

**SPME 2000- September**  
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(1) TI: Application of solid-phase microextraction-gas chromatography- mass spectrometry to characterize intermediates in a joint solar-microbial process for total mineralization of Aroclor 1254. AU: Rhofir\_C, Hawari\_JJN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.53-61 IS: 0021-9673 DT: Article NA: jalal.hawari@NRC.ca, Biotechnol. Res. Inst., National Res. Council Canada, Montreal, PQ H4P 2R2, Canada CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: A combined solid-phase microextraction-GC-MS analytical technique was used to monitor the formation of metabolites in the biodegradation of biphenyl, which were originally obtained from the solar photodechlorination of Aroclor 1254 by *Pseudomonas pseudocaligenes* KF707 and *Burkholderia* sp LB400. In both cases, the following metabolites were detected: 2-hydroxybiphenyl (2-OH-BP), 2,3-dihydroxybiphenyl (2,3-di-OH-BP), and benzoic acid, which was detected as its benzoate derivative 1-methylethyl (isopropyl) benzoate. A time course study for the formation and disappearance of these metabolites was used to construct a degradation pathway, which in both cases, involved the formation of 2-OH-BP and 2,3-di-OH-BP.

(2) TI: Determination of butyltin species in water and sediments by solid-phase microextraction-gas chromatography-flame ionization detection. AU: Millan\_E, Pawliszyn\_J JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.63-71 IS: 0021-9673 DT: Article NA: janusz@sciborg-uwaterloo.ca, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: A procedure for determination of tetraethyltin (TeET) and tetrabutyltin (TeBT) in water by solid-phase microextraction (SPME) using the headspace approach has been developed. The method has been adapted for the simultaneous determination of mono-, di- and tributyltin species (MBT, DBT and TBT) after derivatization with sodium tetraethylborate in water and sediment samples. The analytical procedures were optimized with respect to stirring conditions, extraction time and extraction temperature. The pH and the amount of derivatizing reagent were also considered in derivatization reaction procedures. The analysis was carried out using gas chromatography equipped with flame ionization detection. The detection limits obtained for TeET and TeBT, in equilibrium conditions (room temperature for TeET and 40degC for TeBT) were 28 and 20 ng/l (as Sn), respectively. The detection limit for butyltin species in water, which was limited by signals which are non-specific for the tin compounds and the sensitivity of the FID system, was found ca. 1 mug/l (as Sn). The SPME method was validated for analysis of sediments by analyzing the certified reference material PACS-2 finding a good agreement with the certified values.

(3) TI: Analysis of volatile contaminants in vegetable oils by headspace solid-phase microextraction with Carboxen-based fibres. AU: Page\_BD, Lacroix\_G JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.79-94 IS: 0021-9673 DT: Article NA: denis\_page@hc-sc.gc.ca, Health Protection Branch, Food Directorate, Health Canada, Ottawa, ON K1A 0L2, Canada CO: Presented at ExTech 1999 Symposium: Advances in

Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: The headspace solid-phase microextraction (HS-SPME) efficiencies from vegetable oil of the recently available Carboxen-poly(dimethylsiloxane) (PDMS) and divinylbenzene-Carboxen-PDMS fibres were found to be much greater than those of the PDMS fibre for a number of volatile contaminants. Using these Carboxen-based fibres, the commonly used HS-SPME equilibration times for aqueous matrices of 30-45 min at room temperature for a number of halogenated and aromatic analytes with volatilities ranging from 1,1-dichloroethylene to hexachlorobenzene were found to be insufficient for the effective extraction of the less volatile analytes from vegetable oil. HS-SPME at 100degC for 45 min, followed by rapid cooling to 0degC with a 10 min continuing extraction, however, significantly increased the SPME efficiencies for the less volatile analytes. Spiking solutions were prepared in vegetable oil instead of methanol as the latter was found to displace analytes from the Carboxen material. Using either of the Carboxen-based fibres and SPME at 100degC, all the target analytes could be determined at low or sub-mug kg-1 with repeatability <=10%, even though an equilibrium SPME of the less volatile analytes was not achieved.

(4) TI: Solid-phase microextraction-gas chromatography-mass spectrometry of biologically active substances in water samples.

AU: Moeder\_M, Schrader\_S, Winkler\_M, Popp\_P JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.95-106 IS: 0021-9673 DT: Article NA: moeder@ana.ufz.de, Dept. Anal. Chem., Centre Environ. Res. Leipzig-Halle, 04318 Leipzig, Germany CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: A solid-phase microextraction (SPME) method for determining trace amounts of polar, biologically active substances in water systems was developed and compared with solid-phase extraction followed by derivatization and GC-MS. SPME was examined with respect to the simultaneous determination of pharmaceuticals such as ibuprofen, paracetamol, phenazone, carbamazepine, and nonylphenols known to be xenoestrogens. The extraction performance of different SPME fibre coatings was studied. Coatings like polyacrylate and Carbowax-divinylbenzene proved to be the best suited. The optimum extraction time was found to be 30 min and the detection limits were between 0.2 and 50 mug/l. Low concentrations of accompanying organic matter did not impair these limits. One of the main pharmaceutical contaminants found in ground and river water around Leipzig (Germany) was ibuprofen, with a concentration in the ng/l range. The enantioselective metabolism of ibuprofen was investigated.

(5) TI: Development of a headspace solid-phase microextraction procedure for the determination of free volatile fatty acids in waste waters.

AU: Abalos\_M, Bayona\_JM, Pawliszyn\_J JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.107-115 IS: 0021-9673 DT: Article NA: Environ. Chem. Dept., IIQAB-CID-CSIC, 08034 Barcelona, Spain CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: An analytical procedure based on headspace solid-phase microextraction (SPME) coupled to GC-flame ionization detection/negative-ion chemical ionization mass spectrometry has been developed for the determination of free volatile fatty acids (C2-C7) in waste water samples. Five different coatings have been evaluated and polydimethylsiloxane-Carboxen

was the only fiber that allows a successful extraction of the shortest chain fatty acids (acetic and propionic). Several parameters such as extraction time and temperature, desorption conditions, agitation speed and sample volume have been optimized using a polydimethylsiloxane-Carboxen fiber. The linear dynamic range was over two-four orders of magnitude, depending on the acid. Procedural detection limits were in the low to medium  $\mu\text{g/l}$  levels and the RSDs were between 5.6% and 13.3%. To evaluate the applicability of the developed SPME procedure on real samples, fermented urban wastewaters were analysed.

(6) TI: Screening of Brazilian fruit aromas using solid-phase microextraction-gas chromatography-mass spectrometry. AU: Augusto\_F, Valente\_ALP, dosSantosTada\_E, Rivellino\_SR JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.117-127 IS: 0021-9673 DT: Article NA: Inst. Chem., State Univ. Campinas (Unicamp), 13083-907 Campinas, SP, Brazil CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: Manual headspace solid-phase microextraction (SPME) coupled to gas chromatography-mass spectrometry (GC-MS) was used for the qualitative analysis of the aromas of four native Brazilian fruits: cupuassu (*Theobroma grandiflorum*, Spreng.), caja (*Spondias lutea*, L.) siriguela (*Spondias purpurea*, L.) and graviola (*Anona reticulata*, L.). Industrialized pulps of these fruits were used as samples, and extractions with SPME fibers coated with polydimethylsiloxane, polyacrylate, Carbowax and Carboxen were carried out. The analytes identified included several alcohols, esters, carbonyl compounds and terpenoids. The highest amounts extracted, evaluated from the sum of peak areas, were achieved using the Carboxen fiber.

