds, in contrast the 7 µm and 30

mum materials were less efficient.

Record - 109

TI- Automated drug analysis by in-tube solid-phase microextraction-liquid chromatography-mass spectrometry.

AU- Kataoka, H;Lord, HL;Pawliszyn, J

JN- Chromatography

PY- 1999

VO- 20

NO- 2

PG- 142-145

AB- Construction of the cited system (diagram illustrated) is discussed.

The optimized in-tube SPME conditions for stimulants were: 15 draw and eject cycles of 35 μ l sample in Tris hydrochloride buffer of pH 8.5 at 100 μ l/min, using an Omegawax 250 capillary column (60 cm * 0.25 mm i.d.) Following column switching (schematic shown), the 5 drugs (amphetamines as model compounds; tabulated) were eluted with LC mobile phase onto a 3 μ m Supelcosil LC-CN column (3.3 cm * 4.6 mm i.d.), with 50mM-ammonium acetate/acetonitrile (17:3) as mobile phase (0.4 ml/min) and after eluate splitting, electrospray MS detection operated in selected-ion monitoring mode (m/z listed for each analyte). The calibration graphs were linear from 2- 100 ng/ml for the 5 amphetamines tested, with detection limits in the range 0.4-0.8 ng/ml. Recoveries of these stimulants in urine were >80%. No carryover was observed. The methods is also applicable to antihistamines.

Record - 110

TI- Automated determination of "Ecstasy" and amphetamines in urine by SPME and capillary gas chromatography after propylchloroformate derivatization.

AU- Ugland, HG;Krogh, M;Rasmussen, KE

JN- Journal of Pharmaceutical and Biomedical Analysis

PY- 1999

VO- 19

NO- 3-4

PG- 463-475

AB- To 1200 μ l urine was added methoxyphenamine (5 μ g/ml; internal standard) and 300 μ l 2.5M-K₂CO₃/KHCO₃ buffer of pH 10.8 containing 0.5 g NaCl. After mixing, 8 μ l propyl chloroformate was added with vortex mixing. The mixture was loaded into the GC autosampler equipped with a polydimethylsiloxane coated fibre (film thickness 100 μ m) for automated SPME. The fibre was agitated for 16 min during enrichment. The extracted analytes were thermally desorbed into the heated (300degC) split-splitless injector and into the GC system.

Analysis was on either a polymethylsilicone column (30 m * 0.25 mm i.d.; 0.25 µm film thickness) operated with temperature programming from 180degC (held for 1 min) to 300degC (held for 1 min) at 20degC/min, with He as carrier gas (1 ml/min) and N-P detection, or on a methylsilicone column (12 m * 0.2 mm i.d.; 0.33 µm film thickness) operated with temperature programming as above and 70 eV EIMS detection. The calibration graphs were linear from 0.1-10 µg/ml of amphetamine, methylamphetamine and their methylenedioxyated analogues and detection limits were 5-15 ng/ml. Inter- and intra-assay RSD (tabulated) were 0.8-11.1% (n = 6) and 1.9-10% (n = 15), respectively.

Record - 111

TI- Headspace solid-phase microextraction with 1-pyrenyldiazomethane in-fibre derivatisation for analysis of faecal short-chain fatty acids.

AU- Mills, GA;Walker, V;Mughal, H

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 730

NO- 1

PG- 113-122

AB- Short-chain fatty acids (SCFA) were extracted from faeces by SPME on a polyacrylate fibre (85 µm film thickness) that had been pretreated with 5 mg/ml 1-pyrenyldiazomethane in n-hexane for 15 min (as derivatizing agent for SCFA). The extracted SCFA desorbed from the fibre in the injection port of the Hewlett-Packard GC for 4 min directly onto the fused-silica analytical column (30 m * 0.22 mm i.d.) coated with BPX-5 (0.25 µm), operated with temperature programming from 100degC (held for 2 min) to 280degC (held for 1 min) at 20degC/min then to 310degC (held for 10 min) at 2degC/min, with He as carrier gas (1 ml/min) and EIMS detection operated in selected-ion monitoring mode (operating m/z listed for each SCFA). Good linearity in the µmol range, precision and recoveries were achieved for most fatty acids in the study (results are tabulated). 2-Methylbutyric acid was found for the first time in all faecal samples.

Record - 112

TI- Simultaneous determination of mercury(II) and alkylated mercury, lead, and tin species in human body fluids using SPME-GC-tandem MS.

AU- Dunemann, L;Hajimiragha, H;Begerow, J

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 5-6

PG- 466-468

AB- Urine (10 ml) was mixed with 10 ml 0.2M-acetate buffer of pH 5.3 in a headspace vial, the vial was closed with a PTFE coated septum and 1 ml 1% sodium tetraethylborate was added using a syringe. After 10 min, the derivatized metal species were adsorbed on a poly(dimethylsiloxane)-coated fused-silica SPME fibre which was then thermally desorbed for 3 min at 250degC in the split/splitless injector of a GC system. Separation was performed using a VA-5 MS column (30 m * 0.25 mm i.d.; 0.25 mum film thickness) operated with temperature programming from 40degC (held for 3 min) to 250degC (held for 10 min) at 10degC/min and He as carrier gas (5 ml/min). The ion-trap detector with EI ionization was run either in the MS or tandem MS mode in the mass range 100-350 m/z. The use of tandem MS resulted in detection limits at least one order of magnitude better than with the MS mode. For inorganic mercury, methylmercury, trimethyllead and monobutyl-, dibutyl- and tributylstannane derivatives the linear dynamic range was 10-400 ng/l with use of tandem MS detection; RSD (n = 6) were 4-15% and detection limits were 7-22 ng/nl.

Record - 113

TI- Electrochemical control of solid phase microextraction using unique conducting polymer coated fibres.

AU- Gbatu, TP;Ceylan, O;Sutton, KL;Rubinson, JF;Galal, A;Caruso, JA;Mark, HB_J

JN- Analytical Communications

PY- 1999

VO- 36

NO- 5

PG- 203-205

AB- A Pt wire microfibre electrode (1.5 cm * 200 mum) was conditioned in a 10mM-aqueous H₂SO₄ solution using a model 173 potentiostat/galvanostat with an applied potential of -1.6 V vs. Ag/Ag Cl for 2 min. After conditioning, 3-methylthiophene was polymerized on the surface of the microfibre electrode to poly(3-methylthiophene) by repeatedly cycling the electrode (100 mV/scan) between -0.2 and +1.75 V in an acetonitrile solution containing 50mM-3-methylthiophene and 75mM-tetrabutylammonium tetrafluoroborate electrolytes. The reference electrode was Ag/AgCl and the auxiliary electrode was a Pt sheet (2 * 2 cm²) in a 10mM-aqueous H₂SO₄ solution. The conducting polymer coated microfibre electrode was used to extract arsenate ions from aqueous solutions without derivatization. After immersion in the sample solution, the conducting polymer film was converted to its oxidized positively charged form by applying a constant potential of -1.2 V with respect to the reference electrode (Arsenate ions migrated into the film to maintain electroneutrality). Upon subsequent reversal of the potential to -0.6 V, the polymer film was converted to its reduced

neutral from and the arsenate ions were expelled into a smaller volume (200 µl) of H₂O for analysis by FIA with ICP-MS detection.

Record - 114

TI- Preparation of polymer-based adsorbents for solid-phase extraction-adsorption properties and addition of selectivity.

AU- Hosoya, K;Sasaki, H;Ikegami, T;Kimata, K;Tanaka, N

JN- Chromatography

PY- 1999

VO- 20

NO- 2

PG- 178-179

AB- Adsorbents for SPME were prepared from functionalized monomers by the multi-stage swelling polymerization methods as particles with uniformly controlled sizes and properties. The specific molecular recognition ability, particularly by molecular imprinting technique, was also achievable to provide adsorption selectivity. Especially useful are polymeric adsorbents with enhanced retention power on halogen atom, e.g. for use in enriching environmental pollutants like PCB and dioxins, or those that provide additional hydrophilicity.

Record - 115

TI- Solid-phase microextraction application in gas chromatography/olfactometry dilution analysis.

AU- Deibler, KD;Acree, TE;Lavin, EH

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 4

PG- 1616-1618

AB- Solid-phase microextraction using polydimethylsiloxane fibre coatings of three different thicknesses were used for the sampling of VOC for GC/olfactometry based on dilution analysis (e.g. CharmAnalysis). Fibres were exposed to the headspace of coffee samples for 15 min prior to desorption directly in the injection port of a fused-silica column (25 m * 0.2 mm i.d.) coated with DB-1 (0.33 µm), operated with temperature programming from 40degC (held for 3 min) to 200degC at 4degC/min then to 250degC (held for 15 min) at 20degC/min, with He as carrier gas 925 cm/s) and olfactometry. The application of SPME was feasible because it was possible to vary the fibre thickness and length to achieve various adsorbent volumes. Results obtained by this method were compared to those achieved using serial solvent extraction with Freon 113 and ethyl acetate. Results are tabulated.

Record - 116

TI- Matrix solid-phase dispersion extraction of organophosphorus and synthetic pyrethroid pesticides in cashew nut and passion fruit.

AU- Dorea, HS;Lancas, FM

JN- Journal of Microcolumn Separations

PY- 1999

VO- 11

NO- 5

PG- 367-375

AB- Five organophosphorus pesticides (OPP) and the pyrethroid, permethrin were extracted simultaneously from cashew nuts and the extract was analysed by GC with ⁶³Ni ECD. Analytes were extracted from grated fruit into ethyl acetate followed by cleanup by passage through a glass column (25 cm * 10 mm i.d.) packed with neutral alumina/silica gel (1:1) and subsequent elution of the OPP with ethyl acetate.

Alternatively, extraction was by matrix solid-phase dispersion. GC was effected on a fused-silica column (25 and 50 m * 0.25 mm i.d., for ECD and MS, respectively) coated with 0.33 µm of 5% phenylmethylsilicone (LM-5; 0.33 µm) operated with temperature programming from 160degC (ECD) or 180degC (MS) to 300degC (details given), with H₂ as carrier gas for ECD (He for MS), and 70-eV EIMS detection operated in selected-ion monitoring mode (m/z listed for each analyte). Calibration graphs were linear from 0.03-0.5 (parathion), 0.04-0.7 (diazinon) and 0.06-1.0 µg/ml (malathion, methidathion, pyrazophos and cis- and trans-permethrin. The RSD (n = 3) of recovery and of determination for each pesticide in cashew nuts and passionfruit are tabulated.

Record - 117

TI- A new concept for the measurement of total volatile compounds of food.

AU- Azodanlou, R;Darbellay, C;Luisier, JL;Villettaz, JC;Amado, R

JN- Zeitschrift fuer Lebensmittel-Untersuchung und -Forschung A: Food Research and Technology

PY- 1999

VO- 208

NO- 4

PG- 254-258

AB- An investigation of four different types of SPME fibres for the extraction of total VOC in foods, using strawberries as a model, is presented. Sample (200 g) was placed in a tightly closed flask, fitted with a Teflon-coated silicone septum and the flask was equilibrated at 25degC on a water bath for 30 min. The VOC were collected by inserting the SPME fibres, coated with poly(dimethylsiloxane) [PDMS], polyacrylate, Carbowax/divinylbenzene (CW/DWB) and bipolar PDMS/DVB, into the headspace via the septum and

sampling for 5 min. The fibres were withdrawn, placed in the injection port of a Carbo Erba HRGC 5300 gas chromatograph (Carbo Erba, Milan, Italy) and the VOC were desorbed on a fused-silica column (dimensions not given) operated isothermally at 250degC, with He as carrier gas (5 ml/min) and FID. Results (tabulated) showed that the patterns of VOC obtained with the different types of SPME enabled differentiation between the total VOC present in the sample, depending upon their chemical nature. Based on the analysis of strawberries, the SPME method had a high reproducibility (RSD sime5%); allowed differentiation between the 6 varieties in a way which is consistent with an hedonic evaluation of these varieties and showed the variation in VOC between individual fruits. The method successfully applied to the VOC analysis of coffee and spice samples. The total analysis time was sime40 min and 10-20 samples/day could be analysed by the method.

Record - 118

TI- Studies of the composition of distillates from leachate by gas chromatography-mass spectrometry coupled to solid-phase microextraction.

AU- Saba, A;Pucci, S;Raffaelli, A;Salvadori, P

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 10

PG- 966-970

AB- Distillation at 40 Torr and sime36degC of an untreated leachate (pH sime8) having a COD of 7700 mg/l yielded a distillate having a COD of 70 mg/l; distillation of the same leachate after treatment with NaOH to pH 10 or with HCl to pH 4 yielded distillates having COD of 93 and 273 mg/l, respectively. The volatile components of the untreated leachate and the three distillates were sampled by either immersion of a 100 mum polydimethylsiloxane-coated SPME fibre for 1 h or exposure of the fibre to the headspace for 20 min during magnetic stirring, and were identified by desorption at an injector temperature of 250degC on to a column coated with 5% phenyl polydimethylsiloxane (25 m * 0.25 mm i.d.; film thickness 1 mum) and operated with temperature programming from 80degC (held for 3 min) to 280degC (held for 1 min) at 15degC/min, with He as carrier gas and 70 eV MS detection with scanning from m/z 30-400. Compounds were identified by spectral library search, and their identities were confirmed by off-line ionspray MS. The identities of eight aliphatic (linear and cyclic) and aromatic hydroxy-compounds and four bicyclic compounds common to all four samples are tabulated, as are those of four carboxylic acids found only in the distillate from the acidified leachate. The technique is suitable for the routine examination of

leachates in order to determine what treatment is necessary.

Record - 119

TI- Role of humic acids in the titanium oxide-photocatalysed degradation of tetrachloroethene in water.

AU- Selli, E;Baglio, D;Montanarella, L;Bidoglio, G

JN- Water Research

PY- 1999

VO- 33

NO- 8

PG- 1827-1836

AB- Tetrachloroethene was determined by SPME with a polydimethylsiloxane fibre and GC on a DB-624 fused-silica column (75 m * 0.53 mm i.d.; film thickness 3 µm) operated with He as carrier gas (25 ml/min), temperature programming from 40degC (held for 5 min) to 200degC (held for 5 min) at 8degC/min and MS detection from m/z 50-250. The internal standard was 139 µg/l 4-bromofluorobenzene. Dichloroacetic acid and TFA were methylated with BF₃/methanol (details given), the products were extracted with cyclohexane and the extract was analysed on a HT8 fused-silica column (50 m * 0.25 mm i.d.; film thickness 0.25 µm) operated with H₂ as carrier gas (1.8 ml/min), temperature programming from 35degC (held for 8 min) to 145degC (held for 10 min) at 5degC/min and 15 mCi ⁶³Ni ECD. The methods were used to monitor the effects of humic acids on the TiO₂-photocatalysed degradation of tetrachloroethene in water.

Record - 120

TI- Optimization of a matrix solid-phase dispersion method with sequential clean-up for the determination of alkylphenol ethoxylates in biological tissues.

AU- Zhao, M;vanderWielen, F;deVoogt, P

JN- Journal of Chromatography, A

PY- 1999

VO- 837

NO- 1-2

PG- 129-138

AB- Octadecylsilica was used as solid phase for matrix dispersion and sequential cleanup for isolation and purification of alkylphenols and their ethoxylates as contaminants in fish tissues. A 20-ml glass syringe (with its plunger) was mounted vertically as the separation column, a mixture (20 g) of powdered silica/tissue was applied directly above a layer (3 g) of 5% H₂O-deactivated alumina and methanol was used as eluent. Model samples used were 0.5 g of fish or 0.9 g of mussel tissue, each with 108 ng of t-octylphenol (I) and 300 ng of nonylphenol tetraethoxylate (II) added. Two four-stage elution

programmes (3 * methanol then dichloromethane, and 2 * acetonitrile, methanol and dichloromethane) were applied. Mean test recoveries (n = 5) were 91+-12 and 100+-5%, respectively, for I and II. HPLC analyses of extracts were effected on a 5 µm LiChrospher 100 RP-18 column (12.5 cm * 4 mm i.d.) with aqueous 80% methanol as mobile phase (1 ml/min).

Record - 121

TI- Trace determination of diethyl phthalate in aqueous media by solid-phase microextraction-liquid chromatography.

AU- Kelly, MT;Larroque, M

JN- Journal of Chromatography, A

PY- 1999

VO- 841

NO- 2

PG- 177-185

AB- Into 10 ml of H₂O containing 2.5 g of NaCl was immersed an acetonitrile-conditioned 60 µm polydimethylsiloxane/divinylbenzene fibre for 15 min. After extraction, the fibre was withdrawn and was placed in the interface between valves in a column switching system (schematic shown). The fibre was swept directly onto the HPLC column (details given). Diethyl phthalate (I) was analysed on a 5 µm Nucleosil C18-50 dp column (25 cm * 3 mm i.d.), with aqueous 52.5% acetonitrile as mobile phase (0.5 ml/min) and detection at 226 nm. The calibration graph was linear from 5-75 ng/ml of I with a detection limit of 1 ng/ml. The average RSD (n = 5) was 3.06% over the calibration range. Carbowax/templated resin and polyacrylate fibres were also investigated.

Record - 122

TI- Solid-phase microextraction for the determination of iodinated trihalomethanes in drinking water.

AU- Cancho, B;Ventura, F;Galceran, MT

JN- Journal of Chromatography, A

PY- 1999

VO- 841

NO- 2

PG- 197-206

AB- Water (30 ml) was mixed with 2 ml 1,2-dibromopropane (30 mg/l in methanol; internal standard) and 7.5 g NaCl in a sealed vial. A 65 µm carbowax/divinylbenzene fibre was exposed to the headspace above the solution and the sample was agitated with a magnetic stirrer at 1100 rpm to achieve equilibration. The absorbed iodinated trihalomethanes (ITHM) were desorbed from the fibre directly through the GC injection port onto a fused-silica column (30 m * 0.25 mm

i.d.) coated with DB-1 (1 µm), operated with temperature programming from 35degC (held for 9 min) to 40degC (held for 3 min) at 1degC/min, to 220degC (held for 10 min) at 6degC/min, He as carrier gas (flow rate not stated) and ECD. Headspace calibration graphs were linear in the range 0.1-20 µg/l CHCl₂I, CHBrCl₂, CHBr₂I, CHCl₂I₂, CHBrI₂ and CHI₃ with detection limits of 2-3 ng/l. For 5 µg/l of ITHM, intra- and inter-day RSD (n = 5 and 9, respectively) were <7.2%. ITHM were also extracted from water samples by a liquid-liquid extraction (LLE) method with 2 ml t-butyl methyl ether and analysed by GC (experimental details given). Results (tabulated) showed that the SPME method gave a good agreement with results obtained by the LLE method. The Carbowax-divinylbenzene fibre appeared to be the most suitable fibre material for the analysis of ITHM.

Record - 123

TI- Solid-phase micro-extraction of volatiles from water using open cap phials.

AU- Matisova, E;Sedlakova, J;Simon, P;Welsch, T

JN- Chromatographia

PY- 1999

VO- 49

NO- 9-10

PG- 513-519

AB- Water (1.3 ml), treated with 4.25, 42, 425 or 4250 µg/l of benzene, toluene, ethylbenzene, and p- and o-xylene were transferred to 4 ml glass phials (3 cm * 8 mm i.d.), which were stoppered with PTFE caps bearing a central hole (0.8 mm i.d.). The samples were stirred at 25degC for 5 min, with a fibre coated with 0.1 mm poly(dimethylsiloxane) in the headspace. The fibre was withdrawn into its needle and transferred to the injector port at 180degC for desorption. The compounds were analysed on a de-activated pre-column (1 m * 0.53 mm i.d.) and a column (25 m * 0.32 mm i.d.) coated with CP-Sil 13 CB (1.2 µm), operated with temperature programming from 35degC (held for 1.5 min), to 180degC (held for 12 min) at 18degC/min, with He as carrier gas (27 cm/s) and FID. Calibration graphs were linear for the range cited. The peak areas for the 42.5 µg/l standard were compared with those obtained from phials with PTFE-lined septa. There was no significant difference, confirming a theoretical analysis of expected losses. The RSD were larger for the phials with septa, which was ascribed to the uncontrolled effects of minute cracks in the septa. The method was applied to the analysis of waste water from a paint factory.

Record - 124

TI- Aqueous phase hexyl chloroformate derivatization and solid-phase

microextraction: determination of benzoylecgonine in urine by gas chromatography-quadrupole ion trap mass spectrometry.

AU- Hall, BJ;Parikh, AR;Brodbelt, JS

JN- Journal of Forensic Sciences

PY- 1999

VO- 44

NO- 3

PG- 527-534

AB- A 70 µl portion of acetonitrile/H₂O/hexanol/2-(dimethylamino)-pyridine (5:2:2:1) was added to 1 ml urine and the solution was sonicated at 42degC. A 12 µl portion of hexyl chloroformate was added with further sonication for 3 min. A 250 µl portion of the reaction mixture was then added to 2 ml H₂O and 2 ml pH 7 buffer for SPME. A 100 µm polydimethylsiloxane SPME fibre was used, typically at 55degC for 10 min. The fibre was then transferred to the injector port of a GC for a 7 min desorption at 280degC. GC was performed on a fused-silica column (30 m * 0.25 mm i.d.) coated with HP-5MS (0.25 µm) operated with temperature programming from 60degC (held for 0.1 min) to 270degC (held for 5.5 min) at 25degC/min and ion trap EIMS detection operated in selected-ion monitoring mode at m/z 373, 252 and 82 and 376, 255 and 85, respectively, for benzoylecgonine (I) and its d₃ analogue (internal standard; IS). The calibration graph was linear from 0.1-20 µg/ml of I with a detection limit of 0.03 µg/ml. Intra- and intra-day RSD (n = 6 and 3, respectively) were 6.8-8.8 and 2.2-3.3%, respectively.

Record - 125

TI- Pheromone analysis using capillary gas chromatographic techniques.

AU- Jones, GR;Oldham, NJ

JN- Journal of Chromatography, A

PY- 1999

VO- 843

NO- 1-2

PG- 199-236

AB- A review of GC methods for the analysis of pheromones, which contain complex mixtures of nanogram or picogram amounts of each compound, is presented. Sampling and injection methods (liquid-liquid extraction, trapping of volatile compounds, solventless injection, SPME) are highlighted, together with derivative preparations on the microscale (acids, esters, alcohols, double bonds, chiral derivatives). The preferred types of column (for separation, determination, structure elucidation, vapour pressure estimation) are also discussed as well as detection methods (FID, MS, FT-IR spectrometry and insect response by electroantennogram). Many examples of the technique are given. (180 references).

Record - 126

TI- Investigation by solid-phase microextraction and gas chromatography-mass spectrometry of organic films on stone monuments.

AU- DeAngelis, F;DiTullio, A;Mellerio, G;Quaresima, R;Volpe, R

JN- Rapid Communications in Mass Spectrometry

PY- 1999

VO- 13

NO- 10

PG- 895-900

AB- A surgical lancet was used to obtain a small sample of the coherent black deposit (gypsum coated with organic material) on the surface of 13th-17th century masonry without damaging the stonework itself. The powdered deposit (0.1 g) was heated at 210degC for 30 min in a septum phial with magnetic stirring, then a 100 mum diameter polydimethylsiloxane or 85 mum polyacrylate fibre was exposed to the headspace while the deposit was heated at 210degC for a further 30 min without stirring. The fibre was then transferred to the injection port for desorption at 270degC for 4 min in the splitless mode and analysis on a column coated with 5% phenylmethylsiloxane (30 m * 0.25 mm i.d.; film thickness 0.25 mum) and operated with temperature programming from 35degC (held for 4 min) to 270degC (held for 30 min) at 10degC/min and 70 eV EI ion-trap MS detection (m/z range 45-600). The chromatograms were cleaner than those obtained after Soxhlet extraction of much larger (and more detrimental) samples, and showed higher signal-to-noise ratios. The results for samples from three locations are presented and discussed.

Record - 127

TI- An improved interface for coupling solid-phase microextraction (SPME) to high performance liquid chromatography (HPLC) applied to the analysis of explosives.

AU- Wu, L;Almirall, JR;Furton, KG

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 5

PG- 279-282

AB- The improved SPME/HPLC interface (schematic shown) includes a 200 mul desorption chamber and an 8 mum Supelco C-8 refocusing unit (3 cm * 4.6 mm i.d.). The SPME CW/TPR fibre was preconditioned with mobile phase and the conditioned fibre exposed to a stirred solution of EPA 8330 explosive mixture. HPLC separations were effected on a 5 mum Res-Elut CN column (3 cm * 4.6 mm i.d.) connected in series to a 5 mum Bondesil C-18 column (25 cm * 4.6 mm i.d.), with methanol/H₂O (1:1) as mobile phase, and detection at 254 nm. The new interface

gave an average 83% increase in peak areas and smaller RSD (50% lower on average) than the conventional interface. Reproducibility was also excellent, with an average fall in RSD of 77%. Linearity studies were performed by extracting spiked explosives samples with concentrations of 10 ng/ml to 1000 ng/ml of each. The correlation coefficients of multipoint calibration curves for TNT and RDX were 0.9985 and 0.9971, respectively.

Record - 128

TI- A comprehensive sample preparation scheme for accelerants in suspect arson cases.

AU- Ren, QL;Bertsch, W

JN- Journal of Forensic Sciences

PY- 1999

VO- 44

NO- 3

PG- 504-515

AB- Dynamic and static adsorbent-based heated headspace methods based on charcoal adsorption and solvent extraction for the enrichment of volatiles from fire debris were compared to SPME procedures. The SPME method was optimized with respect to fibre type, sampling time, sampling temperature, analyte concentration and the effects of water. Collection efficiencies were evaluated for a variety of accelerant types ranging from methanol to diesel fuel. A two-step method based on SPME with two different fibres is described. Polar and water-soluble accelerants such as ethanol and light petroleum distillates were most effectively enriched at low temperature on the carbon-based adsorbant Carboxen, whilst low-volatility accelerants such as diesel fuel can be recovered on a methylpolysiloxane-type fibre at elevated temperature. GC and GC-MS were used to evaluate accelerant recovery (details tabulated). Limitations of currently used sample preparation methods are discussed.

Record - 129

TI- In-tube solid phase micro-extraction-gas chromatography of volatile compounds in aqueous solution.

AU- Tan, BCD;Marriott, PJ;Lee, HK;Morrison, PD

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 5

PG- 651-655

AB- In-tube SPME was effected by passing an aqueous solution of the analytes through a coated capillary column (1 m) under N₂ pressure. The column was then dried with a N₂ flow, after which the sorbed

analytes were eluted with a small volume of an organic solvent. The eluate was analysed by GC with FID. The method was applied to several aromatic hydrocarbons and phenols.

Record - 130

TI- Studies on the application of solid-phase microextraction for analysis of volatile organic sulfur compounds in gaseous and liquid samples.

AU- Wardencki, W; Namiesnik, J

JN- Chemia Analityczna (Warsaw)

PY- 1999

VO- 44

NO- 3A

PG- 485-493

AB- The use of a poly(dimethylsiloxane)-coated fibre (100 μ m thickness) for sampling sulfur VOC (thiophene, dimethyl sulfide and diethyl sulfide) from aqueous (100 μ g/l to 5 mg/l) and gaseous (5-100 mg/l) samples was investigated. SPME was carried out for 10 min with stirring: (i) in the aqueous phase, from a 24 ml vial of solution; and (ii) in the gaseous phase, from the headspace above solutions injected into a deactivated glass vessel (1 l volume) filled with either N₂ or propane/butane mixture. The fibre was inserted into the GC injector and the analytes desorbed at 200degC for 3 min, then analysed on a Rtx-1 column (30 m * 0.32 mm i.d.) coated with polydimethylsiloxane (4 μ m) operated with temperature programming from 60degC (held for 3 min) to 110degC at 7degC/min, then to 200degC (held for 1 min) at 15degC/min, with H₂ as carrier gas (1 ml/min) and flame-photometric detection. Optimization of the detector conditions and SPME procedures was performed. Calibration graphs were linear over the concentration ranges studied in the different matrices, and detection limits of 0.1 mg/l and 1 μ g/l achieved for gaseous and aqueous samples respectively. Use of a cryotrap increased sensitivity.

Record - 131

TI- Miniaturised on-line solid-phase extraction for enhancement of concentration sensitivity in capillary zone electrophoresis.

AU- Petersson, M; Wahlund, KG; Nilsson, S

JN- Journal of Chromatography, A

PY- 1999

VO- 841

NO- 2

PG- 249-261

AB- An enrichment capillary (28-58 cm; 21.2-51.2 cm to the detector), comprising three fused silica capillaries of two different internal

diameters were inserted into each other, and the assembly was packed with alkyl-diol silica (details given). Portions (30 nl) of 1-10nM-terbutaline sulfate standard solutions in H₂O (as model compound) were injected into the enrichment capillary. Separation was effected with 40mM-potassium phosphate buffer of pH 6.5 over 0.3 min at 140 kPa onto a capillary column (58 cm * 50 µm i.d.) operated at an applied voltage of 10-20 kV, filled with 40mM-potassium phosphate buffer of pH 6.5 as running buffer and detection at 200 nm. The method enables a concentration sensitivity increase of about 7000, yielding a detection limit of 0.6nM-terbutaline, compared to 4.4µM-terbutaline without enrichment. Separation performance with enrichment was estimated at 250,000 plates. Applications to the analysis of terbutaline enantiomers and terbutaline in plasma are briefly discussed.

Record - 132

TI- A novel solid-phase coating for solid-phase microextraction prepared with sol-gel technology and its applications.

AU- Wang, ZY

JN- Sepu

PY- 1999

VO- 17

NO- 3

PG- 280-283

AB- A 300 µl portion of methyltrimethoxysilane was mixed with 150 mg hydroxy-terminated poly(dimethylsiloxane), 30 mg silicone oil and 200 µl TFA and centrifuged for 3 min. A quartz fibre was inserted the supernatant and after 20 min, a sol-gel layer (40 µm) was formed on the fibre. The fibre was aged by heating at 320degC for 2 h. The property of the sol-gel coatings was investigated, it had higher stability and shorter extraction and desorption time than conventional polydimethylsiloxane coatings. The detection limits of benzene series were ≤ 0.65 ng/ml. The headspace SPME coupled with capillary GC using sol-gel coated fibre was used to determination of hexachlorobenzene in environmental sample with satisfactory results.

Record - 133

TI- New coating surfaces of fibers for solid-phase microextraction.

AU- Ligor, M;Scibiorek, M;Buszewski, B

JN- Journal of Microcolumn Separations

PY- 1999

VO- 11

NO- 5

PG- 377-383

AB- Theoretical and practical aspects of use of coated fibres in SPME are

discussed. Fibres compared are one coated with ethoxydimethylsiloxane and with poly(urethane-acrylate), and fused-silica fibres untreated and etched with HF, plus dimethylsiloxane as reference. Adsorption capacities and extraction times at immersion equilibria were measured for each fibre with standard BTEX (benzene/toluene/xylenes) in methanol. Distribution constants were evaluated for each aromatic hydrocarbon and values were compared with conventional octanol/H₂O partition coefficients.

Record - 134

TI- Theory of analyte extraction by selected porous polymer SPME fibres.

AU- Gorecki, T;Yu, X;Pawliszyn, J

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 5

PG- 643-649

AB- A theoretical description of the analyte extraction process for adsorption-based SPME fibres is presented. The model is based on the Langmuir adsorption isotherm. Expressions describing the amount of analyte extracted by the fibre in two- and three-phase systems are derived and the effect of some experimental variables is considered. The extraction of benzene and 4-methylpentan-2-one from water is used to illustrate the phenomena discussed.

Record - 135

TI- Matrix solid-phase dispersion microextraction and determination by high-performance liquid chromatography with UV detection of pesticide residues in citrus fruit.

AU- Valenzuela, AI;Lorenzini, R;Redonodo, MJ;Font, G

JN- Journal of Chromatography, A

PY- 1999

VO- 839

NO- 1-2

PG- 101-107

AB- Whole orange fruit was homogenized and a 500 mg portion was blended with 0.5 g of C8 silica. The mixture was packed into a glass column (10 cm * 9 mm i.d.) and the carbamate and urea pesticides were eluted with 15 ml CH₂Cl₂ and the eluate was evaporated to dryness with air at 50degC. The residue was dissolved in 500 µl acetonitrile and a 20 µl portion was analysed by HPLC on a 5 µm Kromasil C18 column (25 cm * 4.6 mm i.d.) equipped with a guard column (3 cm * 4.6 mm i.d.) of the same material, with gradient elution (0.5 ml/min) with aqueous acetonitrile (ACN) [from 88 to 90% ACN over 13 min, decreasing to 88% (held for 15 min) over 2 min] and detection at 200 nm. Test

recoveries were 80% for the carbamate, benfuracarb (I) and for four urea insecticides diflubenzuron (II), flufenoxuron (III), hexaflumuron (IV) and hexythiazox (V) were 78, 84, 74 and 75%, respectively. Lower limits of determination were 0.15 ppm for I and V or 0.25 ppm for others, which were below the legal maxima allowed of 2 ppm (I), 1 ppm (II and V) and 0.3 ppm (III and IV). The C8 and C18 silicas were the best of eight tested adsorbents, and C8 silica showed extraction RSD of 2-8% for the five pesticides.

Record - 136

TI- Cryotrapping-SPME-GC analysis of cheese aroma.

AU- Jaillais, B;Bertrand, V;Auger, J

JN- Talanta

PY- 1999

VO- 48

NO- 4

PG- 747-753

AB- Frozen cheese (10 g) was cut and placed in a flask held at 60degC in a water bath, and volatile compounds and water were collected over 45 min in a second flask immersed in liquid N₂. The trapped material was allowed to warm to room temperature, a poly(dimethylsiloxane)-coated SPME fibre was placed in the stirred aqueous phase for 4 min and the fibre was inserted in a GC injector for desorption over 3 min at 200degC. The cheese volatiles were analysed on a fused-silica column (30 m * 0.32 mm i.d.) coated with DB-1 (0.25 µm) and operated with temperature programming from 40 to 200degC at 2degC/min, He as carrier gas (1 ml/min) and FID or 70 eV EIMS detection. Thirty-six analytes were identified by the two detection methods. The RSD (n = 6) for 14 compounds were 6.39-24.07%. Cryotrapping-headspace SPME showed a loss of sensitivity compared with the adopted method.

Record - 137

TI- Determination of polar pesticides in soil by solid-phase microextraction coupled to high-performance liquid chromatography-electrospray mass spectrometry.

AU- Moder, M;Popp, P;Eisert, R;Pawliszyn, J

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 7

PG- 680-685

AB- Air-dried soil (90 g) was extracted with dilute HNO₃ (900 ml) at pH 4, by shaking for 24 h. To a portion (4 ml) of the eluate, NaCl (1 g) was added and the mixture extracted with a polyacrylate coated fibre. The fibre was transferred to an HPLC interface and the analytes were

desorbed with methanol (50 µl). Separation was effected on a Supelcosil LC-18 column (15 cm * 2.1 mm i.d.) with H₂O/methanol gradient (details given) as mobile phase (0.2 ml/min). Identification was by MS, equipped with electrospray interface and operated in the selected-ion monitoring mode. Detection for 13 pesticides and herbicides ranged from 0.1-50 ng/ml. Calibration graphs for all analytes were linear up to 100 ng/ml.

Record - 138

TI- Determination of organophosphorus pesticides in soil by headspace solid-phase microextraction.

AU- Ng, WF;Teo, MJK;Lakso, HA

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 7

PG- 673-679

AB- Humic material and soil were dried at 60degC and sieved to pass through a 2 mm sieve. Portions (3.5 g) were placed into headspace vials, fitted with PTFE/silicone septum. A polyacrylate coated SPME fibre was inserted through the septum and exposed to the headspace without touching the sample. Extraction was effected at 80degC for 1 h. The analytes were desorbed at 250degC for 2 min and analysed by GC on a DB-5MS column (30 m * 0.25 mm i.d.) operated with temperature programming (20degC/min) from 40degC (held for 2 min) to 260degC (held for 4 min), with He as carrier gas (1 ml/min) and FID. Detection limits were 143 ng/g for malathion and parathion, and 28.6 ng/g for phorate, diazinon and disulfoton. Sensitivity was significantly improved by use of MS detection. Calibration graphs for all pesticides were linear for 143 ng/g to 28.6 µg/g.

Record - 139

TI- Solid-phase microextraction of the antifouling Irgarol 1051 and the fungicides dichlofluanid and 4-chloro-3-methylphenol in water samples.

AU- Penalver, A;Pocurull, E;Borrull, F;Marce, RM

JN- Journal of Chromatography, A

PY- 1999

VO- 839

NO- 1-2

PG- 253-260

AB- Dichlofluanid (I), 4-chloro-m-cresol (II) and Irgarol 1051 (III), pesticide additives to antifouling paint formulations, were sampled from waters by SPME and determined by GC-MS. For sampling 85-µm polyacrylate-coated fibres (Supelco) were used for collection of

added amounts from 3-ml samples containing 18% added NaCl in 4-ml phials by immersion for 1 h at 60degC. The analytes were desorbed from the fibre in the chromatograph injection port by heating at 250degC for 2-3 min. The desorbed analytes were analysed on a fused-silica column (30 m, no i.d. given) coated with HP-1 and temperature-programmed from 80degC at 35deg/min to 250degC (held for 8 min), and 70-eV EIMS detection operated in selected-ion monitoring mode (m/z listed for each analyte). A linear response was obtained from 0.2-10 mug/l for each analyte; at 0.5 mug/l, RSD (n = 5) were 18, 12 and 10%, respectively, for I, II and III. A concentration of 0.3 mug/l of III was found in a seawater sample.

Record - 140

TI- Headspace solid-phase microextraction for the determination of trace levels of taste and odour compounds in water samples.

AU- Bao, M;Griffini, O;Burrini, D;Santianni, D;Barbieri, K;Mascini, M

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 4

PG- 459-466

AB- Aqueous sample (40 ml) was mixed with NaCl (30%) in a 62 ml glass vial. The solution was stirred for 10 min after which a 65 mum polydimethyl-siloxane/divinylbenzene SPME fibre was exposed to the headspace above the solution for 40 min. The analytes were desorbed from the fibre at 240degC for 3 min for determination by GC on a 0.25 mum DB-5 fused-silica column (30 m * 0.25 mm i.d.) operated with temperature programming from 40degC (held for 1 min) to 130degC at 4degC/min, then to 280degC at 10degC/min, He as the carrier gas (flow rate not given) and ion-trap MS detection (details given). The standard additions method was used for quantification. The calibration graphs were linear over two orders of magnitude for the 34 taste and odour-causing compounds (alcohols, aldehydes, aliphatic hydrocarbons and ketones) studied. The results are tabulated. The detection limits were 0.5-50 ng/l and the RSD (n = 4) were 4.3-17.2%. The method was applied to river water.

Record - 141

TI- Application of solid-phase microextraction to monitoring indoor air quality.

AU- Gorlo, D;Zygmunt, B;Dudek, M;Jaszek, A;Pilarczyk, M;Namiesnik, J

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 7

PG- 696-699

AB- A SPME fibre coated with polydimethylsiloxane was exposed for 15 min in the room under investigation, at a height of 1-1.5 m above the floor. The fibre was inserted in the desorption chamber of a GC-MS instrument for desorption at 250degC for 40 s. Separation was effected on a column (30 m * 0.32 mm i.d.) coated with DB5-MS operated with temperature programming from 40degC (held for 2 min) to 50degC at 5degC/min and then to 230degC (held for 5 min) at 15degC/min, with He as carrier gas (1.17 ml/min). Detection was by MS (70 eV). Results for CCl4, benzene, toluene, chlorobenzene, p-xylene and n-decane were reported for 16 domestic rooms and two chemical laboratories.

Record - 142

TI- On calibration of solid-phase microextraction-gas chromatography-mass spectrometry system for analysis of organic air contaminants using gaseous standard mixtures.