(7) TI: Automated in-tube solid-phase microextraction-high-performance liquid chromatography for carbamate pesticide analysis. AU: Gou\_Y, Eisert\_R, Pawliszyn\_J JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.137-147 IS: 0021-9673 DT: Article NA: Guelph-Waterloo Centre Graduate Work Chem., Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999 AB: In-tube solid-phase microextraction (SPME) is an automated version of SPME that can be easily coupled to a conventional HPLC autosampler for on-line sample preparation, separation and quantitation. It has been termed "in-tube" SPME because the extraction phase is coated inside a section of fused-silica tubing rather than coated on the surface of a fused-silica rod as in the conventional syringe-like SPME device. The new in-tube SPME technique has been demonstrated as a very efficient extraction method for the analysis of polar and thermally labile analytes. The in-tube SPME-HPLC method used with the FAMOS autosampler from LC Packings was developed for detecting polar carbamate pesticides in clean water samples. The main parameters relating to the extraction and desorption processes of in-tube SPME (selection of coatings, aspirate/dispense steps, selection of the desorption solvents, and the efficiency of desorption solvent, etc.) were investigated. The method was evaluated according to the reproducibility, linear range and limit of detection. This method is simple, effective, reproducible and sensitive. The relative standard deviation for all the carbamates investigated was between 1.7 and 5.3%. The method showed good linearity between 5 and 10 000  $\mu\text{g/l}$  with correlation coefficients between 0.9824 and 0.9995. For the carbamates

studied, the limits of detection observed are lower than or similar to that of US Environmental Protection Agency or National Pesticide Survey methods. Detection of carbaryl present in clean water samples at 1 µg/l is possible.

(8) TI: Developments in extraction techniques and their application to analysis of volatiles in foods.

AU: Sides\_A, Robards\_K, Helliwell\_S JN: Trends in Analytical Chemistry, 2000, Vol.19, No.5, pp.322-329 IS: 0165-9936 DT: Article NA: krobards@csu.edu.au, School Sci. Technol., Wagga Wagga, NSW 2678, Australia AB: Recent developments in analysis of aroma components in foods are reviewed. Aroma compounds are most closely associated with the volatile fraction of foods. Preliminary isolation remains an essential step in such procedures despite rapid developments in measurement techniques. Traditional methods of isolating volatile components have recently been complemented by SPME. GC and GC-MS remain the dominant techniques for measurement of the extracted compounds although new electronic noses are promising techniques. Relating the results from instrumental measurements to human perception requires careful control to ensure valid comparisons. The application of multivariate statistics is important in this respect.

AN: aroma compounds, detmn. of volatile, from food, extraction in, review MX: foods, detmn. of volatile aroma compounds from, extraction in, review.

(9) TI: Speciation of alkyllead and inorganic lead by derivatization with deuterium-labelled sodium tetraethylborate with SPME- GC/MS.

AU: Yu\_XM, Pawliszyn\_J JN: Analytical Chemistry, 2000, Vol.72, No.8, pp.1788-1792 IS: 0003-2700 DT: Article NA: Guelph-Waterloo Center Graduate Work Chem., Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada AB: A method for full speciation and determination of alkyllead and inorganic lead(II) in aqueous samples was developed. This was accomplished by in situ derivatization with deuterium-labelled sodium tetraethylborate  $\text{NaB}(\text{C}_2\text{D}_5)_4$  (DSTEB). The derivatization was carried out directly in the aqueous sample and the derivatives were extracted from the headspace by a solid-phase microextraction (SPME) fibre. The extracted analytes were then transferred to a GC/MS or a GC/FID for separation and detection. The research presented demonstrates that SPME and the derivatization reagent DSTEB can be used successfully for the speciation of  $\text{Pb}^{2+}$ ,  $\text{Pb}(\text{CH}_3)_3^+$ ,  $\text{Pb}(\text{C}_2\text{H}_5)_3^+$ , and  $\text{Pb}(\text{C}_2\text{H}_5)_4$  in water samples. All derivatives,  $\text{Pb}(\text{C}_2\text{D}_5)_4$ ,  $(\text{CH}_3)_3\text{Pb}(\text{C}_2\text{H}_5)$ ,  $(\text{C}_2\text{H}_5)\text{Pb}(\text{C}_2\text{D}_5)$ , and  $\text{Pb}(\text{C}_2\text{H}_5)_4$ , are separated using an SBP-5 column. This method was applied to monitor degradation of tetraethyllead in water. This is the first report of ethylation by DSTEB for full speciation of methyllead, ethyllead, and inorganic lead compounds. This approach can be extended to other organometallic compounds as demonstrated for ethyltin speciation. This full speciation method will aid in monitoring occurrence, pathways, toxicity, and biological effects of these compounds in the environment. It is easily adopted for field analysis.

AN: lead (7439-92-1) speciation of, in water, by GC-MS, reagents for, SPME in

MX: waters, natural, speciation of lead in, by GC-MS, reagents for, SPME in(10)

(11) TI: Analysis of brandy aroma by solid-phase microextraction and liquid-liquid extraction.

AU: Ebeler\_SE, Terrien\_MB, Butzke\_CE JN: Journal of the Science of Food and Agriculture, 2000, Vol.80, No.5, pp.625-630 IS: 0022-5142 DT: Article NA: Dept.

Viticulture and Enol., Univ. California, Davis, CA 95616, USA AB: Headspace SPME and continuous liquid-liquid extraction (LLX) with Freon, methods have evaluated for the extraction of aroma volatiles (AV) in brandy. Brandy samples were diluted to 100 ml/l ethanol and a 3 ml portion was transferred to a sealed vial and heated on a water bath at 50degC. A preconditioned polydimethylsiloxane fibre (100 µm film thickness) was placed in the headspace over the sample and the AV were adsorbed for 30 min. The fibre was retracted and placed into the injection port of a GC. The desorbed AV were analysed on a fused-silica column (30 m \* 0.32 mm i.d.) coated with DB Wax (0.25 µm), operated with temperature programming from 50degC (held for 2 min) to 210degC at 2degC/min, with He as carrier gas (30 cm/s) and FID. Diluted brandy samples (50 ml) were also subjected to LLX with 50 ml redistilled Freon II for 72 h. The Freon extract was allowed to evaporate to 0.5 ml concentrate in a Kuderna-Danish evaporator, containing boiled chips, in a water bath at 30degC. The Freon extract was concentrated to 0.25 ml under a stream of N<sub>2</sub> gas at 2 ml/min and 1 µl portions of the concentrate was analysed by GC as for the SPME analytes (above). Results (tabulated) showed that the LLX extracted the higher alcohols more efficiently than SPME, but the latter was more selective for esters and acids. The differences in concentration of 4 aroma volatiles, viz. hexanol, 3-methylbutyl acetate, 3-methylbutanol and 3-methylbutyl octanoate for 2 *Vitis vinifera* species-containing brandies were observed.

(12) TI: Surface characterization of commercial fibers for solid-phase microextraction and related problems in their application.

AU: HaberhauerTroyer\_C, Crnoja\_M, Rosenberg\_E, Grasserbauer\_M

JN: Fresenius' Journal of Analytical Chemistry, 2000, Vol.366, No.4, pp.329-331 IS: 0937-0633 DT: Article NA: Inst. Anal. Chem., Vienna Univ. Technol., 1060 Vienna, Austria AB: The surfaces of commercially available polydimethylsiloxane (PDMS) and Carboxen-PDMS fibers for solid-phase microextraction (SPME) were investigated by optical and electron microscopy.

Damage to the coating as well as contamination of new fibers and a highly variable number of pores in Carboxen-PDMS coatings were observed. Together with the contamination of the fibers during their use with metallic particles originating from the SPME fiber holder they are possible explanations for the problems encountered in the analysis of organolead, organotin and organosulfur compounds, such as artifact formation and low repeatability.

(13) TI: Optimization of extraction conditions for low-molecular-weight analytes using solid-phase microextraction.

AU: Shirey\_RE JN: Journal of Chromatographic Science, 2000, Vol.38, No.3, pp.109-116 IS: 0021-9665 DT: Article NA: Supelco Inc., Supelco Park, PA 16823, USA AB: A group of volatile analytes under a molecular weight of 90 and representing 11 organic classes are extracted using identical conditions with 6 different solid-phase microextraction fiber coatings. The amount of each of the analytes extracted by the various fibers is shown. The effects of sample modifiers, such as pH and ionic strength, on the recovery of the analytes are presented. A comparison of headspace and immersion extraction techniques is shown.

(14) TI: Analysis of tetrahydrofuran and methanol in distillation residue samples by automated headspace solid-phase microextraction-gaschromatography with flame ionization detection.

AU: Gavlick\_WK JN: Journal of Chromatographic Science, 2000, Vol.38, No.3, pp.117-121 IS: 0021-9665 DT: Article NA: Monsanto, St Louis, MO 63167, USA AB: Automated headspace SPME coupled with GC and FID is used to determine the amounts of methanol and THF in distillation residue samples from a proprietary chemical reaction. A 65- $\mu$ m polydimethylsiloxane/divinylbenzene SPME fiber is used to perform the extractions. Optimized extraction conditions for each analyte are determined using a parts-per-million-level methanol in water standard and a parts-per-million-level THF in water standard. The amount of methanol and THF in distillation residue samples is quantitated by both standard addition and external standard calibration curve. The two methods of quantitation are compared.

(15) TI: An automated solid-phase microextraction-gas chromatography-mass selective detection approach for the determination of sugar-amino acid reaction mechanisms.

AU: Coleman\_WM\_I, Lawson\_SN JN: Journal of Chromatographic Science, 1999, Vol.37, No.10, pp.383-387 IS: 0021-9665 DT: Article NA: R.J. Reynolds Tobacco Co., Winston-Salem, NC 27102-1487, USA AB: Confirmation of the proposed mechanisms of Strecker aldehyde involvement in the production of branched-chain alkyl-substituted pyrazines is easily accomplished using an automated solid-phase microextraction-gas chromatography-mass selective detection (AutoSPME-GC-MSD) approach. The method is relatively elementary with minimal sample preparation. Microwave heated aqueous formulations require only the addition of NaCl to "salt-out" the analytes prior to analysis by AutoSPME-GC-MSD. A 100  $\mu$ m polydimethylsiloxane fibre with accompanying fibre vibration can extract sample quantities of the compounds of interest in as little as 1 min. Such sensitivity negates the requirements for time and reagent-consuming sample extraction via organic solvents or concentration using heat. This approach, therefore, should find additional expanded applications in establishing reaction pathways.

(16) TI: Examination of the enantiomeric distribution of certain monoterpene hydrocarbons in selected essential oils by automated solid-phase microextraction-chiral gas chromatography-mass selective detection.

AU: Coleman\_WM\_I, Lawrence\_BM JN: Journal of Chromatographic Science, 2000, Vol.38, No.3, pp.95-99 IS: 0021-9665 DT: Article NA: R.J. Reynolds Tobacco Co., Winston-Salem, NC 27102-1487, USA AB: A viable approach for the determination of sources of essential oils based on automatic injection solid-phase microextraction-chiral gas chromatography-mass selective detection is demonstrated. With no sample preparation, it is shown that the source of essential oils such as peppermint, spearmint, and rosemary can be easily distinguished. Short fiber exposure times of approximately 6 sec to the headspace above submicroliter quantities of the selected oils are all that is required to obtain both the required sensitivity and resolution to afford analyses with excellent reproducibilities (RSD consistently less than 5.0%).

(17)

(18) TI: Microwave distillation-solid-phase microextraction gas chromatographic analysis of geosmin and 2-methylisoborneol in catfish.

AU: Grimm\_CC, Lloyd\_SW, Zimba\_PV, Palmer\_M

JN: American Laboratory (Shelton, Connecticut), 2000, Vol.32, No.3, p.40, 42, 44, 46, IS: 0044-7749 DT: Article NA: cgrimm@nola.srrc.usda.gov, USDA-ARS-SRRC, New Orleans, LA 70124, USA

AB: Catfish fillet (20 g) was chopped, heated in a microwave, and the condensate was collected using a condenser and a chilled water bath (diagram given). After rinsing the condenser with 5 ml H<sub>2</sub>O and adding to the condensate (14-16 ml), the collected H<sub>2</sub>O was mixed with 5 g NaCl in a 20 ml vial. The vial was sealed and placed in a 60degC water bath. A 2 cm long, divinylbenzene/carboxen/polydimethylsiloxane SPME fibre was exposed to the headspace above the water whilst the solution was stirred. After 20 min, the fibre was withdrawn and desorbed at 250degC for 3 min in the injection port of an HP6890 GC (column details not given) equipped with a mass selective detector. The injection port was operated in splitless mode and subjected to a pressure of 25 psi He and then set at constant velocity of 40 ml/min. The quadrupole MS was operated in electron ionization mode. Levels of geosmin and 2-methylisoborneol down to 0.1 ppb could be detected.

(19) TI: Determination of isoprene in human expired breath using solid-phase microextraction with gas chromatography-mass spectrometry.

AU: Hyspler\_R, Crhova\_S, Gasparic\_J, Zadak\_Z, Cizkova\_M, Balasova\_V

JN: Journal of Chromatography, B: Biomedical Applications, 2000, Vol.739, No.1, pp.183-190 IS: 0378-4347 DT: Article NA: rhy脾er@lfhk.cuni.cz, Med. Fac., Charles Univ., Hradec Kralove 500 01, Czech Republic AB: An analytical method for determination of isoprene in expired breath as a marker of body cholesterol synthesis was developed with a special emphasis on breath sampling. Patients were breathing controlled air using respiratory masks for 2 min (washout period) and then their expired breath was collected in 8-1 Tedlar bags. The bags were heated to 40degC and the solid-phase microextraction fibre Carboxen-polydimethylsiloxane 75 µm was inserted through the septum. Extraction time was 10 min. Analytes were desorbed in the GC injector for 2 min at 270degC. Analyses were performed on a Q-PLOT column and fragment ions 68, 67 and 53 were quantified. The concentration range was 1-40 nmol/l, limit of detection was 0.25 nmol/l, the calibration curve was linear. Precision, expressed as RSD, was 5.5-12.5%. These tests are non-invasive, feasible and relatively inexpensive.