AU- Namiesnik, J;Gorlo, D;Wolska, L;Zygmunt, B

JN- Chemia Analityczna (Warsaw)

PY- 1999

VO- 44

NO- 2

PG- 201-213

AB- The problems associated with instrumental calibration for the SPME-GC-MS of organic pollutants in air are explored. An apparatus and method for the dynamic generation of standard gas mixtures for calibration purposes is presented. Instrumental parameters such as analyte permeation rate and concentration, humidity and temperature control, static and dynamic SPME sampling are discussed. Model calibration graphs for the SPME-GC-MS response for the trace analysis of chlorobenzene and p-xylene are linear up to 40 mg/m³ and 3.5 mg/m³ respectively.

Record - 143

TI- Solid-phase microextraction for cannabinoid analysis in hair and its possible application to other drugs.

AU- StranoRossi, S;Chiarotti, M

JN- Journal of Analytical Toxicology

PY- 1999

VO- 23

NO- 1

PG- 7-10

AB- Washed hair (50 mg) containing 50 ng Delta9-tetrahydrocannabinol-d3 (internal standard) was mixed with 200 μ l 1M-NaOH and incubated for 10 min at 90degC. The mixture was cooled, neutralized with 6M-HCl and

200µl phosphate buffer of pH 7.5 and a portion of the resulting solution was subjected to SPME by immersion of a 30 mm polydimethylsiloxane fibre for 15 min. The fibre was then heated at 260degC for 2 min in a GC injection port and the desorbed analytes were separated on a fused-silica column (12 m * 0.32 mm i.d.) coated with HP-5 (0.33 µm) and operated with temperature programming from 100degC (held for 2 min) to 200degC at 50degC/min and to 270degC (held for 5 min) at 15degC/min and MS detection in the selected ion monitoring mode. The GC carrier gas is not stated. The detection limits were 0.1 ng/mg for Delta9-tetrahydrocannabinol (dronabinol) and cannabinol and 0.2 ng/mg for cannabidiol. RSD are not given. The method was also tested on cocaine and methadone and chromatograms are presented.

Record - 144

TI- Extraction of methylxanthines from human body fluids by solid-phase microextraction.

AU- Kumazawa, T;Seno, H;Lee, XP;Ishii, A;WatanabeSuzuki, K;Sato, K; Suzuki, O

JN- Analytica Chimica Acta

PY- 1999

VO- 387

NO- 1

PG- 53-60

AB- Caffeine, theobromine, paraxanthine and theophylline were extracted from whole blood and urine by micro-SPE using a carbowax/divinylbenzene-coated (CW/DVB) fibre. A 1 ml blood sample was deproteinized with 1 ml 1M-HClO₄. The clear solution was adjusted to pH 7 with KOH and transferred to a 2 ml screw-cap vial. After sealing, the vial was heated to 40degC and the CW/DVB fibre was inserted via the silicone septum. At the end of the 1 h extraction period the fibre was removed and inserted in the injection port of the GC for analysis. Urine was treated in a similar manner except the deproteinization step was omitted. The analysis was carried out on a DB-17 column (30 m * 0.32 mm i.d., 0.25 µm film thickness) with temperature programming from 140degC (held for 1 min) to 280degC at 10degC/min, with He carrier gas (3 ml/min) and N-P detection. The extraction efficiencies were 0.01-0.097% from whole blood and 0.05-0.299% from urine and the within-day RSD (n = 3-4) were <9.3%. Calibration graphs were linear for 1.88-60 µg/ml in blood and urine for all analytes apart from caffeine (0.31-20 µg/ml in blood, 0.16-10 µg/ml in urine). Detection limits were 0.2-0.9 µg/ml in blood and 0.06-0.7 µg/ml in urine. The method was applied to study methylxanthine concentrations in male subjects following ingestion of coffee and cocoa.

Record - 145

TI- Solid-phase microextraction and GC-ECD of benzophenones for detection of benzodiazepines in urine.

AU- Guan, F;Seno, H;Ishii, A;Watanabe, K;Kumazawa, T;Hattori, H;Suzuki, O

JN- Journal of Analytical Toxicology

PY- 1999

VO- 23

NO- 1

PG- 54-61

AB- Urine (1 ml) was mixed with 1 ml 8M-HCl and the mixture was heated at 100degC for 40 min. After cooling, 0.95 ml 8M-KOH and 0.4 ml 6M-NaH₂BO₃ were added and the pH was adjusted to 9.4. The resulting solution was subjected to SPME by immersion of a polydimethylsiloxane fibre for 30 min. The fibre was heated at 270degC in a GC injector port and the desorbed analytes were separated on a column (30 m * 0.32 mm i.d.) coated with DB-17 (0.25µm) and operated with temperature programming from 150degC (held for 1 min) to 230degC (held for 5 min) at 10degC/min and to 300degC (held for 9 min) at 10degC/min, He as carrier gas and ECD. The calibration graphs were linear up to 500 ng/ml for oxazolam, haloxazolam, flunitrazepam, nimetazepam and clonazepam and up to 1000 ng/ml for diazepam, bromazepam, fludiazepam and nitrazepam. The detection limit for the benzodiazepines were 2-20 ng/ml, with the exception of bromazepam (80 ng/ml). Recoveries from SPME were 1-25% (with RSD of 0.1-16%), compared to recoveries of 24-90% by solvent extraction with ethyl ether, but the chromatographic response was better than that obtained with solvent-extracted samples. The detection limits for solvent-extracted samples were 80-160 ng/ml.

Record - 146

TI- Determination of homocysteine and its related compounds by solid-phase microextraction-gas chromatography-mass spectrometry.

AU- Myung, SW;Kim, MS;Min, HK;Yoo, EA;Kim, KR

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 727

NO- 1-2

PG- 1-8

AB- Plasma (0.5 ml) was diluted with 0.5 ml H₂O and heated with 0.1 ml 12.5% DTT at 40degC for 30 min. The proteins were precipitated with 0.1 ml 72% TCA and the mixture was centrifuged. A portion (0.6 ml) of the supernatant was mixed with 0.4 ml pyridine/propan-1-ol (1:4) and 50 µl ethyl chloroformate for 3 min. Portions (2 ml) of H₂O were added, the pH was adjusted to 3 and an 85 µm poly(acrylate) SPME fibre was immersed in the solution contained in a 4 ml phial. The

solution was stirred for 30 min, the fibre was transferred to the GC injection port and heated at 240degC for 0.5 min. The desorbed sulfur-containing amino-acids, homocysteine (Hom), cysteine (Cys) and methionine (Met), were analysed directly by GC on a fused-silica column (16 m * 0.2 mm i.d.) coated with HP-1 (0.11 µm), operated with temperature programming from 100degC (held for 0.5 min) to 300degC (held for 3 min) at 10degC/min, with He as carrier gas (0.9 ml/min) and 70 eV EIMS detection operated in selected-ion monitoring mode at m/z 56, 128 and 189, 74, 146 and 220 and 61, 129 and 189, respectively, for Hom, Cys and Met. Calibration graphs were linear from 5-50 and 40-400µM, respectively, for Hom and Cys and for Met, with detection limits of < 5µM for all 3 analytes.

Record - 147

TI- Solid-phase microextraction

AU- Prosen, H;ZupancicKralj, L

JN- Trends in Analytical Chemistry

PY- 1999

VO- 18

NO- 4

PG- 272-282

AB- An overview is presented of SPME which covers the theory of the technique and conditions affecting SPME fibre performance with particular reference to the choice of stationary phase, extraction conditions, extraction from solid samples, influences on desorption from the SPME fibre, and possible problems with SPME. SPME applications are briefly discussed. (27 references).

Record - 148

TI- Time weighted average sampling with solid phase microextraction device: implications for enhanced personal exposure monitoring to airborne pollutants.

AU- Martos, PA;Pawliszyn, J

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 8

PG- 1513-1520

AB- A mixture of 34 µg/l each of 11 aromatic and aliphatic hydrocarbons (listed) was time weighted average (TWA) sampled with a 1 cm fused silica fibre coated with an absorptive film (100 µm) of poly(dimethylsiloxane) (PDMS) and formaldehyde vapour derivatized with O-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine adsorbed on a fibre coated with PDMS/divinylbenzene. The fibre was retracted, e.g., 3 mm within a stainless steel needle (illustrated). TWA sampling for

various analytes was possible with times of 15 min to at least 16 h, dependent on the sorbent used. The theoretical and practical considerations associated with a solid phase micro-extraction device used as a TWA sampler were discussed.

Record - 149

TI- Headspace solid-phase micro-extraction of sulfides and disulfides using Carboxen-poly(dimethylsiloxane) fibres in the analysis of wine aroma.

AU- Mestres, M;Sala, C;Marti, MP;Busto, O;Guasch, J

JN- Journal of Chromatography, A

PY- 1999

VO- 835

NO- 1-2

PG- 137-144

AB- Wine (25 ml) was mixed in a vial at 4degC with 2.92 g of NaCl, 0.15 g of EDTA and the internal standards ethylmethyl sulfide and thiophene (final concentrations 10 and 5 mug/l respectively). The vial was capped, shaken and equilibrated at 25degC for 30 min. A Carboxen-poly(dimethylsiloxane) fibre (75 um) was introduced through the septum and exposed to the headspace gas for 30 min. It was transferred to the injection port of a GC instrument for thermal desorption at 300degC for 1 min. The sulfides were determined on a column (30 m * 0.32 mm i.d.) coated with SPB-1 (4 um) and operated with He (1.2 ml/min) as carrier gas, temperature programming (50degC for 8 min, to 150degC at 15degC/min, to 280degC at 40degC/min, held for 5 min) and flame photometric detection in sulfur mode. Results on actual samples were also confirmed on a HP-Innowax column. Log.-log. calibration graphs obtained in a synthetic matrix were linear for 0.25-80 mug/l of dimethyl sulfide, diethyl sulfide, methylpropyl sulfide, methyl thioacetate and ethyl thioacetate and for 0.125-40 mug/l of CS₂, dimethyl disulfide and diethyl sulfide, with detection limits of 0.03-1 ng/l. Recoveries of 0.25-25 mug/l from red and white wines by a standard addition method were 80-125%, but RSD for some compounds were high, at up to 30%

Record - 150

TI- Changes in odour of Bartlett pear brandy influenced by sunlight irradiation.

AU- KraljCigic, I;ZupancicKralj, L

JN- Chemosphere

PY- 1999

VO- 38

NO- 6

PG- 1299-1303

AB- A Supelco SPME polydimethylsiloxane fibre (100 µm film thickness) was placed over 5 ml pear brandy at 40degC for 15 min. The analytes were desorbed from the fibre in the GC injector at 220degC then determined by GC on a Supelco VOCOL fused-silica column (60 m * 0.25 mm i.d.; 1.5 µm film thickness) operated with temperature programming from 70degC (held for 4 min) to 210degC at 15degC/min and EIMS detection; no details are given of the carrier gas used. The major aroma compounds found in pear brandy stored in green bottles were ethyl trans-2-cis-4-decadienoate (68 ppm), ethyl trans-2-trans-4-decadienoate (21 ppm) and methyl trans-2-cis-4-decadienoate (27 ppm). In pear brandy kept in colourless bottles, the concentration of trans-2-cis-4-isomers was lower and the concentrations of other isomers were higher.

SPME References 1999 – 2000 Part B

Record - 1

TI- Microwave-mediated distillation with solid-phase microextraction: determination of off-flavors, geosmin and methylisoborneol, in catfish tissue.

AU- Zhu, M;Aviles, FJ;Conte, ED;Miller, DW;Perschbacher, PW

JN- Journal of Chromatography, A

PY- 1999

VO- 833

NO- 2

PG- 223-230

AB- Catfish tissue (10 g), spiked with 1 ppb cis-decahydro-1-naphthol (internal standard, IS), was heated at 120degC for 6 min in a microwave mediated distillation unit (construction details given). The volatiles produced were purged in a flow of Ar (25 ml/min), passed through a condenser at 5degC and the distillate was collected in a 10 ml glass phial. NaCl (2 g) was added to the distillate and the analytes sampled for 25 min with stirring using a polydimethylsiloxane SPME fibre. The fibre was washed with H₂O (to remove excess NaCl) and mounted in the programmable injector of a Varian model 3400 GC (Varian Inc., Walnut Creek, CA, USA). The injector was heated at 250degC and the desorbed volatiles were separated and analysed by GC on a fused-silica column (30 m * 0.25 mm i.d.) coated with DB5MS (0.25 µm) and operated with temperature programming from 60degC (held for 4 min) to 200degC (held for 16.5 min) at 8.5degC and to 250degC (held for 3 min) at 20degC/min and ion-trap EIMS detection operated in selected-ion monitoring mode at m/z 95, 112 and 136 for methylisoborneol (I), geosmin (II) and IS, respectively. Detection limits were 0.043 ppb for I and 0.008 ppb for II, values well below the human threshold for these off-flavour

compounds. Mean recoveries (n = 6) were 81.4 +- 5.4% for I, 30.4 +- 5.3% for II and 42.9 +- 6.6% for III.

Record - 2

TI- Headspace solid-phase microextraction (HSSPME) for the determination of volatile and semivolatile pollutants in soils.

AU- Llompart, M;Li, K;Fingas, M

JN- Talanta

PY- 1999

VO- 48

NO- 2

PG- 451-459

AB- For VOC determination, soil (1 g) was mixed with 1 ml H₂O in a 22 ml vial. After sealing the vial, a 100 µm polydimethylsiloxane SPME fibre was suspended in the headspace above the stirred mixture at 20degC for 30 min. The VOC were desorbed from the fibre at 260degC for 3 min for quantification by GC on a 1.5 µm SPB-1 column (30 m * 0.53 mm i.d.), operated with temperature programming from 40degC (held for 5 min) to 200degC at 7.5degC/min, He as carrier gas (80 ml/min) and mass-selective detection in the selected ion monitoring mode. The calibration graphs were linear up to 2 µg/g for all the VOC studied, the detection limits were 0.05-0.23 ng/g and the within- and between-day RSD (n = 5) were 1-5% and 3-10%, respectively. Attempts to apply the method to the determination of semi-volatile organic compounds (e.g. PAH) in soil were not successful.

Record - 3

TI- Determination of 2,4-dinitrophenol by solid-phase microextraction coupled to GC-MS.

AU- Lu, X;Zhao, XJ;Ye, F;Xu, GW

JN- Sepu

PY- 1999

VO- 17

NO- 2

PG- 131-133

AB- Sample (15 ml), adjusted to pH 2, was saturated with NaCl and a 85 µm polyacrylate-coated fibre probe was introduced into the solution for solid SPME while maintaining constant stirring of the solution. The probe was removed and 2,4-dinitrophenol (I) was desorbed from the probe, by heating at 270degC for 3 min, directly onto a fused-silica GC column (30 m * 0.25 mm i.d.) coated DB-5 ms (0.25 µm), operated with temperature programming from 60degC (held for 4 min) to 260degC at 8degC/min, with He as carrier gas (flow-rate not given) and EIMS detection operated in full-scan mode from 35-350 amu. The calibration graph was linear up to 50 mg/l of I. There was no need

for preconcentration and pretreatment. The method was applied to waste water.

Record - 4

TI- Solid-phase microextraction and GC-MIP-AED for the speciation analysis of organomercury compounds.

AU- Mothes, S; Wennrich, R

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 3

PG- 181-182

AB- Organomercury compounds were extracted from waste water using a SPME fibre in the direct or headspace sampling mode, the fibre was transferred directly into a GC injection port, held at 150degC for 1 min, and the desorbed analytes were separated on a fused silica column (25 m * 0.32 mm i.d.) coated with HP-1 (0.25 µm) and operated with helium as carrier gas (flow-rate not given), temperature programming from 40degC (held for 5 min) to 200degC at 30degC/min and MIP AES detection at 185 and 254 nm. Soil samples were extracted by headspace SPME. The limits of detection for direct SPME extraction were 144 µg/ml Hg for Hg(CH₃)₂ and 30 µg/ml Hg for Hg(CH₂CH₃)₂. RSD were 15%.

Record - 5

TI- Solid-phase microextraction-capillary GC determination of chlorobenzene compounds in water.

AU- Yang, HB

JN- Lihua Jianyan, Huaxue Fence

PY- 1999

VO- 35

NO- 3

PG- 103-105

AB- Sample (1.5 ml) was treated with 0.5 g NaOH and an SPME set-up with a probe coated with 100 µm polydimethylsiloxane was submerged into the solution for agitation for 5 min. The probe was taken off and applied to the sample inlet of a HP6890 gas chromatograph, the inlet was heated at 260degC for 1 min and the chlorobenzenes (CB) were desorbed directly onto a fused silica capillary GC column (30 m * 0.32 mm i.d.) coated with HP-5 (0.25 µm). The column was temperature programmed from 50-85degC at 5degC/min and then to 180degC (held for 2 min) at 25degC/min, with N₂ as carrier gas (60 ml/min) and ECD. The procedure took only 20 min. Chlorobenzenes, viz., o-, m- and p-dichlorobenzenes, 1,2,4-trichlorobenzene and 1,2,3,4-tetrachlorobenzenes were well separated. Calibration graphs were

linear from 0.1-10 µg/l for all CB, with detection limits of 0.1-2 µg/l. The method was applied to river water and reservoir water, with recoveries of 101.5-106.7%.

Record - 6

TI- Determination of hydroxyaromatic compounds in water by solid-phase micro-extraction coupled to high-performance liquid chromatography.

AU- Wu, YC;Huang, SD

JN- Journal of Chromatography, A

PY- 1999

VO- 835

NO- 1-2

PG- 127-135

AB- Samples of 3 ml, in which 20% of Na₂SO₄ had been dissolved, were transferred to 4 ml phials at 25degC and were stirred at 550 rpm for 20 min in the presence of a poly(dimethylsiloxane) fibre (60 µm). The fibre was transferred to a desorption chamber, solutes were desorbed with the mobile phase in static mode for 3 min, then the mobile phase flowed at 0.1 ml/min to a HPLC column for a further 3 min, after which the flow rate was increased to 1 ml/min. The HPLC column was a 4 µm Nova-Pak Phenyl column (7.5 cm * 3.9 mm i.d.) operated with a mobile phase of acetonitrile/0.1M-acetate buffer of pH 4.7 (11:39) and detection at 254 nm. During the analysis the fibre was further washed with mobile phase before removal for reuse. Calibration graphs were linear for 0.01-5 mg/l of 2,7-dihydroxynaphthalene and 1-hydroxy- and 2-hydroxynaphthalene and for 2 µg/l to 1 mg/l of 4,4'-dihydroxybiphenyl and 5-hydroxy-1,4-naphthoquinone, with detection limits of 1-4.2 µg/l. Since the actual recoveries of the compounds under the given conditions were only 1-4%, either better SPME fibres or more sensitive detection methods are required for the analysis of natural waters.

Record - 7

TI- Partition infrared method for total gasoline range organics in water based on solid phase microextraction.

AU- Stahl, DC;Tilotta, DC

JN- Environmental Science and Technology

PY- 1999

VO- 33

NO- 5

PG- 814-819

AB- Organic compounds were extracted from 250 ml H₂O into a PTFE PFA film (3.2 cm * 3.2 cm * 130 µm) which lacked C-H bonds and therefore enabled quantitation of organic compounds by the C-H stretching vibrations (details given). Detection limits were 0.5-1.5 ppm with

RSD of 6-11%. Linear ranges of calibration graphs extended to the water solubility limits of all fuels studied. The procedure was applied to the analysis of waste water containing gasoline. Results compared well with those obtained using a purge-and-trap GC procedure.

Record - 8

TI- Method for analyzing urinary toluene and xylene by solid-phase microextraction (SPME), and its application to workers using organic solvents.

AU- Asakawa, F;Jitsunari, F;Choi, J;Suna, S;Takeda, N;Kitamado, T

JN- Bulletin of Environmental Contamination and Toxicology

PY- 1999

VO- 62

NO- 2

PG- 109-116

AB- Urine samples (5 ml) were mixed with methanol (50 μ l) and NaCl (1 g) for 1 h. A polydimethylsiloxane SPME fibre (film thickness 100 μ m) was inserted into the headspace of the vial containing the mixture and extraction of toluene and xylenes was performed for 5 min while the sample solution was stirred. For quantitative analysis by GC, the SPME fibre was injected into a Shimadzu GC-8A column (30 m * 0.53 mm; film thickness 1.5 μ m) equipped with a FID at 150degC. The carrier gas was He (10 ml/min). The calibration graphs for both toluene and xylene were linear up to a urinary concentration of 100 μ g/l. The RSD for toluene, p-xylene, m-xylene and o-xylene were 1.7%, 4.5%, 3% and 3.5%, respectively. The method is applicable to the monitoring of the toluene and xylene content of urine from workers exposed to organic solvents. The detection limit was at least 0.5 μ g/l with a signal-to-noise ratio of 6:1.