(20) TI: Determination of lidocaine in plasma by direct solid-phase microextraction combined with gas chromatography.

AU: Koster\_EHM, Wemes\_C, Morsink\_JB, deJong\_GJ

JN: Journal of Chromatography, B: Biomedical Applications, 2000, Vol.739, No.1, pp.175-182 IS: 0378-4347 DT: Article NA: Dept. Anal. Chem. and Toxicol., Centre Pharm., Univ. Groningen, 9713 AV Groningen, Netherlands AB: Direct-immersion solid-phase microextraction (SPME) has been used to extract the local anesthetic lidocaine from human plasma. A simplified model shows the relationship between the total amount of drug in plasma and the amount of drug extracted. The model takes into account that the drug participates between the fibre, sample and proteins. Therefore the model can also be used to obtain a good approximation of the drug-protein binding. Extraction yields of lidocaine in plasma are <1%, and the protein binding of lidocaine was found to be about 74% at pH 9.5. A SPME method has been developed for the determination of the total amount of lidocaine in plasma.

The protein binding was reduced by acidification and, subsequently, the sample was deproteinized with trichloroacetic acid. With a 100- $\mu\text{m}$  polydimethylsiloxane-coated fibre and addition of sodium chloride to the sample an extraction yield of about 12% at equilibrium (45 min) has been obtained. The relative standard deviation of this method is <10%. A linear range was found from 25 to 2000 ng ml<sup>-1</sup> lidocaine in plasma ( $r = 0.998$ ) with a detection limit of 5 ng ml<sup>-1</sup> in plasma. An extraction yield of about 80% could be obtained after an overnight extraction by use of a 65- $\mu\text{m}$  polydimethylsiloxane-divinylbenzene-coated fibre. If an extraction time of 10 min is used with this fibre, the same yield is obtained as with the single-phase fibre in 45 min. However, the drawback of this mixed-phase fibre is its much shorter lifetime.

(21) TI: Sorptive extraction of aqueous samples on to stirrer bars.

LA: German

AU: Hoffmann\_A, Bremer\_R, Sandra\_P, David\_F

JN: LaborPraxis, 2000, Vol.24, No.2, pp.60-62

IS: 0344-1733

DT: Article

NA: Gerstel GmbH & Co. KG, 45473 Muelheim an der Ruhr, Germany

AB: The stirrer bars (Twisters, Gerstel) are coated with polydimethylsiloxane (50-300  $\mu\text{m}$ ) and are available in two sizes - 1 cm \* 3.2 mm o.d. for 1-50 ml samples and 2 cm \* 3.2 mm o.d. for 100-250 ml samples. The samples are stirred with the bar for 30-120 min and adsorbed compounds are thermally desorbed for GC analysis. Because of the larger phase ratio of the Twisters and the more effective contacting with the solution, the sensitivity is increased by 100-1000 times compared with SPME fibres. This was demonstrated by a graph of the recoveries of a series of compounds plotted against the log of their octanol/water distribution coefficients for the two methods. The method was exemplified by two applications. An orange juice (20 ml) was stirred with a 1 cm Twister for 90 min, the analytes were desorbed at 240degC for 10 min and cryofocussed at -150degC. The compounds were analysed at a split ratio of 1:20 on a column (30 m \* 0.25 mm i.d.) coated with Satbilwax (0.25  $\mu\text{m}$ ), with He (1 ml/min) as carrier gas, temperature programming (40-230degC, details given) and scanning MS detection. Twelve constituents were identified.

Secondly, a dry white wine (25 ml) was treated with 1 ppb each of thirteen organochlorine pesticides and stirred for 40 min with a 1 cm Twister. The pesticides were analysed splitless on a column (30 m \* 0.25 mm i.d.) coated with HP-5 (0.25  $\mu\text{m}$ ), with He (170 kPa) as carrier gas, temperature programming (70-280degC, details given) and atomic emission detection, tuned for detection of Cl and Br. All were easily detected.

(22) TI: Isolation and determination of flavour fragrance components from natural products by SPME-GC and SFE-GC methods.

AU: Ligor\_M, Szumski\_M, Buszewski\_B

JN: International Laboratory, 2000, Vol.30, No.1, pp.22-25

IS: 0010-2164

DT: Article

NA: bbusz@chem.uni.torun.pl, Dept. Environ. Chem. and Ecoanal.,  
Fac. Chem., Nicholas Copernicus Univ., 87 100 Torun, Poland

AB: Samples were ground, mixed with aqueous methanol and subjected to headspace SPME with polydimethylsiloxane and polyacrylate fibres. Alternatively, samples were subjected to SFE with CO<sub>2</sub> (containing methanol). The SPME fibres were subjected to desorption in a GC injection port, whereas the supercritical extracts were collected in CH<sub>2</sub>Cl<sub>2</sub> and injected into the injection port. GC was then carried out on a Stabilwax column operated with temperature programming, He as carrier gas and FID. The flavour and fragrance compounds were identified and quantified.

(23) TI: Polypyrrole-coated capillary in-tube solid phase microextraction coupled with liquid chromatography-electrospray ionization mass spectrometry for the determination of beta-blockers in urine and serum samples.

AU: Wu\_JC, Lord\_HL, Pawliszyn\_J, Kataoka\_H

JN: Journal of Microcolumn Separations, 2000, Vol.12, No.4, pp.255-266 IS: 1040-7685

DT: Article

NA: Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

AB: Polypyrrole was coated on fused silica capillary's inner surface (GC precolumn) by chemical polymerization. At first, this coated capillary was used successfully for automated in-tube solid-phase microextraction (SPME) and beta-blockers' determination in urine and serum samples when coupled with liquid chromatography-electrospray ionization mass spectrometry (LC-ESI-MS). In-tube SPME is an extraction technique for analytes in aqueous samples, in which analytes are extracted from the sample directly to stationary phase (polymer coating) inside an open tubular capillary by repeated sample solution's draw-eject cycles. Compared with previous study using Omegawax 250 capillary for beta-blockers' extraction (Kataoka et al., Anal. Chem., 1999, 71, 4237), polypyrrole-coated capillary showed better extraction ability for most of compounds studied and therefore lower detection limits were achieved. The optimum extraction conditions are similar to those used for Omegawax 250 capillary. Other operating conditions are similar to our previous study (loc. cit.). beta-Blockers extracted by the capillary are desorbed easily by mobile phase flow without significant carryover. Using in-tube SPME-LC-ESI-MS with selected ion monitoring, beta-blockers' calibration curves are linear from 1-100 ng/ml with correlation coefficients above 0.9954 (n = 24), and detection limits (S/N = 3) less than 0.1 ng/ml for most compounds. This method was successfully applied to beta-blockers' analysis in biological samples.

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(25) TI: The use of solid-phase microextraction in conjunction with a benchtop quadrupole mass spectrometer for the analysis of volatile organic compounds in human blood at the low parts-per-trillion level.

AU: Cardinali\_FL, Ashley\_DL, Wooten\_JV, McCraw\_JM, Lemire\_SW

JN: Journal of Chromatographic Science, 2000, Vol.38, No.2, pp.49-54

IS: 0021-9665

DT: Article

NA: Div. Environ. Health. Lab. Sci., Natl. Center Environ. Health,

Centers Disease Control and Prevention, Public Health Service ,U.S. Dept. Health and Human Services, Atlanta, GA 30341, USA AB: The analysis of volatile organic compounds (VOC) in whole human blood at the low parts-per-trillion level has until

recently required the use of a high-resolution mass spectrometer to obtain the specificity and detection limits required for epidemiological studies of VOC exposure in the general public. Because of the expense and expertise required to operate and maintain a high-resolution instrument, the applicability of this method has been limited. These limitations are overcome in a new method using automated headspace solid-phase microextraction (SPME) in conjunction with a gas chromatograph and a benchtop quadrupole mass spectrometer. A combination of SPME and multiple single-ion monitoring minimizes the interferences and chemical noise associated with whole blood samples. This method permits the analysis of 10 VOC in human blood while simplifying the sample preparation and reducing the possible exposure of the analyst to blood aerosols. Twelve samples can be run successively in a fully automated mode, thus eliminating the need for operator attention. Detection limits are below 50 ppt (pg/ml) for a majority of the VOC tested with a 5 ml sample.

(26) TI: Determination of trichloromethane, tetrachloromethane and trichloroethane by using microextraction with GC-ECD detection.

AU: LuksBetlej\_K, Bodzek\_D

JN: *Chemia Analytyczna* (Warsaw), 2000, Vol.45, No.1, pp.45-51

IS: 0009-2223

DT: Article

NA: Dept. Chem., Fac. Med., Silesian Med. Acad., 41-808 Zabrze, Poland

AB: Microextraction technique in liquid phase with the use of the fibres coated with polydimethylsiloxane phase for separation trichloromethane, tetrachloromethane and 1,1,1-trichloroethane from drinking water samples coming from the Upper Silesia was applied. Quantitative analysis was performed by using gas chromatography with electron-capture detection (ECD) detector.

The contents of investigated compounds did not exceed the maximum permissible concentration recommended by WHO.

(27) TI: Determination of polycyclic aromatic hydrocarbons in sediment using solid-phase microextraction with gas chromatography-mass spectrometry.

AU: Cam\_D, Gagni\_S, Meldolesi\_L, Galletti\_G

JN: *Journal of Chromatographic Science*, 2000, Vol.38, No.2, pp.55-60

IS: 0021-9665

DT: Article

NA: Environ. Res. Center Montecatini, Univ. Bologna, Bologna, Italy

AB: Manual SPME coupled with GC-MS is applied for the determination of polycyclic aromatic hydrocarbons (PAH) from natural matrix through a distilled water medium. Seven of the 16 PAH standards (naphthalene, acenaphthene, fluorene, anthracene, fluoranthene, pyrene, benzo[a]anthracene) are spiked on a marine muddy sediment. The samples, containing PAH in the range of 10-20 ppm, are then aged at room temperature more than 10 days before analysis. The influence of the matrix, SPME adsorption time, pH, salt content, and SPME adsorption temperature are investigated. The reproducibility of the technique is less than 13% (RSD) for the first 6 considered PAHs and 28% (RSD) for benzo(a)anthracene with a fibre containing a 100 µm polydimethylsiloxane coating. Linearity extended in the range of 5-50 picograms for PAH direct injection, 5-70 picograms for PAH in water, and 1-170 picograms for PAH in sediment. The detection limit is

estimated less than 1 mug/kg of dry sample for the first 6 considered PAH in sediment and 1.5 mug/kg of dry sample for benzo(a)anthracene using the selected ion monitoring mode in GC-MS. The recoveries of the considered PAH are evaluated.

(28) TI: Analysis of biogenic amines by solid-phase microextraction and high-performance liquid chromatography with electrochemical detection.

AU: Auger\_J, Boulay\_R, Jaillais\_B, DelionVancassel\_S

JN: Journal of Chromatography, A, 2000, Vol.870, No.1-2, pp.395-403

IS: 0021-9673

DT: Article

NA: auger@univ-tours.fr, Inst. Recherche Biol. Insecte, Univ.

Francois Rabelais, 37200 Tours, France

CO: Presented at the 23rd International Symposium on High-Performance Liquid Phase Separations and Related Techniques, held in Granada, Spain, 30 May-4 Jun 1999

AB: SPME was investigated by HPLC with electrochemical detection for the analysis of biogenic amines. The Carbowax-Templated Resin 50 mum (purple) fibre coating offers good performances for dopamine and serotonin separation, i.e., good selectivity and high sensibility (0.1 mug/l). This fibre was also tested for biogenic amine quantification of rat striatum. The coating seems to be selective towards the amines and has low affinity for the metabolites, allowing a good separation and preventing drawbacks from the biological matrix. Preliminary results indicate the method may have a large applicability to many biological samples.

(29) TI: Solid-phase microextraction of inorganic anions based on polypyrrole film.

AU: Wu\_JC, Yu\_XM, Lord\_H, Pawliszyn\_J

JN: Analyst (Cambridge, U. K.), 2000, Vol.125, No.3, pp.391-394

IS: 0003-2654

DT: Article

NA: <mailto:janusz@uwaterloo.ca>, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

CO: Presented at SAC 99, held in Dublin, Ireland, 25-30 Jul 1999

AB: The natural anion exchange property of conducting polymer polypyrrole (PPY) was examined using solid-phase microextraction (SPME) methods. Our preliminary results demonstrated that the anion exchange property of PPY could be utilized for direct SPME of anionic species from aqueous solutions without derivatization. This paper presented the first example of coupling SPME to ion chromatography (IC).

(30) TI: In-tube solid-phase microextraction coupled with HPLC and its automated system.

LA: Japanese

AU: Saido\_Y

JN: Bunseki, 2000, No.3, p.169

IS: 0386-2178

DT: Article

NA: Fifth Dept. Eng., Toyohashi Univ. Technol., Toyohashi, Japan

AB: The automation of a system for in-tube SPME coupled with HPLC (schematic given) is described and exemplified by the determination of agrochemicals in water. (4 references).

(31).

(32)

(33) TI: Online coupling of in-tube solid phase microextraction (SPME) to HPLC for analysis of carbamates in water samples: comparison of two commercially available autosamplers.

AU: Gou\_Y, Tragas\_C, Lord\_H, Pawliszyn\_J

JN: Journal of Microcolumn Separations, 2000, Vol.12, No.3, pp.125-134

IS: 1040-7685

DT: Article

NA: janusz@sciborg.uwaterloo.ca, Guelph-Waterloo Centre Graduate Work Chem., Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

CO: Presented at the Twenty-First International Symposium on Capillary Chromatography and Electrophoresis, held in Park City, UT, USA, 20-24 Jun 1999

AB: In-tube solid phase microextraction (SPME) in a new on-line sample preparation technique that is easily installed on a conventional high-performance liquid chromatography (HPLC) autosampler, and has previously been used with the LC Packings

FAMOS autosampler without modification of the autosampler itself. This paper describes the on-line coupling of in-tube SPME to a Hewlett Packard (HP) model 1100 HPLC autosampler, and reports on several fundamental aspects of the technology. The extraction and desorption processes with this system were thoroughly studied for a group of carbamate pesticides in water samples. The similarities and differences of the in-tube SPME technique with the two different autosamplers, FAMOS and Hewlett Packard, were compared during method development. The optimized conditions were successfully applied to extract the carbamates of interest from spiked surface, well, and tap water sample, exhibiting excellent method precision. The % relative standard deviation (RSD) for all the carbamates studied in the different environmental water samples was between 0.8 and 6.0%.