Record - 9

TI- An introduction to solvent-free (SPME) technique.

AU- Hu, KC;Xing, GQ;Liang, HC

JN- Sepu

PY- 1999

VO- 17

NO- 2

PG- 171-174

AB- An overview of the theory of SPME, as well as its operating models including instrumentation and factors affecting its sensitivity is presented. Applications particularly in sample preparation of gas, liquid and biological materials are given, together with a discussion on its future prospects. (19 references).

Record - 10

TI- Solid-phase micro-extraction (SPME) - a universal sample preparation technique?

AU- James, AD;Mills, GA

JN- CAST, Chromatography and Separation Technology

PY- 1999

NO- 6

PG- 8-12

AB- A brief overview is given which covers the theory of SPME, optimization of experimental conditions, derivatisation methods (with particular reference to subsequent GC detection), applications and future developments.

Record - 11

TI- Analysis of diacetyl in wine using solid-phase microextraction combined with gas chromatography-mass spectrometry.

AU- Hayasaka, Y;Bartowsky, EJ

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 2

PG- 612-617

AB- SPME fibre was exposed to the headspace of a 3 ml wine sample. The analytes adsorbed on the SPME fibre were thermally desorbed into the GC port and were separated on a 1.2 µm Chrompack WCOT FS CP-Wax 57 CB column (50 m * 0.32 mm i.d.) operated with temperature programming from 60degC (held for 1 min) to 110degC at 5degC/min, then to 180degC (held for 10 min) at 20degC/min, with He as carrier gas (14 psi) and 70 eV EIMS detection. [2H6]diacetyl was used as the internal standard for quantitation. Selected-ion monitoring was performed at m/z 86 for diacetyl (biacetyl) and m/z 92 for deuterated internal standard. The calibration graph was linear up to 10 µg/ml of diacetyl. The detection limit was 0.01 µg/ml. SO₂ did not interfere with the determination.

Record - 12

TI- Validation of a solid-phase micro-extraction method for the determination of organophosphorus pesticides in fruits and fruit juice.

AU- Simplicio, AL;VilasBoas, L

JN- Journal of Chromatography, A

PY- 1999

VO- 833

NO- 1

PG- 35-42

AB- Fruit juices (20 g) were diluted 1 + 99 and centrifuged. Fruit (20 g) was chopped, homogenized with 60 ml H₂O and a 4 ml portion of the homogenate was diluted to 100 ml and centrifuged. Portions (3 ml) of supernatant were stirred at 1 250 rpm for 3 min with a poly(dimethylsiloxane) fibre (100 µm) immersed in the sample. The fibre was transferred to the GC injection port for desorption at 250degC for 2 min. The desorbed organophosphorus pesticides (OPP) were analysed on a fused-silica column (15 m * 0.32 mm i.d.) coated with DB-1 (0.25 µm), operated with temperature programming (100-230degC at 20degC/min, held for 1 min) with H₂ as carrier gas (flow rate not given) and flame photometric detection in phosphorus mode. Calibration graphs were linear from 0.25-25 µg/l of for 7 OPP, viz. diazinon, fenitrothion, fenthion, quinalphos, triazophos, phosalone and pyrazophos, with detection limits (in the final solution) of 3-14 ng/l. The dilution of the sample was required to overcome matrix interferences. Recoveries of 25-250 ppb of OPP added to the original samples were 75.9-102.6% for juice and 70-99% for pears, except for pyrazophos in fruit (53%). At 25 ppb, the intra-assay RSD (n = 3) were 1.7-8.7%.

Record - 13

TI- Volatile reduced sulfur compounds in butter by solid phase microextraction.

AU- Shooter, D;Jayatissa, N;Renner, N

JN- Journal of Dairy Research

PY- 1999

VO- 66

NO- 1

PG- 115-123

AB- Butter samples (10 g) were heated to 30-35degC in a silanized glass vial (44.3 ml) capped with a Teflon-faced silicone rubber septum and magnetically stirred for 30 min. A polyacrylate SPME fibre was exposed to the vial headspace for 10 min. The adsorbed volatiles were desorbed in the injection port of a GC-MS system comprising a Hewlett-Packard G1800A GCD system (gas chromatograph and electron ionization detector) with a split-splitless injector used in splitless mode and fitted with a SPME injection port liner (0.75 mm i.d.). The system included a HP1 capillary column (30 m * 0.32 mm i.d.; film thickness 4 µm), an effluent splitter kit and He as carrier gas (1.6 ml/min), with temperature programming from 70degC (held for 2 min) to 150degC (at 10degC/min). The injector and detector temperatures were 200 and 275degC, respectively. The mass range scanned was 30-200 amu. The concentrations of dimethyl disulfide (I) and methanethiol (II) in the butter headspaces of five samples were 0.04-1.2 and 0.1-0.22 µg/ml, respectively.

Concentrations of dimethylsulfide (III) were below the detection limits of the system (0.01, 0.01 and 0.1 mug/l for I, II and III, respectively). The concentrations of I and II in the butter itself were calculated to be 0.27-7.4 and 0.27-1.3 mug/g, respectively, using the distribution coefficient between headspace and butter which was determined using spiked samples (details given). Seasonal variations in the concentrations of I and II are discussed.

Record - 14

TI- SPME-MS-MVA as an electronic nose for the study of off-flavors in milk.

AU- Marsili, RT

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 2

PG- 648-654

AB- SPME combined with MS and multivariate analysis (MVA) was used to study off-flavours in milk. The analytical column of a GC-MS instrument was replaced with a 1 m deactivated fused-silica column to serve as a transfer line between the Carboxen SPME fibre and the mass spectrometer. Samples of reduced fat milk were abused by light, heat, Cu and microbial contamination by *Pseudomonas fluorescens*, *Pseudomonas aureofaciens* and *Pseudomonas putrefaciens*. Milk (3 g) was stirred with 7 μ l internal standard (20 mug/ml; 4-methyl-2-pentanone) in a glass vial. The vial was placed in a 45degC water bath and exposed to the SPME fibre (75 μ m) for 12 min. The analytes were thermally desorbed for 2 min at 250degC and transferred to the mass spectrometer using He as carrier gas and transfer-line temperature of 50degC. EIMS was performed with scanning from m/z 50-150 and an ion trap manifold temperature of 170degC. Mass fragmentation data were subjected to multivariate analysis. Different types of abuse were differentiated by visual examination of class clustering in 2D principal-component analysis plots. The method should be more useful than current commercial electronic noses for quality assurance.

Record - 15

TI- Analysis of volatile fatty acids in waste water collected from a pig farm by a solid-phase micro-extraction method.

AU- Yo, SP

JN- Chemosphere

PY- 1999

VO- 38

NO- 4

PG- 823-834

AB- A Supelco carbowax/divinylbenzene coated fibre was placed in 2 ml waste water for 20 min for SPME with continuous stirring. The fibre was then introduced into the heated chamber of a GC injection port, set at 250degC, for thermal desorption for 3 min. The desorbed volatile fatty acids were then quantified using a Restek Stablewax DA fused-silica column (30 m * 0.25 mm) operated with temperature programming from 100degC (held for 2 min) to 250degC (held for 9.25 min) at 8degC/min and 70 eV EIMS detection with scanning from m/z 30-500 every 2 s. The detection limits were 16, 28, 17, 14, 16, 13, 13, 12, 12 and 10nM for acetic, formic, propanoic, isobutyric, butanoic, isovaleric, n-valeric, isocaproic, n-caproic and heptanoic acid; corresponding RSD were 1.55, 17.59, 1.76, 2.64, 2.96, 4.03, 8.68, 9.02, 12.13 and 11.85%. Recoveries were 80-120%.

Record - 16

TI- Solid-phase microextraction of volatile polar compounds in water.

AU- Matisova, E;Sedlakova, J;Slezackova, M;Welsch, T

JN- Journal of High Resolution Chromatography

PY- 1999

VO- 22

NO- 2

PG- 109-115

AB- Volatile organic compounds (low molecular weight alcohols and esters) were analysed using solid phase micro-extraction (SPME) with both direct sampling on a 85 µm polyacrylate fiber, and headspace sampling SPME at 50degC followed by analysis on a fused-silica column (60 m * 0.53 mm i.d.) coated with SPB-5 (5 µm) with a pre-column (1 m * 0.53 mm i.d.). The fibers were desorbed in the GC inlet in the splitless mode at 250degC. At the onset of fiber desorption the column temperature was 35degC (held for 3 min), then increased to 184degC (held for 1 min) at 8degC/min, with H₂ as carrier gas (30 cm/s) and FID. The direct sampling SPME method was only recommended for clean water samples. Headspace SPME was useful for ppm concentrations of samples with a complex matrix. Alcohols in waste water and in beer gave RSD of 5.3-7.1 and 2.7-8.0%, respectively at the low ppm levels.

Record - 17

TI- Solubility and partitioning studies with polycyclic aromatic hydrocarbons using an optimized SPME procedure.

AU- Paschke, A;Popp, P;Schueerman, G

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 4

PG- 426-428

AB- Water or octanol-saturated water (4 ml) was continuously magnetically stirred with a glass-coated impeller for 60 min and subjected to SPME with a 100 µm polydimethylsiloxane fibre. The fibre was inserted into a GC injection port and held at 250degC for 2 min. The analytes were separated on a column (25 m * 0.32 mm i.d.) coated with CP-SIL 8 CB (0.25 µm) and operated with temperature programming (details given) and FID. Alternatively, a column (30 m * 0.32 mm i.d.) coated with HP 5 (0.25 µm) was used, also operated with temperature programming (details given) and MS detection. The detection limit for fluoranthene in octanol-saturated water was 7.6 µg/l, obtained using GC with FID. The detection limits for fluoranthene, phenanthrene, pyrene, benz[a]anthracene and benz[a]pyrene in octanol-saturated water were 122, 106, 147, 88 and 15 ng/l, respectively, obtained using GC-MS. The SPME procedure was used in the measurement of the water solubilities and octanol/water partition coefficients of PAH.

Record - 18

TI- Determination of benzodiazepines in human urine and plasma with solvent modified solid-phase micro-extraction and gas chromatography; rationalization of method development using experimental design strategies.

AU- Reubsæet, KJ;Norli, HR;Hemmersbach, P;Rasmussen, KE

JN- Journal of Pharmaceutical and Biomedical Analysis

PY- 1998

VO- 18

NO- 4-5

PG- 667-680

AB- SPME and GC were used for the determination of oxazepam, diazepam, nordiazepam, flunitrazepam, alprazolam and prazepam in urine and plasma. Several factors likely to affect the analyte recovery were screened in a fractional factorial design in order to examine their effect on the extraction recovery. Parameters found effective in the screening were further investigated with the use of surface response methodology. Final conditions for extraction were as follows: octanol was immobilized on a polyacrylate fibre for 4 min. The fibre was placed in the sample and extraction took place at pH 6 for 15 min. Urine samples were added to 0.3 g/ml NaCl. In plasma, release of the drugs from proteins using 1M-HCl in glycerol, followed by protein precipitation with trichloroacetic acid was performed before sampling. Fibres were desorbed in the GC injector for 1 min at 300degC. GC was performed on a CPSIL 8 CB column (25 m * 0.25 mm i.d.; 0.25 µm) operated with temperature programming from 150-230degC (held for 2 min) at 40degC/min then to 250degC (held for 1 min) at 5degC/min then to 300degC (held for 3 min) at 15degC/min and N-P detection. Calibration graphs were linear from 0.1-3µM and

detection limits were 0.01-0.48µM (details given). Within- and between-day RSD are tabulated.

Record - 19

TI- Residual solvents determination in pharmaceutical products by GS-headspace analysis (GC-HS) and GC-MS-SPME.

AU- Camarasu, CC;MezeiSzuts, M;BertokVarga, G

JN- Journal of Pharmaceutical and Biomedical Analysis

PY- 1998

VO- 18

NO- 4-5

PG- 623-628

AB- Headspace SPME using three fibres with different polymer films was compared with gas-tight SPME (diagram of device shown) and headspace sampling (HS). Of the different polymer films, the polydimethylsiloxane/divinylbenzene coated fibre was the most sensitive for the solvents tested. GC was performed with a 5 µm CP-SIL 5CB column (50 m * 0.32 mm i.d.) operated with temperature programming from 40degC (held for 1 min) at 3-130degC/min to 180degC (held for 15 min). For headspace GC the carrier gas was H₂ (30 cm/s) and FID, and for GC-MS the carrier gas was He (35 cm/s) with 70 eV EIMS detection. Results indicated that gas-tight-SPME was the most sensitive, with detection limits of 5 pg/ml to 2 ng/ml. Headspace SPME was more precise with RSD of 2-3%. Validation data are tabulated. Compared with the static headspace technique, both SPME methods showed superior results.

Record - 20

TI- Quantitative determination of trimethylamine in urine by solid-phase micro-extraction and gas chromatography-mass spectrometry.

AU- Mills, GA;Walker, V;Mughal, H

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 723

NO- 1-2

PG- 281-285

AB- Urine (2 ml) was mixed with 3 ml acidified water, 2 g K₂CO₃, 1 ml aqueous 56.8 µM-deuterated trimethylamine (internal standard, IS) and six pellets KOH in 22 ml headspace phials. The sealed phials were immersed in a water bath at 59degC with continuous stirring and the SPME poly(dimethylsiloxane) fibres, of 0.1 mm film thickness were inserted. After headspace sampling for 15 min, the fibres were withdrawn and introduced into the injection port of the GC at 250degC for 2 min. The volatiles were desorbed onto and analysed by GC on a fused-silica column (30 m * 0.25 mm i.d.) coated with Carbowax (0.25

mum), operated with temperature programming from 40degC (held for 5 min), to 180degC at 25degC/min), with He as carrier gas (1 ml/min) and EIMS detection operated with selected-ion monitoring at m/z 58 and 66 and 59 and 68, respectively, for trimethylamine (I) and IS. The calibration graph was linear from 0.8- 157muM-I. Intra-assay RSD (n = 7) for 10.7, 50.6 and 95.2muM-I aqueous standards were 6.1-9.9% and for a urine with 2.4muM-I was 12.2% (n = 8). Recoveries of 7.2, 31.4 and 125.6muM of added I averaged 84, 75 and 85% respectively. Recent work with a Carboxen-poly(dimethylsiloxane) fibre (75 mum film thickness) gave lower detection limits of 10nM. In ten healthy adults, the I in urine averaged 0.8% of the total, determined after reduction of trimethylamine oxide with titanium(III) sulfate.

Record - 21

TI- Headspace solid-phase micro-extraction for the determination of benzene, toluene, ethylbenzene and xylenes in urine.

AU- Fustinoni, S;Giampiccolo, R;Pulvirenti, S;Buratti, M;Colombi, A

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1999

VO- 723

NO- 1-2

PG- 105-115

AB- Samples (2 ml) were transferred to 7.9 ml headspace phials each containing 1 g of NaCl and the phials were sealed. Portions (0.5 ml) of a methanolic solution of benzene-d₆, toluene-d₈ and p-xylene-d₁₀ (4.75, 4.71 and 4.74 mug/ml, internal standards) were added to each through the septum. The contents were equilibrated at 40degC for 30 min and the SPME fibres (1 cm long, 0.1 mm coating of poly[dimethylsiloxane]), were introduced for 15 min. The fibres were retracted and transferred to the injection port at 250degC, where the adsorbed compounds were desorbed for 3 min. They were analysed by GC on a fused-silica column (60 m * 0.25 mm i.d.) coated with DB1 (1 mum), with He (1 ml/min) as carrier gas, temperature programming (50degC for 3 min, to 160degC at 8degC/min, cleaning at 260degC) and 70 eV EIMS detection operated in selected-ion monitoring mode (operating m/z listed for each analyte). Calibration graphs were linear from the detection limit of 12-34 ng/l to 5 mug/l. The extraction efficiencies varied from 4.4% for benzene to 14.6% for o-xylene. In the range 125-1250 ng/l of each compound, the intra-assay RSD (n = 6) were 1-9%. Samples stored at -20degC were stable for up to two months. The method was applied to two subjects exposed to urban air pollution.

Record - 22

TI- Solid-phase microextraction coupled with high-performance liquid

chromatography for the determination of aromatic amines.

AU- Wu, YC;Huang, SD

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 2

PG- 310-318

AB- Poly(dimethylsiloxane) - divinylbenzene (PDMS-DVB), Carbowax - templated resin (CW-TPR), Carbowax - divinylbenzene and polyacrylate fibres (50-85 μm) were evaluated for the SPME of six aromatic amines. The analytes were determined on 4 μm Nova-Pak C18 columns (15 cm * 3.9 mm i.d.) with acetonitrile/0.1M-acetate buffer of pH 4.66 (2:3) as mobile phase and detection at 280 nm. The best extraction efficiencies were obtained with CW - TPR fibres for polar amines and PDMS - DVB fibres for less polar amines. Optimization studies indicated use of the static and dynamic modes for these two fibre types, respectively, the mobile phase (0.2 ml/min) as desorption solvent, a soaking period of 2 min and a desorption time of 2 min. The effects of carry-over, absorption-time profile, pH, extraction temperature and ionic strength were also examined. Detection limits were 0.65-1.5 ng/ml using CW - TPR fibres and 0.33-2.4 ng/ml using PDMS - DVB fibres, the calibration graphs were linear in the ranges 1000-10 and 500-10 ng/ml, respectively, and the RSD was 3-8% (n = 7). The method was applied to the determination of aromatic amines in lake water.

Record - 23

TI- Application of isotope dilution to ion-trap gas chromatography-mass spectrometry.

AU- Barshick, CM;Barshick, SA;Walsh, EB;Vance, MA;Britt, PF

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 2

PG- 483-488

AB- A 10 μl portion of inorganic Hg (NIST SRM 3133 in HNO_3) was added to 4 ml NaHCO_3 buffer of pH 10, then 0.5 ml of 1% KI solution and 0.5 ml 0.1M-methylbis(dimethylglyoximate)pyridinecobalt(III) in ethanol were added and alkylation was performed at 50degC for 55 min. The methylmercury iodide produced was sampled with a SPME fibre for 5 min, desorbed for 3 min and analysed on a column (30 m * 0.25 mm i.d.) coated with DB-5MS (0.25 μm) operated with temperature programming from 40-250degC at 25degC/min, with He as carrier gas (1 kg/cm² head pressure) and ion-trap quadrupole MS detection at m/z 342, 344 and 346. For isotope dilution experiments an enriched isotopic spike was added to the sample before alkylation and a second

temperature programme, 125-150degC at 2degC/min, was included to slow elution of the analyte peak. The transient nature of the GC profile and pulsed ion trapping did not limit the precision, as demonstrated by the RSD of 0.19-2.5%, depending on the relative abundance of the isotopes and the concentration of Hg in the sample. Isotope dilution had an accuracy at the 400 ppb level which was improved by a factor of 30 over the calibration graph approach and a factor of 14 over the standard addition method.

Record - 24

TI- Comparison of gas-sampled and SPME-sampled static headspace for the determination of volatile flavour components.

AU- Miller, ME;Stuart, JD

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 1

PG- 23-27

AB- The volatile flavour compounds in fruit juices and calibration standards were sampled by static headspace extraction and headspace SPME, and analysed by GC-MS (details given). The traditional static method lacked the sensitivity needed for volatiles such as alpha-terpinene and linalool, whereas SPME had improved extraction ability, particularly with 65 µm poly(dimethylsiloxane)-divinylbenzene fibres. Of the nine components analysed, the average increase in sensitivity was sim1800. The SPME method could also detect the oxidation of flavour compounds in orange juice aged for three months.

Record - 25

TI- Analysis of 2-methylisoborneol and geosmin in catfish by microwave distillation - solid-phase microextraction.