There was no noticeable extraction efficiency decrease relative to pure laboratory samples when the method was applied to spiked environmental water samples. The method was found to be linear between 2 and 20,000 mug/l with correlation coefficients in the range of 0.9791 to 0.9991. With respect to the different water samples, the limits of detection (LODs) for all the carbamates studied were in the range of 0.44-17 mug/l. For carbaryl, the LODs were between 0.44 and 0.67 mug/l, which were lower than those obtained using the in-tube SPME method with the FAMOS autosampler.

(36) TI: Preliminary study on determination of prohibited azo dyes by solid-phase microextraction.

LA: Chinese

AU: Xu\_H, Tong\_H

JN: Fenxi Ceshi Xuebao, 2000, Vol.19, No.1, pp.76-78

IS: 1004-4957

DT: Article

NA: Tianjin Import and Export Commodity Inspection Bureau, Tianjin 30020, China

AB: Defatted leather (1 g) was agitated with 12 ml 60mM-citrate buffer of pH 6 at 70degC for 30 min in a sealed phial. The digest was treated with 3 ml aqueous 200 g/l Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> at 70degC for 30 min, cooled to room temperature and filtered. The filtrate was stirred with a 85 µm PA SPME extraction fibre at 50degC for 30 min. The fibre was retrieved and inserted into the injector port of a gas chromatograph and the adsorbed azo dyes (as aromatic amines) were desorbed directly onto a fused-silica column (30 m \* 0.2 mm i.d.) coated with PE-35ms (0.25 µm), operated with temperature programming from 30-130degC (held for 5 min) at 15degC/min, and to 250degC (for 1 min) at 5degC/min, (no carrier gas/flow rate details given) and 70 eV EIMS detection operated in full-scan mode from 30 and 300 amu. Eighteen amines were extracted by SPME (all listed). The detection limits were 30 µg. Recoveries were 50-94.6%.

(37) TI: Toxicology and evaluation of microcystins.

AU: Lam\_PKS, Yang\_MS, Lam\_MHW

JN: Therapeutic Drug Monitoring, 2000, Vol.22, No.1, pp.69-72

IS: 0163-4356

DT: Article

NA: Dept. Biol. and Chem., City Univ. Hong Kong, Kowloon, Hong Kong SAR, China

AB: A review is presented on microcystins, which exhibit acute toxicity and tumour-promoting characteristics in humans. Recent developments indicate the potential application of biosensor technology to studying the effects of these toxins. In particular, biosensors based on surface plasmon resonance offer a quantitative approach to studying biochemical effects, and the structure of toxin-bound protein complexes. Methods for the screening, identification and determination of microcystins are compared and discussed (table and figure); these include bioassay, HPLC, ELISA, NMR and MS. The article focuses on relatively new extraction and detection techniques that are simple and allow rapid analysis, e.g. SPME coupled to HPLC, and CZE with laser-induced fluorescence detection. Of the treatments applied to remove cyanobacterial toxins, only granular activated carbon and ionization are effective in treating contaminated environmental waters. (24 references).

(38) TI: A comparison of solid-phase microextraction (SPME) fibres for measurement of hexanal and pentanal in cooked turkey.

AU: Brunton\_NP, Cronin\_DA, Monahan\_FJ, Durcan\_R

JN: Food Chemistry, 2000, Vol.68, No.3, pp.339-345

IS: 0308-8146

DT: Article

NA: Dept. Food Sci., Univ. College Dublin, Dublin 4, Ireland

AB: Turkey breasts (500 g) were cooked in a domestic oven set at 190degC, until the internal temperature of the samples reached 85degC. Samples were cooled to 4degC and analysed immediately, or after storage for 1, 2, 4 and 6 days at 4degC. Portions (5g) were homogenized with H<sub>2</sub>O (25 ml) and 2-methyl pentanal was added as internal standard. Portions (3 ml) of each homogenate was transferred to 5-ml phials, fitted with PTFE septa and 10 µl 0.01% hexanal and 10mM-butylated hydroxyanisole were added to each phial. The phials were mixed and heated to 40degC on a water-bath for 5 min. Fibres coated with polydimethylsiloxane (I), or divinylbenzene (II) or a mixture of both (III), were introduced

into the phial headspace for different periods of time up to 20 min. The fibres were placed in the injection port of a Pye Unicam series 204 gas chromatograph and the adsorbed VOC were desorbed directly onto a fused-silica column (15m\*0.53mm i.d.) coated with Carbowax 20M (1 µm), operated with temperature-programming from 60degC to 160degC at 3degC/min, with H<sub>2</sub> as carrier gas (2 ml/min) and FID. Detection limits for the fibres coated with I, II or III were 2, 12 and 7 ng/g, respectively. The mixture-coated fibre exhibited best performance as regards linearity and reproducibility. Results showed that the average hexanal level increased from 0.8 µg/g to 4.01 µg/g after storage for 6 days.

(39)

(40)

(41) TI: Solid phase microextraction (SPME): a new procedure for the control of butyl- and phenyltin pollution in the environment by GC-flame photometric detection (FPD).

AU: Aguerre\_S, BanconMontigny\_C, Lespes\_G, PotinGautier\_M

JN: Analyst (Cambridge, U. K.), 2000, Vol.125, No.2, pp.263-268

IS: 0003-2654

DT: Article

NA: Lab. Chim. Anal. Bio-Inorg. et Environ. (LCABIE), EP CNRS 132, Univ. Pau et des Pays de l'Adour, Pau 64000, France

AB: A new alternative to liquid-liquid extraction for the speciation of organotin compounds is proposed using sodium tetraethylboride (BEt<sub>4</sub>) ethylation-simultaneous solid phase microextraction (SPME)-GC-FPD. SPME is carried out using a polydimethylsiloxane (PDMS) fibre immersed in 100 ml of solution for an adsorption time of 60 min. The optimisation step confirms the importance of stirring the solution. The higher efficiency of mechanical stirring is also clearly demonstrated. Under these conditions, SPME was applied for the first time to the simultaneous extraction of butyl- and phenyltin compounds and the analytical performances were evaluated. Very low detection limits were reached, in the range 0.006-0.031 ng Sn I-1 for butyltins and 0.2-0.6 ng Sn II for phenyltins. The repeatability is also improved compared with classical liquid-liquid extraction thanks to the small volume of the fibre and the on-line procedure (between 3 and 9% except for triphenyltin). The new method was applied to various environmental samples such as natural aqueous samples, sediment and sewage sludge. The competitive extractions between some organotins and organic matter present in complex matrices are discussed.

(42) TI: Polyacrylate-coated SPME fibres as a tool to simulate body residues and target concentrations of complex organic mixtures for estimation of baseline toxicity.

AU: Verbruggen\_EMJ, Vaes\_WHJ, Parkerton\_TF, Hermens\_JLM

JN: Environmental Science and Technology, 2000, Vol.34, No.2, pp.324-331

IS: 0013-936X

DT: Article

NA: j.hermens@ritox.vet.uu.nl, Res. Inst. Toxicol., Utrecht Univ., 3508 TD Utrecht, Netherlands

AB: Biomimetic extractions, used to estimate total body residues, act to mimic the bioconcentration process occurring by simple physical partitioning (passive diffusion) thereby mimicking the bioconcentration process in organisms that do not metabolize.