AU- Lloyd, SW;Grimm, CC

JN- Journal of Agricultural and Food Chemistry

PY- 1999

VO- 47

NO- 1

PG- 164-169

AB- Minced sample (20 g) was placed in a 100 ml round-bottomed flask with a double offset inlet adapter, one inlet of which held a length of 5 mm i.d. PTFE tubing that carried N₂ (80 ml/min) to the bottom of the flask, and the other a delivery tube of the same material that led to a 50 ml graduated cylinder in a water bath at 0degC. The two lengths of tubing passed to the adapter and flask through holes drilled in the side of a microwave oven. The flask was mounted in the oven on a 250 ml beaker containing 10 ml of water, and was heated at 370 W for

3 min. The distillate was diluted to 8 ml with H₂O, treated with 2.5 g of NaCl, placed in a 12 ml septum phial containing a magnetic stirrer, and stirred for 15 min in a water bath at 40degC with a SPME fibre (100 µm polydimethylsiloxane coating) exposed to the headspace. The fibre was then heated in the GC injection port at 270degC for 1 min in the splitless mode, and the desorbed volatiles were separated on a column (30 m * 0.25 mm) coated with 5% phenyl methylpolysiloxane (0.25 µm) and operated with temperature programming from 40degC (held for 1 min after insertion of the fibre) to 80degC at 40degC/min, then to 115degC at 15degC/min, and then to 250degC (held for 3 min) at 40degC/min and ion-trap MS detection operated in selected-ion monitoring mode (m/z listed for each analyte). Calibration graphs were linear for 0.1-30 µg/kg of either compound added to untainted fish, and the detection limits were 0.01 µg/kg. Results for 4 samples agreed well with those returned by a taste panel.

Record - 26

TI- Techniques for Analyzing Aroma

AU- Marsili, R

AB- US \$165.00; hardcover. Volume 79 in the Food Science and Technology Series which covers solvent extraction and distillation techniques, the use of headspace-GC, direct thermal desorption, SPME, multidimensional GC, the importance and resolution of enantiomers, ion-trap MS, mechanisms of formation and analysis of off flavours, GC-olfactometry for the determination of key odorants and the electronic nose.

Record - 27

TI- Comparative studies of the leachate of an industrial landfill by gas chromatography-mass spectrometry, liquid chromatography-nuclear magnetic resonance and liquid chromatography-mass spectrometry.

AU- Benfenati, E;Pierucci, P;Fanelli, R;Preiss, A;Godejohann, M;Astratov, M;Levsen, K;Barcelo, D

JN- Journal of Chromatography, A

PY- 1999

VO- 831

NO- 2

PG- 243-256

AB- Leachate (200 ml) was adjusted to pH to 9 with NaOH and extracted with 3 * 20 ml CH₂ to remove excess neutral and basic analytes. The aqueous fraction was acidified with HCl (pH 1), cleaned up on a LiChrolut EN SPE cartridge and the organic pollutants (OP) were eluted with acetonitrile (2 * 3 ml). For LC, LC-MS, or LC-NMR; the combined organic extracts were evaporated to dryness (under a stream

of N₂) and the residue was dissolved in mobile phase. Portions were analysed by LC on a 5 µm LiChrospher 100 RP-18 column (25 cm * 4.6 mm i.d.), with gradient elution (0.4 ml/min), with methanol/0.2% formic acid (program details given) diode-array detection. For LC-MS, the conditions were the same but ammonium formate was added post column for thermospray MS detection on a Finnigan Model 4500. LC-NMR was performed with a 7 µm Merck LiChrolut column (7.5 cm * 4 mm i.d.), acetonitrile/0.1% trifluoroacetic acid in 2H₂O (3:2) as mobile phase (0.017 ml/min) and a Bruker AMX 600 spectrometer. For GC-MS analysis the leachate was extracted by SPE or SPME and analysis performed on a 0.25 µm CPSil8CB column (25 m * 0.25 mm) or a 1.2 µm CPSil8CB column respectively. Over 60 OP (tabulated) were identified by the 3 methods.

Record - 28

TI- Determination of organometallic compounds in surface water and sediment samples with SPME-CGC-ICP MS.

AU- DeSmaele, T; Moens, L; Sandra, P; Dams, R

JN- Mikrochimica Acta

PY- 1999

VO- 130

NO- 4

PG- 241-251

AB- Water (25 ml) was adjusted to pH 5 with sodium acetate/acetic acid buffer and tripropyltin acetate (1 µg/ml) was added as internal standard. The vial was sealed with a PTFE-coated silicone rubber septum and 500 µl aqueous 1% sodium tetraethylborate was subsequently added. The needle of the SPME device pierced the septum and the SPME fibre was exposed to the headspace for 10 min. Sediments were treated similarly except 0.5 g sediment was mixed ultrasonically with 1 ml concentrated acetic acid, 1 ml methanol and internal standard before adjusting to pH 5 with sodium acetate/acetic acid buffer. The compounds were thermally desorbed into the GC port. The sorbed compounds were subjected to capillary GC (CGC) on a FSOT column (30 m * 0.25 mm i.d.) coated with polydimethylsiloxane (0.5 µm) operated with temperature programming from 60°C (held for 1 min) to 120°C (held for 0.5 min) at 30°C/min, then to 230°C (held for 0.7 min) with Xe/H₂ (1:99) as carrier gas (435.1 bar) and ICP MS detection with an r.f. power of 1250 W and carrier, auxiliary and plasma gas flow rates of 1.1-1.25, 1.2 and 15 l/min, respectively. The analysis time was 10 min. Detection limits ranged from 0.13-3.7 ng/l as metal.

Record - 29

TI- In-situ derivatisation of degradation products of chemical warfare

agents in water by solid-phase microextraction and gas chromatographic-mass spectrometric analysis.

AU- Sng, MT;Ng, WF

JN- Journal of Chromatography, A

PY- 1999

VO- 832

NO- 1-2

PG- 173-182

AB- Degradation products of chemical warfare agents in water were extracted by SPME on Carboxen coated fibers at room temperature, followed by on-line derivatization with N-methyl-N-(tert-butyl)dimethylsilyl)trifluoroacetamide with 1% tert-butyl)dimethylsilyl chloride, and analysed on a fused-silica column (25 cm * 0.25 mm i.d.) coated with HP-5MS (0.25 µm), operated with temperature program from 40degC (held for 2 min) to 280degC (held for 4 min) at 20degC/min, He as carrier gas (35 cm/s) and EIMS detection. Detection limits were in the range 1-200 ppb with an RSD of 10-35%.

Record - 30

TI- Determination of methylcyclopentadienylmanganese tricarbonyl (MMT) in aqueous samples by SPME-GC-AED.

AU- Yang, F;Chau, YK

JN- Analyst (Cambridge, U. K.)

PY- 1999

VO- 124

NO- 1

PG- 71-73

AB- The sample (20 ml) was placed in a 40 ml vial with a septum cap. SPME of MMT was performed by exposing a polydimethylsiloxane-coated silica fibre to the headspace above the stirred sample for 15 min at room temperature. The fibre was then heated at 250degC for 15 s and the thermally desorbed MMT was determined by GC on a SPB-1 column (30 m * 0.53 mm i.d.), operated with temperature programming from 45degC (held for 0.5 min) to 200degC at 30degC/min, He as the carrier gas (flow rate not given) and atomic emission detection at 259 nm. The calibration graph was linear for 1-1000 pg/l MMT, the detection limit was 0.3 pg/l and the RSD (n = 4) at the 10 pg/l MMT level was 7.1%. The method was applied to highway run-off, lake water and waste water.

Record - 31

TI- Non-equilibrium quantitation of volatiles in air streams by solid-phase microextraction.

AU- Bartelt, RJ;Zilkowski, BW

JN- Analytical Chemistry

PY- 1999

VO- 71

NO- 1

PG- 92-101

AB- The kinetics of extraction by poly(dimethylsiloxane) SPME fibres were studied for C9-22 alkanes, C6-13 primary alcohols and methyl esters of C6-16 carboxylic acids. Air streams containing constant concentrations of analytes were sampled for 30 min to 3 days. Temperature, fibre coating thickness, air flow rate and tubing diameter in which sampling took place were variable parameters, resulting in over 1900 data points. The results were described by a simple kinetic equation including the relationship between fibre sensitivity and equilibration time. Non-linear regression was used to link the equation parameters to known analyte properties and sampling conditions. This allowed calculation of the absolute concentration of the analyte in the air stream directly from the amount captured by the SPME fibre, regardless of whether equilibrium was established. In practice, if one of the four studied configurations of air flow rate and sampling port diameter was used, then only sampling temperature, sampling time, analyte functional group and analyte GC retention index need be known.

Record - 32

TI- Solid-phase micro extraction coupled with semi-microcolumn high-performance liquid chromatography for the analysis of benzodiazepines in human urine.

AU- Jinno, K;Taniguchi, M;Hayashida, M

JN- Journal of Pharmaceutical and Biomedical Analysis

PY- 1998

VO- 17

NO- 6-7

PG- 1081-1091

AB- Benzodiazepines (BDAZ) in urine were extracted over 60 min by SPME at 60degC and with a saturated salt concentration, neutral-weak alkaline pH and desorption with 30 μ l acetonitrile over 30 min. Extract (1 μ l) was analysed on a Superiorex ODS column (25 cm * 1.5 mm i.d.) with aqueous 35% acetonitrile as the mobile phase (0.1 ml/min) and detection at 220 nm. Calibration graphs were linear from 5-1000, 5-400, 5-1000, 5-400, 5-1000, 20-400 ppb, respectively, with corresponding detection limits of 4, 2, 1, 1, 2, and 6 pbb, for BDAZ viz. nitrazepam, flunitrazepam, fludiazepam, diazepam, clotiazepam and medazepam, respectively. RSD were <14% (n = not stated).

Record - 33

TI- Biomonitoring of benzene and toluene in human blood by headspace-

solid-phase microextraction.

AU- Schimming, E;Levsen, K;Koehme, C;Schuermann, W

JN- Fresenius' Journal of Analytical Chemistry

PY- 1999

VO- 363

NO- 1

PG- 88-91

AB- SPME fibres coated with 65 µm carboxene/polydimethylsiloxane were used for biomonitoring. A fibre was exposed to the headspace, at room temperature, above the blood sample. Benzene-d₆ and toluene-d₈ were used as internal standards. During fibre exposure, the blood was stirred. After an extraction time of 30 min the fibre was withdrawn from the headspace and transferred to the GC injector. GC was performed on a 0.25 µm Supelcowax column (30 m * 0.32 mm i.d.) operated with temperature programming from 40degC (held for 1 min) to 100degC at 5degC/min and then to 250degC (held for 8 min) at 25degC/min, He as carrier gas and MS detection with selected-ion monitoring. Calibration graphs were linear from 25-5000 ng/l. Limits of quantification were 5 ng/l for benzene and 25 ng/l for toluene. RSD were 4-17.1% (n = 4-5). Results compared well with those from existing methods. Results of a field study are tabulated.

Record - 34

TI- Determination of Henry's law coefficients by combination of the equilibrium partitioning in closed systems and solid-phase micro-extraction techniques.

AU- Dewulf, J;VanLangenhove, H;Everaert, P

JN- Journal of Chromatography, A

PY- 1999

VO- 830

NO- 2

PG- 353-363

AB- Air-water partitioning of twenty VOC, including aliphatic and aromatic hydrocarbons, chlorinated and fluorinated compounds, ethers, esters, biphenyl and nitrogen compounds was measured by GC. Portions (0.5 ml) of demineralized water were injected into three phials of 118 ml capacity and 90 ml into three others. Magnetic stirrer bars (0.55 ml) were added to each and 5 µl aliquots of methanolic stock solutions of 4-6 compounds were injected under the water. The sealed phials were stirred for 15 h at 2, 6, 10, 18 and 25degC. The headspace was sampled for 30 min with a 0.1 mm dimethylpoly(siloxane) fibre or, for the more polar compounds, with a 65 µm divinylbenzene-Carbowax fibre. The fibres were transferred to the injector at 220degC. The desorbed analytes were analysed by GC on a fused-silica column (30 m * 0.53 mm i.d.) coated with CP-SIL 5 CB (5 µm), operated with temperature programming from 40 or 45degC to 240degC,

(details given) with He (6.4 ml/min) as carrier gas and FID. Henry's law coefficients were calculated from the results of the two measurements. The range of values reported is 0.00042 for nitrobenzene at 2degC to 13.5 for 1,1,2-trichlorotrifluoroethane at 25degC; RSD were <10% for 84% of the measurements. There was generally good agreement with published results, except for the less volatile compounds, for which very little published data was available.

Record - 35

TI- Study of polymer coatings for solid-phase microextraction.

AU- Zhang, DN;Wu, CY;Ai, F

JN- Sepu

PY- 1999

VO- 17

NO- 1

PG- 10-13

AB- Silica fibres coated with polymethylvinylsiloxane (PMVS; with 1% vinyl content) to 88 and 44 µm in thickness after curing were prepared (experimental details given) and studied as adsorbents for the SPME of semi-volatile organic compounds (SVOC) and VOC from liquid matrices. In particular, the use of the polymer coatings for headspace SPME-GC, e.g. separation of CH₂Cl₂, benzene, 1-bromobutane, toluene, 1-bromopentane, ethylbenzene, p-xylene, n-propylbenzene, bromocyclohexane and n-butylbenzene from waste water, and ethyl acetate, ethanol, pentanol, isopentanol, acetic acid, furfural and butyric acid from white spirit was investigated. The results were compared with those obtained by the commercially-available polymer coatings. The adsorption and desorption kinetics of PMVS were also investigated. Results indicate that PMVS was efficient for the extraction of SVOC and VOC, with its high thermostability and easy-coating properties. The detection limit of 88 µm PMVS coating was 1-5 µg/l of benzene in 3 min.

Record - 36

TI- Comparison to two sample preparation techniques of sniffing experiments with broccoli (*Brassica oleracea* var. *italica* Plenck).

AU- Ulrich, D;Krumbein, A;Schonhof, I;Hoberg, E

JN- Nahrung

PY- 1998

VO- 42

NO- 6

PG- 392-394

AB- Aroma compounds were extracted from broccoli (300 g) using two sampling methods, viz. (i) dynamic head-space sampling using air (150

ml/min) passed through Tenax for 30 min. The volatiles were eluted with 3 ml acetone, concentrated to 50 µl and (ii) head-space SPME with adsorption onto the SPME fibre carried out in a headspace vial at 35degC. Samples were injected onto a fused-silica column (30 m * 0.32 mm i.d.) coated with HP-INNO wax (0.5 µm), operated with temperature programming from 40degC (held for 3min) to 60degC (held for 2 min) at 1degC/min and to 180degC (held for 10 min) at 5degC/min, with H₂ as carrier gas (2 ml/min) and following a carrier gas split (1:1), both FID and sniffing port sensory evaluation (SE) detection. Method ii was found to be more useful for qualitative GC-SE.

Record - 37

TI- A novel sorbent for the determination of clenbuterol in bovine liver.

AU- Horne, E;O'Keefe, M;Desbrow, C;Howells, A

JN- Analyst (Cambridge, U. K.)

PY- 1998

VO- 123

NO- 12

PG- 2517-2520

AB- The best C18 sorbent for the matrix solid-phase dispersion (MSPD) extraction of clenbuterol from liver (cf., Barker et al., J. Chromatogr., 1989, 475, 353; Long et al., J. Agric. Food Chem., 1990, 38, 423) in terms of efficient blending with the sample and packing of the mixture for washing and elution was Isolute MSPD-grade C18 sorbent (end-capped). Recoveries of 5 ng/g of clenbuterol from liver in two studies with use of this sorbent were 90 and 92%. After enzymic deconjugation and liquid-liquid extraction, further clean up and decolorization were necessary, and were best achieved by SPE on a mixed-mode cation-exchange packing, e.g., XtrackT. The clenbuterol was determined with an RIA kit (Laboratoire d'Hormonologie, Marloie, Belgium).

Record - 38

TI- Solid-phase microextraction applied to the analysis of pesticide residues in honey using gas chromatography with electron-capture detection.

AU- Jimenez, JJ;Bernal, JL;delNozal, MJ;Martin, MT;Mayorga, AL

JN- Journal of Chromatography, A

PY- 1998

VO- 829

NO- 1-2

PG- 269-277

AB- Samples (5 g) were diluted to 25 ml with H₂O and 3 ml portions were transferred to 4 ml vials. The vials were sealed and a 0.1 mm non-

bonded poly(dimethylsiloxane) fibre (Supelco) was introduced. Extraction was carried out for 1 h at 70degC with stirring. The fibre was transferred to the injection port for desorption at 260degC for 4 min. GC was performed on a column (60 m * 0.25 mm i.d.) coated with 50% phenylmethylpoly(siloxane) (0.25 µm) with He (0.7 ml/min) as carrier gas, temperature programming (50-275degC, details given) and ECD. For the 22 pesticides tested, calibration graphs were linear for 6.5-500 ng injected, with detection limits of 0.1-30 ppb. Recoveries of 0.08, 0.2 and 0.5 ppm were $\geq 82\%$, with RSD of 1.5-18% (n = 5), with an average of 9.3%. The results were superior to those obtained with 7 µm bonded poly(dimethylsiloxane) and partly crosslinked 85 µm poly(acrylate) fibres, but the high RSD made the method better suited for semiquantitative screening purposes. The performance of the fibre deteriorated after about 60 analyses.

Record - 39

TI- The application of solid phase microextraction in the analysis of organophosphorus pesticides in a food plant.

AU- Chen, WQ;Poon, KF;Lam, MHW

JN- Environmental Science and Technology

PY- 1998

VO- 32

NO- 23

PG- 3816-3820

AB- Organophosphorus pesticides were extracted from a mixture of *Chrysanthemum coronarium* tissue in H₂O by an SPME fiber coated with 100 µm poly(dimethylsiloxane) and determined by GC with flame photometric detection (details given). Pesticide recoveries were related to the plant tissue/H₂O ratio (f) and the partition coefficient (K_{WV}). The model was verified using phorate, diazinon, methyl parathion, and ethion. Good correlations were obtained between the K_{WV} values and their octanol/H₂O partition coefficients (K_{OW}).

Record - 40

TI- Solid-phase microextraction coupled to gas chromatography-mass spectrometry for the determination of the adsorption coefficients of triazines in soil.

AU- Zambonin, CG;Catucci, F;Palmisano, F

JN- Analyst (Cambridge, U. K.)

PY- 1998

VO- 123

NO- 12

PG- 2825-2828

AB- Air-dried sieved soil or sediment (3 g) was mixed with 5 ml of H₂O, then 50 µl mixed triazine herbicide (TH) solution (containing 6 TH,

viz.) ametryn, prometryn, propazine, sebuthylazine, terbuthylazine and terbutryn) in acetonitrile was added, and the mixture was stirred, set aside overnight and centrifuged. The supernatant liquid was submitted to SPME on a polyacrylate-coated silica fibre with subsequent thermal desorption on to an SPB-5 column for selected-ion monitoring at m/z 227 and 212; 241 and 184; 229 and 172; 229 and 200; 214 and 173; and 226 and 185 for the respective triazines. The response was linear for 1-220 ng/ml. None of the triazines was recovered from peat. Values of log. Koc, where Koc is the adsorption coefficient (= Kd/foc, where Kd is the soil-to-H₂O distribution coefficient and foc is the fraction of organic C in the soil or sediment) are tabulated and compared with literature values.

Record - 41

TI- Use of automated SPME coupled to GC-AED for the determination of metazachlor in waste water.

AU- Wenner, A; Wortberg, M

JN- Journal of High Resolution Chromatography

PY- 1998

VO- 21

NO- 12

PG- 661-664

AB- Metazachlor in waste water was extracted using (i) 85 µm polyacrylate (PA) fibres and (ii) 7 µm and 100 µm polydimethylsiloxane (PDMS) fibres. After extraction, samples were desorbed at 280degC and analysed on a fused silica column (30 m * 0.32 mm i.d.) coated with DB5 (0.25 µm), operated with temperature programming from 60degC (held for 1 min) to 200degC at 35degC/min then to 270degC (held for 1 min) at 6degC/min, with He as carrier gas (94 kPa) and atomic-emission detection (AED). The 85 µm PA fibres had the highest capacity and were used throughout. Recoveries of metachlor were sim100% at the 200 µg/l level.