The study typically involves two stages: the bioconcentration process stimulated by a hydrophobicity-dependent extraction and the determination of the total molar concentration of the analyte in the hydrophobic phase. In this study SPME extraction of aqueous methanolic solutions of various aromatic and other compounds were performed in 250 ml flat-bottomed screw cap flasks (details given) with use of polyacrylate SPME fibres and determination of compounds by GC-MS (details given). Results are presented and discussed. The experimental SPME-H<sub>2</sub>O partition coefficients correlated well with the octanol-H<sub>2</sub>O partition coefficients indicating that these passive sampling devices provide a good surrogate for lipid partitioning.

(43) TI: Rapid detection of tetramethylenedisulfotetramine in human blood by solid-phase microextraction-gas chromatography.

AU: Luan\_TG, Li\_GK, Zhao\_MQ, Zhang\_ZX

JN: Analytica Chimica Acta, 2000, Vol.404, No.2, pp.329-334

IS: 0003-2670

DT: Article

NA: cep971tg@nct.zsu.edu.cn, Dept. Chem., Zhongshan Univ., Guangzhou 510275, China

AB: SPME coupled with GC was used to develop a simple, rapid and sensitive method for the determination of tetramethylenedisulfotetramine (tetramine) in human blood. The tetramine was extracted with a fused silica fibre coated with a 100 µm polydimethylsiloxane and detected by GC with a N - P detection. The effects of extraction and desorption times as well as the extraction temperature were studied. A linear response of tetramine over a concentration range of 0.008-0.5 µg/ml with a correlation coefficient of 0.998 and detection limit of 0.001 µg/ml (3s criteria) were obtained. Spiked human blood samples were analysed. The analytical recoveries ranged from 92-114% and the mean RSD was 10.5%.

(44) TI: Tetrachloroethylene and trichloroethylene fatality: case report and simple headspace SPME-capillary gas chromatographic determination in tissues.

AU: Dehon\_B, Humbert\_L, Devisme\_L, Stievenart\_M, Mathieu\_D, Houdret\_N, Lhermitte\_M

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.1, pp.22-26 IS: 0146-4760

DT: Article

NA: mlhermitte@chrul-lille.fr, Lab. Biochim. et Biol. Mol., CHR Univ. Lille, 59037 Lille Cedex, France

AB: We describe a simple, precise, and sensitive assay of tetrachloroethylene and trichloroethylene in tissues, suitable both for emergency cases and forensic medicine. The method employs headspace solid phase microextraction-capillary gas chromatography and electron capture detection. The case is relative to a 45-year-old woman discovered unconscious in a laundry area. The concentrations of the solvents in tissues were determined and compared to other previously published fatalities.

(45) TI: Rapid analysis of amphetamine, methamphetamine, MDA, and MDMA in urine using solid-phase microextraction, direct on-fiber derivatization, and analysis by GC-MS.

AU: Jurado\_C, Gimenez\_MP, Soriano\_T, Menendez\_M, Repetto\_M

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.1, pp.11-16 IS: 0146-4760

DT: Article

NA: Inst. Nacional Toxicologia, 41080 Sevilla, Spain

AB: A rapid, sensitive, and solvent-free procedure for the simultaneous determination of amphetamine, methamphetamine, 3,4-methylenedioxyamphetamine (MDA), and 3,4-methylenedioxymethamphetamine (MDMA) in urine was developed using solid-phase microextraction (SPME) and gas chromatography-mass spectrometry (GC-MS) operated in the selected-ion monitoring mode. A headspace phial containing the urine sample, NaOH, NaCl, and amphetamine-d<sub>3</sub> as the internal standard was heated at 100degC for 20 min. A polydimethylsiloxane fiber was maintained in the phial headspace for 10 min in order to adsorb the amphetamine derivatives, which were subsequently derivatized by exposing the fiber to trifluoroacetic anhydride for 20 min in the headspace of another phial maintained at 60degC for 20 min. The trifluoroacetyl derivatives were desorbed in the GC injection port for 5 min. Several parameters were considered during the method optimization process. These included a comparison of SPME with or without headspace, the required derivatization procedure, and the influence of temperature on the headspace extraction and derivatization methods. The optimized method was validated for the four compounds tested. Calibration curves showed linearity in the range 50-1000 ng/ml ( $r = 0.9946-0.9999$ ). Recovery data were 71.89-103.24%. The quantitation limits were 10 ng/ml for amphetamine and methamphetamine and 20 ng/ml for MDA and MDMA. All of these data recommend the applicability of the method for use in the analytical routine for a forensic laboratory.

(46) TI: Use of solid-phase microextraction (SPME) for the determination of methadone and its main metabolite, EDDP, in plasma by gas chromatography-mass spectrometry.

AU: Bermejo\_AM, Seara\_R, dosSantosLucas\_AC, Tabernero\_MJ, Fernandez\_P, Marsili\_R

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.1, pp.66-69

IS: 0146-4760

DT: Article

NA: Dept. Legal Med., Forensic Toxicol. Service, Fac. Med., Univ. Santiago de Compostela, Santiago de Compostela, Spain

AB: A simple, rapid method for the determination of methadone and its metabolite 2-ethylene-1,5-dimethyl-3,3-diphenylpyrrolidine (EDDP) in plasma using solid-phase microextraction (SPME) and gas chromatography-mass spectrometry is proposed. A 100- $\mu$ m polydimethylsiloxane film fibre was exposed by immersion for 30 min in a diluted plasma solution (1:4 with buffer pH 9) containing both compounds and an internal standard (proadifen).

Calibration curves were linear over the concentration range 50-2000 ng/ml. The analysis time was 45 min per sample. The determination of methadone and EDDP was subject to no interference. The performance of SPME was compared with that of liquid-liquid extraction, obtaining lower limits of detection for EDDP. The method using the two extraction procedures was applied to 10 plasma samples from methadone-treated patients.

(47) TI: Saliva/plasma ratio of methadone and EDDP.

AU: Bermejo\_AM, Lucas\_ACS, Tabernero\_MJ

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.1, pp.70-72

IS: 0146-4760

DT: Article

NA: Inst. Legal Med., Univ. Santiago de Compostela, Santiago de Compostela, Spain

AB: The concentrations of methadone (I) and its main metabolite

EDDP (2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine; II) in saliva and plasma from 10 patients in a I-maintenance program at the Galicia Autonomic Community (Spain) was investigated.

Samples were taken just before the daily dose of I and at least 30 min after consumption of food. Saliva was collected by the spitting method, in which patients were asked to close their lips and collect the saliva over 5 min, then spit it into a universal plastic container (Salivette, Sarstedt, Numbrecht, Germany). Blood obtained by venipuncture was separated by centrifugation at 5000 rpm for 5 min. Plasma and saliva samples spiked with I-d3 and II-d3 (internal standards) were subjected to SPME by the method of Bermejo et al. (Ibid., 2000, 24, 66). Portions of extracts were analysed by GC on a fused-silica column (12 m \* 0.22 mm i.d.), coated with HP-5 (0.33 µm), and operated with temperature programming from 90degC (held for 2 min) to 200degC (held for 5 min) at 30degC/min, and 70 eV EIMS detection operated in selected-ion monitoring mode at m/z 294, 295 and 223 and 277, 276 and 262, respectively, for I and II. Results (tabulated) showed that the concentrations of I and II in saliva and plasma were in the range 0.12-3.46 and 0.04-0.1 and 0.21-0.64 and 0.03-0.42 mg/l, respectively. The saliva/plasma ratios of I were in the range 0.6-7.2. Correlation analysis showed a significant inverse correlation between salivary pH and the concentration of I; there was no significant correlation for its metabolite II.