Record - 42

TI- Automatic analytical method for the determination of acetaldehyde in drinking water in poly(ethylene terephthalate) (PET) bottles by headspace solid-phase micro-extraction.

AU- Huynh, CK; VuDuc, T

JN- Mitteilungen aus dem Gebiete der Lebensmitteluntersuchung und Hygiene

PY- 1998

VO- 89

NO- 6

PG- 705-714

AB- A 10 ml sample in a 15 ml flask was mixed with 6 g NaCl, and a 2 cm long Carboxen PDMS fibre (pre-conditioned at 280degC) was used to

adsorb compounds from the headspace vapour in the flask during 25 min at room temperature. The fibre was transferred to an on-column injector maintained at 260degC to desorb the retained compounds during 5 min on to a fused-silica GC column (30 m * 0.32 mm i.d.) coated with DB-1 (3 mum) and operated with temperature programming from 40degC (held for 1 min) at 20degC/min to 280degC, with N2 as carrier gas (14 psi) and FID. Acetaldehyde (I) was identified from its mass spectrum recorded with an ion-trap spectrometer. The calibration graph was linear up to 1200 mug/l of I in water and detection limit was 1 mug/l. The procedure was readily automated and had advantages over conventional headspace GC and HPLC.

Record - 43

TI- Determination of a non-ionic surfactant by solid-phase microextraction coupled with high-performance liquid chromatography and online derivatization.

AU- Aranda, R;Burk, RC

JN- Journal of Chromatography, A

PY- 1998

VO- 829

NO- 1-2

PG- 401-406

AB- The poly(dimethylsiloxane)-divinylbenzene SPME fibres (60 mum film thickness) were immersed in stirred 6 ml water samples containing Brij 56 for 1 h, dried in the headspace for 5 min and transferred to the 60 mul SPME-HPLC interface, filled with a reagent of 0.05% each of 4-(dimethylamino)pyridine and 1-naphthoyl chloride in pyridine. After 30 min reaction time, unconsumed reagent was partially vented through a valve. The mobile phase (2 ml/min) of a gradient of aqueous 70% acetonitrile in methanol (details given) was pumped through the interface and on to the guard column (1.25 cm * 4.6 mm i.d.) and the analytical column (25 cm * 4.6 mm i.d.) of 5 mum Zorbax ODS, with fluorescence detection of the derivative at 366 nm (excitation at 228 nm). Calibration graphs were linear for 0.1 (detection limit) to 10 mg/l of Brij 56. The high detection limit was due to inefficient extraction (15-20%) and excess derivatizing reagent. Deterioration of the fibres caused by the naphthoyl chloride was also a limiting factor.

Record - 44

TI- Poly(dimethylsiloxane) films as sorbents for solid-phase microextraction coupled with infra-red spectroscopy.

AU- Merschman, SA;Lubbard, SH;Tilotta, DC

JN- Journal of Chromatography, A

PY- 1998

VO- 829

NO- 1-2

PG- 377-384

AB- Pieces of poly(dimethylsiloxane) film (3.2 cm * 3.2 cm, 127 µm thick) were cut and four 6 mm holes were punched in the corners for mounting in aluminium holders as described previously (Environ. Sci. Technol., 1996, 30, 1219). The films were conditioned (details given), mounted in the holders and placed in 250 ml of the water sample, with stirring at similar rates as judged by the vortex height. The films were removed from the sample, dried briefly to minimize evaporation losses and examined by FTIR at a scan rate of 6.25 kHz, with triangular apodization. Thirty-two scans were co-added both for the sample and the background correction spectra. The method was tested with five analytes, trifluralin, o-xylene, p-xylene, trichloroethylene and perchloroethylene. Equilibration times for 20-60 ppm of these compounds were 60-85 min. Linear calibration graphs were obtained for 0.2-2, 5-100, 5-100, 4-100 and 1-40 ppm, in the order named, with RSD of 6.7-12% (mainly due to volatility losses) and detection limits of 0.19-4 ppm. The matrix in a lake water sample did not interfere. In comparison with the Parafilm M film tested previously (loc. cit), the polymer film had similar distribution constants, shorter equilibration times, higher detection limits and poorer optical transparency. Both films were re-usable.

Record - 45

TI- Solid-phase microextraction with pH adjustment for the determination of aromatic acids and bases in water.

AU- vanDoorn, H;Grabanski, CB;Miller, DJ;Hawthorne, SB

JN- Journal of Chromatography, A

PY- 1998

VO- 829

NO- 1-2

PG- 223-233

AB- Solutions of test compounds in buffers of pH 2, 7 or 12 (1.9 ml) were stirred for 30 min in contact with the three SPME fibres tested: 0.1 mm poly(dimethylsiloxane); 85 µm poly(acrylate); and 65 µm Carbowax-divinylbenzene. Analytes were desorbed in the injection port at 300degC inside a 2 mm i.d. liner. They were determined on a column (25 m * 0.33 mm i.d.) coated with HP-5 (0.17 µm), with He as carrier gas, temperature programming (35-300degC, details given) and FID. The results of sorptions of five phenols and seven bases were used to compare the fibres. For both sets of compounds, sorption behaviour as a function of pH was as expected from their dissociation coefficients. The poly(dimethylsiloxane) fibre gave lower sorption than the other two. The Carbowax-divinylbenzene fibre gave lower detection limits (0.5-10 µg/l) than the poly(acrylate) fibre (1-50

mug/l), the latter gave better selectivity and was preferred. The method was applied directly to a sample of wetland water. Portions (1.9 ml) were adjusted to a pH of 2 or 12 before extraction. Recoveries of an added 5 mug/ml of each compound were 73-117%, with RSD of 5-26%. A sample of highly contaminated coal gasification waste water was adjusted to a pH of 2 and diluted 1:49 with buffer of pH 2 before analysis. The results for 10 phenols agreed well with those obtained by solvent extraction.

Record - 46

TI- Analysis of volatile organic compounds in environmental water samples and soil gas by solid-phase microextraction.

AU- Nilsson, T;Montanarella, L;Baglio, D;Tilio, R;Bidoglio, G;Facchetti, S

JN- International Journal of Environmental Analytical Chemistry

PY- 1998

VO- 69

NO- 3

PG- 217-226

AB- The effects of some compounds found in environmental water on the SPME of 1 mug to 1 g/l of 1,1,1-trichloroethane and tri- and tetrachloroethene from water at 20degC by a fibre coated with 100 mum polydimethylsiloxane were studied. The VOC were desorbed from the fibre in a GC instrument injection port for 5 min at 250degC before GC on a HT-8 column (50 m * 0.22 mm i.d.; film thickness 0.25 mum) with temperature programming from 40degC (held for 5 min) to 150degC at 8degC/min and then to 250degC (held for 3 min) at 50degC/min, with H2 as carrier gas (40 cm/s) and ECD. The extraction efficiency was improved by the presence of 4% NaCl and unaffected by the presence of 5 mg/l of humic acids, 1 g/l of TiO2 or 1 g/l of TiO2/0.1M-NaClO4. Calibration graphs were linear with standard deviations of 5% and detection limits were in the ng/l range. The method should be useful for studying TiO2-catalysed VOC degradation. A SPME probe (diagram and description given) was used in situ to detect or determine eight VOC in ground water giving results which are higher than those obtained by traditional sampling (procedure described).

Record - 47

TI- Recent advances in solid-phase microextraction.

AU- Lord, HL;Pawliszyn, J

JN- LC-GC INTERNATIONAL

PY- 1998

VO- 11

NO- 12

PG- 776-777, 780-782

AB- The authors' review their recent work using SPME including the design of new interfaces for separation and detection and the development of new SPME technology. The use of conventional fibre-based SPME coupled with GC for the detection of styrene, petroleum hydrocarbons and formaldehyde in air, and amphetamines and other drugs in urine and other complex matrices is discussed. The use of conventional fibre-based SPME coupled with HPLC for the detection of non-ionic surfactants in waste water is also discussed together with in-tube SPME coupled with HPLC for the automated analysis of phenylurea pesticides and pharmaceuticals.

Record - 48

TI- Solid-phase microextraction in headspace analysis. Dynamics in non-steady-state mass transfer.

AU- Ai, J

JN- Analytical Chemistry

PY- 1998

VO- 70

NO- 22

PG- 4822-4826

AB- A mathematical model dealing with the situation of non-steady-state mass transfer for headspace SPME is presented. Silica fibres were used, coated with 85 µm polyacrylate and 100 µm poly(dimethylsiloxane) and the chemical used was octan-2-ol. Analysis was by GC on a 0.25 µm DB5 column (30 m * 0.25 mm i.d.) operated at 200degC with He as carrier gas (1 ml/min) and 70 eV EI MS detection. A mathematical solution was obtained for the dynamic process of the non-steady-state mass transfer by correlating the variation of the analyte concentration in the headspace with the analyte extraction rate. The solution provided an equation relating the extracted amount of analyte to the extraction time. A better description of experimental observations was given compared with models dealing with steady-state mass transfer.

Record - 49

TI- The effect of sample volume on quantitative analysis by solid-phase microextraction. Part 2. Experimental verification.

AU- Gorecki, T;Khaled, A;Pawliszyn, J

JN- Analyst (Cambridge, U. K.)

PY- 1998

VO- 123

NO- 12

PG- 2819-2824

AB- Verification is presented of the theoretical predictions made in Part 1 (Ibid., 1997, 122, 1079) in respect of (i) the effect of the sample

volume on the amount extracted in two-phase (fibre/sample) and three-phase (fibre/headspace/sample) systems and on the measurement of the fibre-to-sample partition coefficient in two-phase systems and (ii) the effect of the headspace capacity on the amount extracted and the extraction kinetics. Problems involved in the measurement of large partition coefficients and of partition coefficients of semivolatile compounds in H₂O are discussed.

Record - 50

TI- An investigation of the aroma fraction of some Italian wines by solid-phase microextraction gas chromatography-mass spectrometry and membrane inlet mass spectrometry.

AU- Favretto, D;Grandis, G;Allegri, G;Traldi, P

JN- Rapid Communications in Mass Spectrometry

PY- 1998

VO- 12

NO- 21

PG- 1595-1600

AB- In SPME of aroma compounds from wine, polydimethylsiloxane-divinyl benzene fibres were either immersed directly in the liquid sample or exposed to headspace vapours for various times at different temperatures. The analytes were thermally desorbed during 5 min at 220degC and the resulting mixtures were analysed by GC on a Varian Saturn 3 system equipped with a fused-silica column (30 m * 0.25 mm i.d.) coated with DB5-MS (0.25 µm), operated with temperature-programming from 40degC (held for 5 min) to 120degC at 5degC/min, and to 300degC at 10degC/min and 70 eV EIMS detection. Membrane inlet MS (MIMS) studies were performed with use of the inlet separation device developed previously by Virkki et al. (c.f., Anal. Chem., 1995, 67, 1421). Results are presented for aroma compounds in 5 Chianti wines. SPME-GC-MS was very specific and permitted differentiation between wines grown from the same grape variety by producers in different areas. However, the specificity of MIMS was poorer than that of SPME-GC-MS, but was more rapid and easier to perform and hence should prove useful for rapid screening exercises.

Record - 51

TI- Use of digital aroma technology and SPME GC-MS to compare volatile compounds produced by bacteria isolated from processed poultry.

AU- Arnold, JW;Senter, SD

JN- Journal of the Science of Food and Agriculture

PY- 1998

VO- 78

NO- 3

PG- 343-348

AB- The VOC produced by 7 bacterial species were analysed by digital aroma technology on an Aromascanner (Foss Food Technology Corp., Eden Prairie, MN, USA). Graphical output by the Sammon mapping technique produced patterns of differences or similarities among the samples. Artificial neural network software was used to model groups of sample and to classify subsequent unknowns. A 100 µm polydimethylsiloxane SPME fibre was used for headspace sampling of VOC from each species. GC was carried out on a fused-silica column (60 m * 0.25 mm i.d.) coated with DB-1 (0.25 µm), operated with temperature programming from 40degC to 275degC (held for 5 min) at 8degC/min with He as carrier gas (30 cm/s) and FID. Isolated VOC were identified by GC on the same column, operated with temperature programming from 40degC (held for 1 min) to 275degC (held for 5 min) at 8degC/min, with He as carrier gas (28 cm/s) and 70 eV EIMS detection. The digital system was able to distinguish between species after training and the method was reproducible. Profiles obtained by GC differed by species and were used as objective standards to compare against results obtained by the digital system.

Record - 52

TI- Headspace solid-phase microextraction for the analysis of volatiles in a meat product: dry cured Iberian ham.

AU- Ruiz, J;Cava, R;Ventanas, J;Jensen, JT

JN- Journal of Agricultural and Food Chemistry

PY- 1998

VO- 46

NO- 11

PG- 4688-4694

AB- A Supelco SPME fibre (10 mm length), coated with a 100 µm poly(dimethylsiloxane) film, was inserted into the headspace of a 10 ml vial containing 2 g ham. The vial was held at 40 or 60degC for 20, 40 or 60 min, after a 15 min re-equilibration stage and the VOC were adsorbed on the fibre. A fibre was subsequently transferred to the injection port at 220degC and the VOC were desorbed onto a fused-silica GC column (30 m * 0.25 mm i.d.) coated with phenyl/dimethylpolysiloxane (1:19; 1 µm) operated with temperature programming from 40degC to 250degC with He as carrier gas (1.6 ml/min) and 70 eV EIMS detection operated in full-scan mode with data collection (1 scan/s) for m/z 40-300. The majority of the 82 volatiles tentatively identified coincided with literature data.

Record - 53

TI- Determination of the plasticizer N-butylbenzenesulfonamide and the pharmaceutical ibuprofen in wastewater using solid-phase microextraction (SPME).

AU- Huppert, N;Wurtele, M;Hahn, HH
JN- Fresenius' Journal of Analytical Chemistry
PY- 1998
VO- 362
NO- 6
PG- 529-536

AB- The plasticizer N-butylbenzenesulfonamide (I) and the drug ibuprofen (II) were extracted from filtered wastewater samples by absorption on polyacrylate-coated fibres for 30 min. The fibres were rinsed with water and the analytes were thermally desorbed at 270degC for 5 min onto a GC column (30 m * 0.25 mm i.d.) coated with HP-5 MS (0.25 µm thick), operated with temperature-programming from 50degC (held for 2 min) to 150degC (held for 1 min) at 30degC/min and then to 300degC (held for 6 min) at 15degC/min and 70 eV EIMS detection operated in selected-ion monitoring mode at m/z 170 and 206, respectively, for I and II. Calibration graphs were linear up to 2 mg/l for both I and II with detection limits for both analytes were 0.1 µg/l. The results obtained on samples of municipal wastewater treatment plants were presented.

Record - 54

TI- Analysis of water samples for trace levels of oxygenate and aromatic compounds using headspace solid-phase microextraction and comprehensive two-dimensional gas chromatography.

AU- Gaines, RB;Ledford, EB;Stuart, JD
JN- Journal of Microcolumn Separations
PY- 1998
VO- 10
NO- 7
PG- 597-604

AB- Contaminated groundwater was added to a vial containing NaCl and heated at 40degC for 15 min then the headspace was sampled for 10 min with a SPME Carboxen/polydimethylsiloxane fibre. The fibre was removed and the trapped volatiles desorbed for 15 s and analysed by GC. The GC system comprised a fused-silica column (2 m * 0.1 mm i.d.) coated with (5% phenyl)methylsilicone (5 µm) serially connected to a second column (1 m * 0.1 mm i.d.) coated with (14% cyanopropylphenyl)methylsilicone (0.14 µm) which were separated by a modulator tube (0.47 m * 0.1 mm i.d.) coated with (5% phenyl)methylsilicone (5 µm) on the first 0.21 m and with deactivated fused silica on the remaining section and equipped with a deactivated fused silica precolumn (0.5 m * 0.1 mm i.d.). H₂ was used as carrier gas (70 cm/s) and FID was employed. The columns were operated at 25degC (held for 2 min) then the temperature was raised at 5degC/min until the last compound eluted (time 11 min). Calibration graphs were linear for 1-75 ppb methyl-t-butyl ether, 1-182 ppb

benzene and typically 1-450 ppb for ethylbutyl ether, toluene, ethylbenzene, and o-, m- and p-xylene, with detection limits of about 0.5 ppb.

Record - 55

TI- Derivatization/solid-phase microextraction followed by gas chromatography-mass spectrometry for the analysis of phenoxy acid herbicides in aqueous samples.

AU- Nilsson, T;Baglio, D;GaldoMiguez, I;OgaardMadsen, J;Facchetti, S

JN- Journal of Chromatography, A

PY- 1998

VO- 826

NO- 2

PG- 211-216

AB- Methods of derivatization, SPME and GC-MS were examined for determination of phenoxy-acid herbicides (phenoxy herbicides; PH) in waters. In the preferred procedure, the PH were adsorbed on to a fibre coated with crosslinked polydimethylsiloxane-divinylbenzene (65 µm) and impregnated with benzyl (or pentafluorobenzyl) bromide as derivatization agent. Desorption of products was at 290degC in the GC injection port. GC was effected a fused-silica column (30 m * 0.31 mm i.d.) coated with DB-5 (1 µm) and temperature-programmed from 120degC (held for 5 min) to 280degC (held for 2 min) at 8degC/min or a column (30 m * 0.25 mm i.d.) coated with SPB-5 (0.25 µm) and temperature-programmed from 50degC (held for 5 min) to 100degC at 20degC/min and to 250degC both at 10degC/min with He as carrier gas and ion-trap or EIMS detection operated in selected-ion monitoring mode (m/z listed for each PH). Characteristic ions and SPB-5 retention times are tabulated for each ester of 2,4-D, MCPA, dichlorprop and mecoprop and for the benzyl esters of these herbicides, respectively, lower detection limits (µg/l) were 1, 0.5, 0.2 and 0.1, with corresponding RSD of 32, 42, 18 and 14%.

Record - 56

TI- Optimization of solid-phase microextraction for the speciation of butyl- and phenyltin compounds using experimental designs.

AU- Lespes, G;Desauziers, V;Montigny, C;PotinGautier, M

JN- Journal of Chromatography, A

PY- 1998

VO- 826

NO- 1

PG- 67-76

AB- Organotin compounds in aqueous samples were determined by GC with flame photometric detection following in situ ethylation and recovery of the derivatives by solid-phase microextraction. Ethylation was

carried out by adding sample (100 ml) to 0.1 ml sodium tetraborate in 100 ml acetate buffer of pH 4.8 and reacting at room temperature with stirring. Microextraction was on fibres coated with either polydimethylsiloxane or Carbowax-divinylbenzene. The analytes were desorbed at 250degC in the GC injection port and separated on a fused-silica column (30 m * 0.25 mm i.d.) coated with methylsilicone (0.25 µm), operated with temperature programming from 70degC (held for 1 min) to 190degC at 30degC/min and to 270degC (held for 6 min) at 15degC/min with N₂ as carrier gas (0.7 ml/min) and FID. Experimental design methodology was used to evaluate the influence of the nature of the fibre, adsorption time, sample volume, injection temperature, desorption time and laboratory temperature upon peak area. The optimum operating conditions were determined by means of a mathematical model enabled detection down to 2-4 pg/ml. Calibration plots were linear for 50-600 pg/ml. The mean within-day RSD (n = 5) was 9%. The accuracy of the method was shown to be comparable methods involving solvent extraction.

Record - 57

TI- Using solid-phase microextraction to determine partition coefficients to humic acids and bioavailable concentrations of hydrophobic chemicals.

AU- Ramos, EU;Meijer, SN;Vaes, WHJ;Verhaar, HJM;Hermens, JLM

JN- Environmental Science and Technology

PY- 1998

VO- 32

NO- 21

PG- 3430-3435

AB- Negligible depletion SPME was used to determine free concentrations of highly hydrophobic chemicals in aquatic samples containing humic acids (details given). The method was used to analyse pairs of samples (with and without humic acids) with identical free concentrations and in addition was used to determine the dissolved organic carbon/H₂O partition coefficients by measuring the decrease in the free fraction of the compound when adding different amounts of humic acids. The method was used to study the effects of humic acids on the bioaccumulation of PCB in *Daphnia magna*.

Record - 58

TI- Determination of benzene, toluene, ethylbenzene and xylenes in indoor air at environmental levels using diffusive samplers in combination with headspace solid-phase microextraction and high-resolution gas chromatography-flame ionization detection.

AU- Elke, K;Jermann, E;Begerow, J;Dunemann, L

JN- Journal of Chromatography, A

PY- 1998

VO- 826

NO- 2

PG- 191-200

AB- Benzene, ethylbenzene, toluene and xylenes, (the so-called BTEX aromatic hydrocarbons) were sampled from indoor air with 3M 3500 OVM passive diffusive samplers or by active sampling on to tubes of SKC GK 26-16 charcoal (800 mg plus 200 mg backup). Alternatively, headspace SPME sample was used with a Supelco/Carboxen-PDMS fibre. BTEX analytes were extracted from the sample with CS₂/methanol, followed by xanthation to remove CS₂, for GC analysis on a fused-silica column (60 m * 0.32 mm i.d.) coated with DB-1701 (1 µm; or DB-5) and temperature-programmed from 35degC (held for 10 min) to 150degC at 5degC/min and to 200degC (held 10 for min) at 20degC/min for passive samples; or from 35degC (held for 5 min) to 160degC (held for 30 min) at 4degC/min for active samples, with He as carrier gas (2 ml/min) and FID. Lower detection limits were 0.4-2 µg/m³ for 2-h sampling or 0.4-1.1 µg/m³ for 24-h sampling. Intra- and inter-day RSD were 6.6-12.8 and 11.1-15.2%, respectively. Use of passive samplers enabled sample storage for up to six months.

Record - 59

TI- Solid-phase microextraction (SPME) of drugs and poisons from biological samples.

AU- Junting, L;Peng, C;Suzuki, O

JN- Forensic Science International

PY- 1998

VO- 97

NO- 2-3

PG- 93-100

AB- A review is presented of the application of SPME to biological samples. Since 1995, SPME has been used to extract drugs and poisons from whole blood, plasma, urine, hair and breath. The fibres, matrices and extraction times used in SPME are discussed. The acceleration of the extraction by agitation, heating, pH adjustment and the addition of salts is also discussed. The advantages of SPME are: it does not need a solvent; it requires a small sample volume (0.1-1 ml); it has a high sensitivity when coupled with GC or GC-MS; and it is simple and convenient for on-site sampling. (33 references).

Record - 60

TI- New methods in MS for the rapid analysis of biomedically relevant substances.

AU- Volmer, DA

JN- CLB CHEMIE IN LABOR UND BIOTECHNIK

PY- 1998

VO- 49

NO- 11

PG- 414-420

AB- A review of the principles and applications of LC-electrospray tandem MS and are presented. LC separations in <10 min were achieved on 3 µm C8, C18 or phenyl columns (5 cm * 4 mm i.d.), usually with acid acetonitrile/H₂O gradients. Twenty-one sulfonamides were separated within 6 min on a C18 column and detected with 200 ms or shorter MS scans; the tandem MS conditions are discussed. For the determination of sulfonamides in milk; initial precursor-ion screening was followed by selected-ion monitoring (SIM) of the pseudomolecular ions [M + H]⁺, and substance-specific product ions were identified by tandem MS. Quinolone antibiotics were separated in 7 min on a phenyl column at pH 2.75; low-energy collisional fragmentation with scanning at constant mass difference was used in identification. The method was applied to urine, fish and milk. SPME on special fibres was combined with LC-tandem MS for determining corticosteroids and steroid conjugates in urine and for characterizing decomposition products of erythromycin in aqueous solution; a diagram of the extraction-HPLC interface is included. (13 references).

Record - 61

TI- Analysis of estrogens and anabolic steroids by SPME with on-fibre derivatization and GC-MS.

AU- Okeyo, PD; Snow, NH

JN- Journal of Microcolumn Separations

PY- 1998

VO- 10

NO- 7

PG- 551-556

AB- Into 1.5 ml aqueous sample solution was immersed an 85 µm polyacrylate fibre or a 60 µm carbowax-divinylbenzene fibre for extraction of oestrogens or anabolic steroids, respectively. After 30 min the fibres were recovered, placed in 5 µl bis(trimethylsilyl)trifluoroacetamide at 70degC for 60 min then placed in a GC system. The analytes were desorbed at 250degC and determined on a 0.5 µm DB-5MS column (30 cm * 0.25 mm i.d.) operated with temperature programming from 100degC (held for 5 min) to 250degC at 30degC/min, then to 300degC at 3degC/min (held for 1.5 min), He as carrier gas (15 psig) and MS detection. The method was also used for the analysis of urine. Calibration graphs were linear for 1-1000 ng/ml testosterone in H₂O, and 20-100 ng/ml 19-nortestosterone, 5-alpha-androstan-17beta-ol-one and 1-dehydrotestosterone in urine, with all analytes being determined against a testosterone-d₃ internal

standard. A detection limit of 200 pg/ml testosterone in H₂O was obtained.

Record - 62

TI- Solid phase microextraction coupled to capillary electrophoresis.

AU- Whang, CW;Pawliszyn, J

JN- Analytical Communications

PY- 1998

VO- 35

NO- 11

PG- 353-356

AB- The performance of a specially designed interface giving zero-dead volume (schematic given) for the online coupling of SPME with capillary electrophoresis was evaluated by the determination of spiked phenols (prepared in 0.01M-HCl saturated with NaCl) in natural water. The SPME fibres (40 µm diameter) were custom-made from silica optical fibres coated with poly(methyl methacrylate) to a thickness <1 µm. SPME was carried out on the sample for 20 min and the SPME fibre was transferred to an interface which facilitated the direct insertion of the thin fibre into the inlet end of the separation capillary. The interface, made from a PTFE block (20 * 30 * 10 mm³), contained conical guide tubes (2 mm o.d.) for the capillaries which entered the block from opposite sides and were separated by a gap of 1 mm. The gap was positioned in the centre of a reservoir for the separation buffer (20 mM-sodium borate adjusted to pH 9.9 with NaOH). A Pt wire (0.5 mm diameter) sealed to the bottom of the reservoir served as the separation anode for capillary electrophoresis. The interface was connected to a separation capillary (40 cm * 75 µm i.d., effective length 32 cm) for capillary electrophoresis at 8-10 kV with detection at 220 nm.

Record - 63

TI- Solid-phase microextraction with rotation of the microfibre.

AU- Geppert, H

JN- Analytical Chemistry

PY- 1998

VO- 70

NO- 18

PG- 3981-3982

AB- Rotation rather than vibration or stirring of the microfibre was used to accelerate absorption in SMPE. The holder was shortened and the upper end was connected to an electric motor with a constant rotation speed of 12 000 rpm. Experiments showed that the absorption efficiency was comparable with the vibration method, and the technique could be automated.

Record - 64

TI- Preparation of a brewed coffee aroma extract by an optimized supercritical carbon dioxide-based process.

AU- Ramos, E;Valero, E;Ibanez, E;Reglero, G;Tabera, J

JN- Journal of Agricultural and Food Chemistry

PY- 1998

VO- 46

NO- 10

PG- 4011-4016

AB- SFE was applied to obtain an extract of brewed coffee with an aroma as similar as possible to that of the original brewed coffee. The extraction method was optimized by a sequential simplex method based on sensorial evaluation. Under the optimum conditions, a 3 ml sample of brewed coffee was extracted with supercritical CO₂ at 60degC and 0.5 g/ml, a flow rate of 1.8 ml/min and an extraction time of 1.42 min. The extract was collected on a piece of filter paper (sime0.06 g) at -25degC. The extract was analysed by purge and trap GC-MS. The loaded filter paper was purged with He (35 ml/min) at 100degC for 15 min. The eluted compounds were trapped on Carbopack B/Carbosieve S-III at room temperature. The compounds were then desorbed at 220degC for 5 min and cryofocused at -100degC. Finally the compounds were desorbed at 200degC for 2 min and analysed using a fused-silica column (50 cm * 220 mum i.d.) coated with BP-20 poly(ethylene glycol) [0.20 mum] and operated with temperature programming from 40degC (held for 5 min) to 180degC (held 15 min) at 15degC/min with He carrier gas (flow rate not given) and 70 eV EIMS detection operated in full-scan mode. Comparison of the results with those obtained for extracts prepared by other techniques, viz. liquid-liquid extraction, headspace SPME, showed considerable difference in composition.

Record - 65

TI- Analysis of volatile fruit components by headspace solid-phase microextraction.

AU- Ibanez, E;LopezSebastian, S;Ramos, E;Tabera, J;Reglero, G

JN- Food Chemistry

PY- 1998

VO- 63

NO- 2

PG- 281-286

AB- Fruit (1 g) was placed in a 20 ml phial with fused silica fibre coated with 100 mum dimethylpolysiloxane and extracted for 30 min at 30degC using headspace-SPME. The VOC extracted were desorbed in the GC injector at 200degC for 15 min in splitless mode for 15 min. The desorbed VOC were analysed by GC on a fused-silica column (50 m *

0.25 mm i.d.) coated with CP-Sil-5CB (0.25 µm), and operated with temperature programming from 50degC (held for 3 min) to 250degC (held for 17 min) at 5degC/min with He as carrier gas (flow rate not given) and FID. GC-MS analysis was also carried out by coupling the above GC system to a 70 eV EIMS detector. Recoveries of 8 VOC (listed) were in the range 10-45%, with results being reproducible.

Record - 66

TI- Simple methods for the extraction and identification of amine malodours from spoiled foodstuffs.

[Full text delivery]

AU- Jones, PRH;Ewen, RJ;Ratcliffe, NM

JN- Journal of Food Composition and Analysis

PY- 1998

VO- 11

NO- 3

PG- 274-279

AB- Sterile potato tubers (Pentland dell cultivar) were inoculated with 0.5 ml *Erwinia carotovora* suspension placed in holes made in the tubers. The inoculated tubers were stored in sealed polythene bags at 20degC under moist conditions (relative humidity >95%) for 14 days. The VOC in headspace above the potatoes were sampled by SPME, (i) passively on a 65 µm partial cross-linked polydimethylsiloxane/divinylbenzene fibres and dynamically (ii) by purging with air (200 ml/min) onto Tenax GR and Chromosorb 103 packed thermal desorption tubes (TDT; experimental details given). The VOC were desorbed by purging with air (400 ml/min, 5 min) directly onto a fused-silica GC column (30 m * 0.32 mm i.d.) coated with SPB-1 sulfur (4 µm; phase ratio beta20). A 5-cm length of the injector end of the column was placed in a liquid N₂ on-column cryogenic trap and the trapped VOC were desorbed by temperature programming the analytical column from 40degC (held for 12 min) to 200degC (held for 2 min) at 10degC/min and 70 eV EIMS detection. Iberian ham was also analysed for VOC in a similar manner. Results (tabulated) showed that NH₃ was the major amine component in all cases, with other amines accounting for <5% of the ammonia content. The potato headspace contained triethylamine while the ham headspace contained methylamine and diethylamine, identified by SPME and by both TDT.

Record - 67

TI- Solvent-free method for the determination of polynuclear aromatic hydrocarbons in waste water by solid-phase microextraction - high-performance liquid chromatography with photodiode-array detection.

AU- Negrao, MR;Alpendurada, MF

JN- Journal of Chromatography, A

PY- 1998

VO- 823

NO- 1-2

PG- 211-218

AB- Treated effluent from a petrochemicals plant (4 ml) were extracted by SPME with a 0.1 mm poly(dimethylsiloxane) fibre at 20degC for 30 min with magnetic stirring. The fibre was then withdrawn into its protective syringe and transferred to the desorption chamber mounted instead of the injection loop on a 6-port valve. The analytes were desorbed by the mobile phase (0.8 ml/min) of a gradient of aqueous 87% acetonitrile for 5 min, increasing to 100% acetonitrile in 20 min, on to a column (25 cm * 4.6 mm i.d.) of 5 µm C18 Vydac 201 TP at 30degC, with detection at 254 nm. The six PAH tested, fluoranthrene, benzo[b]fluoranthrene, benzo[k]fluoranthrene, benzo[a]pyrene, benzo[ghi]perylene and indeno[1,2,3-cd]pyrene, were separated in 24 min. Calibration graphs were linear up to 300 µg/l, with detection limits of 1-5 µg/l. The recoveries of 100 µg/l were 73-104%. At 200 µg/l, the intra-assay RSD were 5.9-12.2% (n = 3) and the inter-assay RSD were 5-20.8% (n = 5 on three days). Each fibre could be used up to 50 times. There were large differences between two fibres from the same manufacturer.

Record - 68

TI- Solid-phase microextraction as a source of data for the design of stripping aeration towers.

AU- Janda, V;Linek, V;Sinkule, J;Vejrosta, J

JN- Journal of Chromatography, A

PY- 1998

VO- 823

NO- 1-2

PG- 523-525

AB- Water containing 50 mg/l each of CHCl₃, trichloroethylene, benzene, tetrachloroethylene, chlorobenzene and bromobenzene was purified in a pilot aeration tower (1.2 m * 29 cm i.d.) packed with plastic rings. The water flow rate at 20degC was 12.7-60 m/h and the countercurrent air flow was 0.6 m/s. Samples were taken at intervals, portions (1 ml) were treated with 2 µg toluene (internal standard) and the solutes were extracted on a 0.1 mm poly(dimethylsiloxane) SPE fibre for 20 min with vigorous stirring. The fibre was transferred to the GC injection port for desorption at 220degC for 3 min. The compounds were analysed on a column (30 m * 0.32 mm i.d.) coated with SPB-1 (4 µm), with He as carrier gas, temperature programming (35degC for 3 min, to 120degC at 10degC/min) and 70 eV EIMS detection in full scan mode. The dissolved O₂ in the samples was measured with a membrane-covered electrode. The rate of removal of the compounds measured thus agreed within 10% of that calculated from equations based on the

kinetics of saturation of the water with O₂.

Record - 69

TI- Headspace solid-phase microextraction for the determination of volatile and semivolatile pollutants in water and air.

AU- Llompart, M;Li, K;Fingas, M

JN- Journal of Chromatography, A

PY- 1998

VO- 824

NO- 1

PG- 53-61

AB- The use of SPME fibres for both extraction and concentration of both semivolatile and water-soluble volatile components of samples in both immersion and headspace modes was studied. Both synthetic and natural samples of water that had been equilibrated with oil or petroleum products were used. The SPME fibres used had a film coating of 0.1 mm of dimethylsilicone. Headspace equilibration was at 95degC for 30 min and SPME equilibration in headspace or immersion was for 20 min at room temperature. For GC analysis, desorption was at 260degC during 3 min in He as carrier gas at 80 ml/min. The analysis column was (30 m * 0.53 mm i.d.) coated with 1.5 µm of SPB-1 and programmed from 40degC (held 5 min) at 7.5degC/min to 200degC or, for analysis of tyre fire particulates, (30 m * 0.25 mm i.d.) coated with DB-5 (0.25 µm) and programmed similarly. The MS detection was by selected-ion monitoring or by total ion scanning for m/z 40-400. Each of the procedure variants yielded similar results except that alkyl-naphthalenes were detected only with use of headspace SPME.

Record - 70

TI- Solid-phase micro-extraction (SPME) fibre performance in turbid aqueous samples.

AU- Rogers, HR;Comber, SDW

JN- Chemosphere

PY- 1998

VO- 37

NO- 8

PG- 1413-1418

AB- The performance of SPME fibres for the extraction of organic pollutants from water samples containing suspended solids was studied. Supelco SPME fibres were exposed to water from the River Thames containing variable solids concentration (up to 10 g/l) spiked with 1,2-dimethylnaphthalene (I) at 1 ppm. The fibres were also repeatedly exposed to a solution of 1 ppm I at a fixed solids level of 1 g/l to investigate the effect of particulates on fibre reusability. After sampling, the fibres were desorbed in a GC

injector at 300degC and I was determined using a DB-1 column (30 m) operated with temperature programming from 50degC (held for 4 min) to 140degC at 20degC/min, then to 200degC at 4degC/min and finally to 300degC at 20degC/min, and FID. No details are given of the carrier gas used. It was found that the extractive efficiency of the fibres decreased with repeated immersions in samples with high solids levels and with increased suspended solids concentrations. Similar experiments with phenol showed no decrease in extractive efficiency with increased solids concentration.

Record - 71

TI- Analysis of volatile halogenated hydrocarbons in water by solid-phase micro-extraction and GC-MS.

AU- Janda, V;Viden, I

JN- Chemicke Listy

PY- 1998

VO- 92

NO- 9

PG- 751-755

AB- Dimethylpolysiloxane (thickness of 0.1 mm) and dimethylpolysiloxane/Carbonex (thickness of 75 mum) fibres from Supelco were used for SPME of halogenated VOC from 2 ml water for 15 min. The resulting extracts were analysed by GC on a 1 mum SPB-1 column (30 m * 0.32 mm i.d.) operated with temperature programming from 35degC (held for 3 min) to 120degC at 10degC/min and 70 eV EIMS detection. The detection limit was below 1 mug/l for most of the halogenated VOC tested.

Record - 72

TI- Determination of chlorophenols using SPME and GC-MS.

AU- Guidotti, M;Ravaioli, G

JN- Annali di Chimica (Rome)

PY- 1998

VO- 88

NO- 9-10

PG- 629-635

AB- Sample (5 ml) was treated with 25 mul of 25% H2SO4 and 1.5 g NaCl and sealed in a 7 ml vial. The solution was magnetically stirred at 1000 rpm and a 85 mum polyacrylate SPME fibre was exposed to the headspace of the vial for 30 min. The analytes were then desorbed from the SPME fibre for 3 min at 290degC in to the injection port of a gas chromatograph. GC analysis was performed on a column (30 m * 0.25 mm i.d.) coated with HP 5-MS (0.25 mum) operated with temperature programming from 40degC (held for 4 min) to 136degC at 12degC/min; and then to 280degC (held for 10 min) at 25degC/min, with He as

carrier gas (1 ml/min) and MS detection. Selected-ion monitoring was performed at m/z 128 and 130 for 2-chlorophenol, 162 and 126 for 2,4- and 2,3-dichlorophenols, 196 and 160 for 2,4,6-trichlorophenol, and 266 and 168 for pentachlorophenol. The method was applied to water from 12 different aqueducts in the Rieti province and water from the river Turano. The method should be suitable for the determination of chlorophenols in drinking and superficial water samples.

Record - 73

TI- Determination of geosmin and 2-methylisoborneol in water using solid-phase microextraction and gas chromatography-chemical ionization/electron impact ionization-ion-trap mass spectrometry.

AU- McCallum, R;Pendleton, P;Schumann, R;Trinh, MU

JN- Analyst (Cambridge, U. K.)

PY- 1998

VO- 123

NO- 10

PG- 2155-2160

AB- The sample (30 ml) was placed in a 40 ml phial and mixed with 10.5 g NaCl and 50 µl d5-geosmin and d3-2-methylisoborneol (internal standards) solution (5 µg/l). The phial was sealed and the solution was heated to 60degC. SPME of geosmin (I) and 2-methylisoborneol (II) was carried out by sampling the headspace above the stirred solution with a 65 µm polydimethylsiloxane/divinylbenzene fibre for 20 min. I and II were desorbed by heating the fibre at 250degC for 4 min and were analysed by GC on a fused-silica column (30 m * 0.25 mm i.d.), coated with DB-5 ms (0.25 µm) and operated with temperature programming from 50degC (held for 4 min) to 150degC at 10degC/min, then to 250degC (held for 3 min) at 25degC/min, He as the carrier gas (100 kPa) and both CI- or EIMS detection for I and II, respectively (details given). The calibration graph was linear for 5-40 ng/l of both I and II, the detection limits were 0.8-0.9 ng/l, the RSD (n = 3-4) were 2-12% and the recoveries were 93-110%. The method was applied to natural and potable waters.

Record - 74

TI- Determination of benzophenone-3 and metabolites in water and human urine by solid-phase microextraction and quadrupole ion trap GC-MS.

AU- Felix, T;Hall, BJ;Brodbeck, JS

JN- Analytica Chimica Acta

PY- 1998

VO- 371

NO- 2-3

PG- 195-203

AB- A method based on SPME and GC-MS was developed for determining

benzophenone-3 (BZ-3, a common ingredient of sunscreens) and its metabolites in urine. The performance of three kinds of SPME fibres were compared and various experimental parameters were optimized. The optimized method was performed by immersing a 65 µm carbowax-divinylbenzene fibre into 4 ml stirred urine for 10 min. The fibre was then transferred to the injection port of the GC and desorption was carried out at 265degC for 13 min. The released benzophenones were analysed on a DB5-MS column with He as carrier gas and temperature programming from 50degC (held for 0.1 min) to 150degC at 30degC/min, and then to 250degC (held for 12 min) at 18degC/min. The quadrupole ion trap MS detector was operated in the electron ionization mode and the following ions were used for quantitative measurements; 151+, 227+ and 228+ for BZ-3, 137+ and 213+ for 2,4-dihydroxybenzophenone (DHB) and 227+ and 244+ for 2,2'-dihydroxy-4-methoxybenzophenone (DHMB). The linear ranges in urine were 10-1000 ng/ml BZ-3 and 50-1000 ng/ml DHB and DHMB and the detection limits were 5-10 ng/ml. The RSD (n = 4) at the 50 ng/ml level were <8%. The method was applied to a human urine sample collected 4 h after the application of a benzophenone containing sunscreen; BZ-3 (at 250 ng/ml) and DHB were detected.

Record - 75

TI- Headspace solid-phase microextraction and gas chromatographic determination of dinitroaniline herbicides in human blood, urine and environmental water.

AU- Guan, F;Watanabe, K;Ishii, A;Seno, H;Kumazawa, T;Hattori, H;Suzuki, O

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1998

VO- 714

NO- 2

PG- 205-213

AB- Water or urine (1 ml), spiked with 0.1-10 ng each of 6 dinitroaniline herbicides and profluratin (internal standard), was mixed with 0.28 g Na₂SO₄ and heated at 70degC in a capped vial. After 10 min, a SPME fibre (10 mm * 100 µm diameter) coated with 100 µm film of polydimethylsiloxane and protected within a syringe needle was injected into the headspace for 30 min and subsequently placed in a GC injection port at 270degC for 5 min. Desorbed analytes were separated on a DB-1 column (30 m * 0.32 mm i.d.; 0.25 µm film thickness) operated with He as carrier gas (80 kPa), temperature programming from 100-300degC (details given) and ECD. Blood (0.5 ml) and 0.5 ml H₂O were heated at 90degC, without Na₂SO₄, prior to GC analysis. Calibration graphs were linear for 0.1-10 ng/ml of herbicide in urine and water and 1-60 ng/0.5 ml in blood, with detection limits of 0.1 ng/ml in urine and water and 0.5 ng/0.5 ml in blood. Intra- and inter-day RSD were <14%. Recoveries from urine and

water were 35-64% and from blood were 3.2-7.2%.

Record - 76

TI- Determination of methylmercury in biological samples and sediments by capillary gas chromatography coupled with atomic- absorption spectrometry after hydride derivatization and solid-phase microextraction.

AU- He, B;Jiang, GB;Ni, ZM

JN- Journal of Analytical Atomic Spectrometry

PY- 1998

VO- 13

NO- 10

PG- 1141-1144

AB- Biological material or sediment was macerated with 10 ml of acetate buffer solution of pH 3 plus 10 drops of HNO₃ for 96 or 24 h, respectively, and 5 ml of the resulting liquid phase was placed in a 50 ml headspace vial and mixed with 5 ml of the buffer solution and 1 g of NaCl. An SPME device made from a microsyringe was used to insert a fused-silica extraction fibre through the septum of the vial and expose a 5 cm length of the fibre (steeped in HF for 3.5 h) to the vapour; then 1 ml of 4% KBH₄ solution was injected into the vial, and after 90 min the fibre was withdrawn and inserted into the injector (200degC) of the gas chromatograph, in which it was kept for 2 min. The CP-SIL 5CB fused-silica capillary column (10 m * 0.25 mm i.d.) was operated at 40degC with AAS detection at 253.7 nm in a T-shaped quartz absorption cell kept typically at 700degC (cf. Jiang et al., Fresenius' Z. Anal. Chem., 1989, 334, 27). Calibration was effected with standard solutions; the absorbance (peak height) varied linearly with the amount of methylmercury chloride in the vial, and the detection limit was 26 ng. The RSD (n = 11) for 2 mug of methylmercury chloride in 10 ml of H₂O was 91%. Results for mink skin, mink fur and soil agreed with those obtained after a much less economical liquid-liquid extraction.

Record - 77

TI- Solvent microextraction of polycyclic aromatic hydrocarbons from mineral oils.

AU- Slebioda, M;Kot, A;Kolodziejczyk, AM

JN- Chemia Analityczna (Warsaw)

PY- 1998

VO- 43

NO- 5

PG- 867-872

AB- Mineral oil (3 g) was diluted with CH₂Cl₂ and made up to 10 ml. A 200 µl portion of the solution was shaken for 5 min with 1000 µl

cyclohexane and 1000 µl DMSO. After phase separation, a 20 µl portion of the lower layer was analysed by HPLC on a Vydac 201TP54 column (25 cm * 4.6 mm i.d.) with gradient elution (1.5 ml/min) with acetonitrile/H₂O (1:1; solvent A) and acetonitrile (solvent B) from 0% B (0.3 min) to 100% B (10 min) and fluorescence detection (wavelengths tabulated). Recoveries ranged from 57-88% in the range of tens µg/kg up to hundreds of mg/kg. Reproducibility (n = 7) was better than 5%. The determination of PAH at very low mg/kg level was possible. The method was applied to the determination of PAH in the products from one of the Polish petroleum refineries.

Record - 78

TI- Application of solid-phase microextraction and gas chromatography-time-of-flight mass spectrometry for rapid analysis of flavour volatiles in tomato and strawberry fruits.

AU- Song, J;Fan, L;Beaudry, HM

JN- Journal of Agricultural and Food Chemistry

PY- 1998

VO- 46

NO- 9

PG- 3721-3726

AB- Fruit samples were placed in a jar and after the container had been flushed for 60 min, SPME fibres (PGMS/DVB 1 cm * 65 µm thickness) were exposed to the effluent for 4 min and the trapped volatile flavour compounds (VFC) were desorbed directly through the glass-lined GC injector port at 200degC onto a fused-silica column (30 m * 0.25 mm i.d.) coated with HP-5 (0.25µm), operated with temperature programming from 40degC to 250degC (held for 1 min) at 60degC/min, with He as carrier gas (1.5 ml/min) and 70 eV EIMS detection. Results for VFC are tabulated and agreed well with published results obtained by GC/FID analysis.

Record - 79

TI- Strategies for the analysis of chlorobenzenes in soils using solid-phase microextraction coupled with gas chromatography - ion-trap mass spectrometry.

AU- Sarrion, MN;Santos, FJ;Galceran, MT

JN- Journal of Chromatography, A

PY- 1998

VO- 819

NO- 1-2

PG- 197-209

AB- Two methods were used for the SPME, both using 0.1 mm film thickness poly(dimethylsiloxane) fibres (results with 7 µm film fibres were more variable). In the headspace method, soil samples (0.03-0.1 g)

and 0.2 ml of H₂O were heated in a 40 ml phial at 30degC for 1 h in the presence of the fibre. In the direct method, samples (0.03 g) were stirred magnetically at 1000 rpm with 40 ml of aqueous 30% acetone at 30degC for 50 min in the presence of the fibre. Adsorbed compounds were desorbed in the injection port at 250degC for 1 min and were analysed on a column (30 m * 0.25 mm i.d.) coated with DB-5 MS (90.25 µm), with He (31 cm/s) as carrier gas, temperature programming (50degC for 1 min, then in three stages to 280degC, details given) and ion-trap EIMS detection in positive mode, with selected-ion monitoring of the two most abundant ions. Calibration graphs were linear for 100-30 000 pg of the trichloro-, tetrachloro- and pentachlorobenzenes injected, with detection limits of 30-100 ppb. In five analyses of a candidate reference clay soil, the results by the two methods were in good agreement, with RSD of 2-8%. They also agreed well with the means of an inter-laboratory exercise, with nine participants, mainly using a Soxhlet extraction method.

Record - 80

TI- Effect of organic matter content in the trace analysis of triazines in various types of soils with GC-NPD.

AU- Molins, C;Hogendoorn, EA;Heusinkveld, HAG;vanBeuzekom, AC;vanZoonen, P;Baumann, RA

JN- Chromatographia

PY- 1998

VO- 48

NO- 5-6

PG- 450-456

AB- Interference by organic compounds in determination of atrazine (I) and simazine (II) in soils by GC was studied. Standard humic and real forest and sandy soils were used and their water and total organic matter contents were determined by thermogravimetry. Soil extracts were obtained by microwave-assisted solvent extraction (SPME) at 950 W during 20 min at 100degC and 950 kPa and metazachlor (0.25 µg) in acetone was added as internal standard. Analytes were transferred into dichloromethane for SPE on a 100-mg silica cartridge from which sample elution was with hexane/acetone (3:1) for GC a fused-silica column (25 m * 0.32 mm i.d.) coated with HP-1 (0.17 µm) and operated with temperature programming from 70degC in three steps (program details given) to 270degC (held for 15 min) with He as carrier gas (2 ml/min) and N-P detection. Results were evaluated relative to analyses by GC with positive-ion CIMS detection. Down to 2 ppb of I and II could be determined directly in SPME extracts of soils of <5% organic content. At >5% organic content inclusion of the SPE step was vital. Recoveries of 2-50 ppb of the triazines were 70-100% with RSD of 5.1-9.5%.

Record - 81

TI- Solid-phase microextraction and gas chromatography for rapid analysis of pesticides.

AU- Miede, C;Dugay, J

JN- Analisis

PY- 1998

VO- 26

NO- 6

PG- M137-M143

AB- An overview of the application of the cited procedure to aqueous samples is given. SPME and the effects of various experimental parameters on SPME are described. An example of a validation study and examples of SPME-GC determinations of pesticides containing N, P and Cl, e.g., in pure, drinking, river and waste water, are given. When MS detection is used pesticide detection limits of 5-100 ng/l can be obtained. (13 references).

Record - 82

TI- Determination of amphetamine, methamphetamine and dimethylamphetamine in human urine by solid-phase microextraction-gas chromatography-mass spectrometry.

AU- Myung, SW;Min, HK;Kim, SK;Kim, MS;Cho, JB;Kim, TJ

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1998

VO- 716

NO- 1-2

PG- 359-365

AB- Samples (3 ml) were mixed with 15 ml 5M-KOH and 0.9 g of NaCl and stirred magnetically for 30 min, with a 0.1 mm coated poly(dimethylsiloxane) fibre immersed in the solution. The analytes were thermally desorbed in the injection port at 250degC and analysed on a column (20 m * 0.2 mm i.d.) coated with Ultra-2 (0.33 µm), with He (0.9 ml/min) as carrier gas, temperature programming (100degC for 1 min, to 300degC at 20degC/min, held for 5 min) and 70 eV EI MS detection, with the transfer line at 290degC. The selected ions measured were m/z = 44, 58 and 72 respectively; all three compounds also gave a peak at m/z = 91. Calibration graphs were linear for 0.05-1 µg/ml of all three drugs, with detection limits of 10 ng/ml for amphetamine and methamphetamine and 1 ng/ml for dimethylamphetamine. Within-day RSD for 0.3-1 µg/ml of the three drugs were 1.4-6.6% (n = 3). The method was suitable for rapid screening tests.

Record - 83

TI- Simple analysis of tetracyclic antidepressants in blood using headspace-solid-phase microextraction and GC-MS.

AU- Namera, A;Watanabe, T;Yashiki, M;Iwasaki, Y;Kojima, T

JN- Journal of Analytical Toxicology

PY- 1998

VO- 22

NO- 5

PG- 396-400

AB- A method was developed for the determination of maprotiline (I), mianserin (II) and setiptiline (III) in blood. Blood (0.5 g), internal standard (imipramine; 50 µg/ml, 5 µl) and 0.5 ml 1N-NaOH were sealed in a vial and heated at 120°C. The needle of a SPME device was exposed for 45 min in the headspace of the vial. The needle was removed and the compounds absorbed on the SPME fibre were desorbed by exposing the fibre for 5 min into the injection port of the GC-MS system. GC was performed on a column (30 m * 0.32 mm i.d.) coated with SPB-1 (0.25 µm) operated with temperature programming from 100°C (held for 5 min) to 280°C at 20°C/min, with He as carrier gas (0.8 ml/min) and MS detection with selected-ion monitoring. Quantitation was performed at m/z 277 for I; m/z, 193 for II; m/z 261 for III; and m/z 234 for the internal standard. Calibration graphs were linear from 0.005-5 µg/g for II and III, and from 0.025-25 µg/g for I. There was no interference. The method was applied to a suspected case of acute III poisoning; III was detected in the left and right heart blood samples of the victim at concentrations of 1.77 and 0.78 µg/g, respectively.

Record - 84

TI- Bioanalysis of drugs.

AU- Soltes, L

JN- Chemicke Listy

PY- 1998

VO- 92

NO- 3

PG- 209-214

AB- A review is presented of the determination of drugs and their metabolites in biological materials by HPLC with discussion of various sample preparation techniques including SPME. (19 references).

Record - 85

TI- Solid-phase microextraction - gas chromatography - direct deposition infra-red spectrometry as a convenient method for the determination of volatile compounds from living organisms.

AU- Auger, J;Rousset, S;Thibout, E;Jaillais, B

JN- Journal of Chromatography, A

PY- 1998

VO- 819

NO- 1-2

PG- 45-50

AB- A poly(dimethylsiloxane) fibre (0.1 mm o.d.) was introduced into a 4 ml box close to a single male asparagus fly. After 1 min exposure, the fibre was withdrawn and adsorbed compounds were desorbed into the GC injector at 200degC for 2 min. The compounds were analysed on a column (20 m * 0.22 mm i.d.) coated with HP-1 (0.33 µm), with He (1 ml/min) as carrier gas, temperature programming (40-200degC at 2degC/min) with the transfer line at 250degC. The compounds were analysed by FTIR with direct deposition of the eluate on the Zn-Se window at 77 K. The method was used to identify the male pheromone, with sensitivity in the pg range, similar to that of GC-MS. The SPE step prevented the deposition of ice on the cold window.

Record - 86

TI- Solid-phase microextraction and its utilization in environmental analysis.

AU- Sedlakova, J;Matisova, E;Slezackova, M

JN- Chemické Listy

PY- 1998

VO- 92

NO- 8

PG- 633-642

AB- A review is presented of the use of SPME for the preconcentration of organic pollutants in environmental matrices. The use of stationary phases coating fused silica fibres to extract analytes from samples are discussed, after which the sorbed substances are introduced into the GC system by thermal desorption. (100 references).

Record - 87

TI- Solid-phase microextraction for determining the binding state of organic pollutants in contaminated water rich in humic organic matter.

AU- Poerschmann, J;Kopinke, FD;Pawliszyn, J

JN- Journal of Chromatography, A

PY- 1998

VO- 816

NO- 2

PG- 159-167

AB- The binding of phenols and PAH in contaminated water rich in humic organic matter was studied by SPME in the conventional and headspace modes (both modes gave similar results and agreed well with liquid-

liquid extraction). Deuterated phenols and PAH were used as internal standards at the 10 ppm to 10 ppb levels. Following extraction the phenols and PAH were analysed on fused-silica columns (30 m * 0.25 mm i.d.) coated with HP-5 (0.25 µm) or SPB-5 (0.25 µm) operated with temperature programming from 40degC (held for 3 min) to 290degC (held for 15 min) at 10degC/min. The injector temperature was 300degC for both the desorption of the fibres (7 µm) and for solvent injection (details of carrier gas are not given). Detection was by SIMS. Samples rich in dissolved organic matter were diluted progressively to give the freely-dissolved fraction of an unknown analyte and its partition coefficient.

Record - 88

TI- Simultaneous analysis of trace chemical substances in environmental water by solid-phase microextraction.

AU- Kadokami, K

JN- Bunseki

PY- 1998

NO- 8

PG- 608

AB- A discussion is provided on the application of SPME in the preconcentration of substances like endocrine disturbing chemicals, especially agrochemicals for GC-MS, with a mention on the problems with interfering particulates, detection limits and recoveries.

Record - 89

TI- Analysis of aromatic hydrocarbons by solid-phase microextraction and gas chromatography.

AU- Fang, RB;Zhang, WB;Zhang, KL;Lian, KH

JN- Fenxi Huaxue

PY- 1998

VO- 26

NO- 8

PG- 1029-1032

AB- The cited extractor comprises a graphite stick (diagram shown), similar to the device supplied by Supelco of USA, with which aromatic hydrocarbons in the atmosphere are adsorbed for their determination after desorption in the gasification chamber of a gas chromatograph. As an example, SPME of benzene, toluene, ethylbenzene, o-, m- and p-xylenes was directly performed in a paint-spraying workshop. Calibration graphs were linear from 0.002-2000 mg/m³ for each analyte. The apparatus could also be applicable to the analysis of soil and water.

Record - 90

TI- Urinary organic acid screening by solid-phase microextraction of the methyl esters.

AU- Liebich, HM;Gesele, E;Woell, J

JN- Journal of Chromatography, B: Biomedical Applications

PY- 1998

VO- 713

NO- 2

PG- 427-432

AB- Urine (2 ml) was mixed (under stirring) with sodium carbonate (20 mg), and 30 mg trimethyloxonium tetrafluoroborate (TMO) was added in 5 portions, and after 1 min, the solution was neutralized with NaHCO₃. The process of adding 30 mg TMO and the NaHCO₃ was repeated twice. Then a further 30 mg TMO was added, followed by sodium carbonate (20 mg). Within 4 min, a further 30 mg of TMO was added, followed by NaHCO₃, and the solution was incubated for 2 min at 100degC. A polyacrylate fibre (85 µm thickness) was then used to extract the organic acid methyl esters from the urine by SPME. The analytes were desorbed in the injection port. Analysis was performed on a OV1701 column (25 m * 0.25 mm i.d.), temperature programming from 40degC to 280degC at 2degC/min. with He as carrier gas (flow rate not stated) and FID. Mass spectrometry was performed with use of a Finnigan TSQ 70 instrument. Preparation time was up to GC-MS was 40 min. The organic acids in urine were well separated and 29 methyl esters were identified by GC-MS.

Record - 91

TI- Sampling techniques for gas-chromatographic - mass-spectrometric analysis of long-chain free fatty acids from insect exocrine glands.

AU- Maile, R;Dani, FR;Jones, GR;Morgan, ED;Ortius, D

JN- Journal of Chromatography, A

PY- 1998

VO- 816

NO- 2

PG- 169-175

AB- Fatty acids in insect exocrine glands were sampled by SPME on fibres coated with either polydimethylsiloxane (7 µm), polyacrylate (85 µm) or carbowax-divinylbenzene (65 µm) or by a solid injector technique. Both techniques gave reliable results if deactivated injection liners and glass wool in the injector were used. Deactivated pre- and post-column prevent the detection of long-chain fatty acids present in mug amounts. Avoiding preheating the sample in the injector and removing the glass powder of the sample capillaries after each injection enhanced results from the solid injector. For SPME, adsorption from headspace and moderate heat gave better results than high-temperature heating. Both methods were preferable to

extraction with solvents.

Record - 92

TI- Mobile sampling for SPME [solid-phase micro-extraction]. Rapid and safe on-site sampling technique for field analysis.

AU- Schaefer, C;Shirey, R

JN- LaborPraxis

PY- 1998

VO- 22

NO- 7-8

PG- 82-83

AB- Details and a diagram are given of a portable field sampler of syringe type equipped with a quartz fibre coated with a GC stationary phase or packing. The fibre is conditioned at 300degC for 30 min in the GC injector block before use. After being used to extract VOC from environmental materials the fibre is withdrawn through a needle and a sealing septum into the sampler housing and thus protected from the atmosphere. Subsequently, the needle is positioned within the GC injector block and the fibre is pushed out to allow desorption of the analytes. The sample can be used for 50-100 extractions. A PDMS-Carboxen (PDMS is polydimethylsiloxane) fibre is used for trace analysis of volatile compounds; a PDMS-divinylbenzene fibre is used for general purposes. Minimal loss of 31 analytes occurred when a loaded PDMS-Carboxen fibre was stored at -4degC for 3 days.