(48) TI: Use of headspace solid-phase microextraction (HS-SPME) in hair analysis for organic compounds.

AU: Sporkert\_F, Pragst\_F

JN: Forensic Science International, 2000, Vol.107, No.1-3, pp.129-148

IS: 0379-0738

DT: Article

NA: Inst. Legal Med., Humboldt Univ., 10115 Berlin, Germany

CO: Presented at the Second International Meeting of the Society for Hair Testing, held in Martigny, Switzerland, 14-16 Jun 1999

AB: The use of headspace (HS) SPME for the determination of drugs in hair by GC-MS is described. For lipophilic basic drugs, hair (10 mg) was digested with 1 ml 1M-NaOH and 0.5 g Na<sub>2</sub>SO<sub>4</sub> in the presence of a suitable internal standard (100 ng) for 30 min at 70-90degC. SPME was performed at 60-90degC by exposing a SPME fibre to the headspace above the digest for 15-20 min. The drugs were desorbed from the fibre at 250-290degC for 5 min and quantified by GC on a 0.25 µm HP column (30 m \* 0.25 mm i.d.), with He as carrier gas (1 ml/min) and MS detection in the selected-ion monitoring mode. Several different GC temperature programmes were used depending on the drug(s) to be determined (details given). The modification of the method to enable other drugs to be determined was also investigated. For the drugs studied, the detection limits were 0.05-1 ng/mg, the RSD (n =

5) was typically 8.3% and the absolute recoveries were 0.04-

5.7%. The method was applied to hair from several forensic and clinical cases.

(49) TI: Use of solid-phase microextraction (SPME) for the determination of methadone and EDDP in human hair by GC-MS.

AU: Lucas\_ACS, Bermejo\_AM, Tabernero\_MJ, Fernandez\_P, StranoRossi\_S

JN: Forensic Science International, 2000, Vol.107, No.1-3, pp.225-232

IS: 0379-0738

DT: Article

NA: Dept. Clinical Anal. and Toxicol., Univ. Amazonas, Manaus, Brazil

CO: Presented at the Second International Meeting of the Society for Hair Testing, held in Martigny, Switzerland, 14-16 Jun 1999

AB: Hair was washed with H<sub>2</sub>O and acetone and then incubated with Pronase E/DTT at 37degC for 12 h. Following a fourfold dilution with borax buffer of pH 9.2 containing methadone-2H<sub>3</sub> and 2-ethylene-1,5-dimethyl-3,3-diphenylpyrrolidine (EDDP)-2H<sub>3</sub> as internal standards, SPME was performed by immersing a 100 µm polydimethylsiloxane-coated fibre in the resulting solution for 30 min. The analytes were desorbed from the fibre at 250degC for 5 min and quantified by GC on a 0.33 µm HP-5 column (12 m \* 0.22mm i.d.) operated with temperature programming from 90degC (held for 1 min) to 200degC (held for 5 min) at 30degC/min, then to 290degC at 30degC/min, with He as carrier gas (1 ml/min) and 70 eV EIMS detection in the selected-ion monitoring mode (details given). The calibration graphs were linear from 1-50 ng/mg for both methadone (I) and its metabolite EDDP (II), the detection limits were 2.48 ng/mg I and 0.15 ng/mg II, the inter-assay RSD (n = 8) were 7.3-13.3% and the recoveries were 102.5-107.2%. The method was applied to hair from patients on a I maintenance programme (results tabulated).

(50) TI: Contribution of volatiles to the flavour of oatmeal.

AU: Zhou\_MX, Robards\_K, GlennieHolmes\_M, Helliwell\_S

JN: Journal of the Science of Food and Agriculture, 2000, Vol.80, No.2, pp.247-254

IS: 0022-5142

DT: Article

NA: School Sci. Technol., Charles Sturt Univ., Wagga Wagga, NSW 2678, Australia

AB: Oatmeal (20 g) was mixed with 100 ml H<sub>2</sub>O and the mixture was heated until boiling. The volatile flavour compounds (VFC) in the headspace above the mixture were trapped by SPME using a polydimethylsiloxane non-bonded fibre (100 µm). The fibre was transferred to the inlet chamber of a GC at 220degC for 1.5 min. The VFC were desorbed directly onto a fused-silica GC column (30 m \* 0.2 mm i.d.) coated with SE30 (0.25 µm). The column was operated with temperature programming from 50degC (held for 1 min) to 300degC (held for 4 min) at 10degC/min (no carrier gas details given) and FID. The VFC were identified by desorption in GC/MS and injected onto a fused-silica column (60m \* 0.32 mm i.d.) coated with DBWax, and operated with temperature programming from 35degC (held for 5 min) to 220degC (held for 15 min) at 3degC/min and 70 eV EIMS detection.

Results (tabulated) were compared with results obtained by sensory testing.

(51) TI: Absorption of hydrophobic compounds into the poly(dimethylsiloxane) coating of solid-phase microextraction fibers: high partition coefficients and fluorescence microscopy images.

AU: Mayer\_P, Vaes\_WHJ, Hermens\_JLM

JN: Analytical Chemistry, 2000, Vol.72, No.3, pp.459-464

IS: 0003-2700

DT: Article

NA: p.mayer@ritox.vet.uu.nl, Environ. Toxicol. and Chem., RITOX,

Utrecht Univ., 3508 TB Utrecht, Netherlands

AB: The use of solid-phase microextraction with poly(dimethylsiloxane) (PDMS)-coated glass fibers for the extraction and analysis of hydrophobic organic analytes is increasing. The literature on this topic is characterized by large discrepancies in partition coefficients and an uncertainty of whether highly hydrophobic analytes are retained by absorption into the fibre coating or by adsorption to the fibre surface. We applied a new method, which minimizes the impact of experimental artifacts, to determine PDMS including chlorinated benzenes, PCB, PAH, and p,p'-DDE. These partition coefficients are several orders of magnitude higher than some reported values. Two observations strongly suggest that the retention of hydrophobic organic substances is governed by partitioning into the PDMS coating. (1) The partition coefficients are proportional with octanol/H<sub>2</sub>O partition coefficients. (2) The fluorescence of fluoranthene was observed to be homogeneously distributed within the polymer coating when studied by means of fluorescence microscopy. Implications of these findings for the application of solid-phase microextraction with respect to potential detection limits, with respect to biomimetic extraction, and with respect to measurements in multicompartment systems are discussed.

(52) TI: Comparative study and partial characterization of Azorean green tea polyphenols.

AU: Baptista\_JAB, Tavares\_JFdP, Carvalho\_RCB

JN: Journal of Food Composition and Analysis, 1999, Vol.12, No.4, pp.273-287

IS: 0889-1575

DT: Article

NA: Dept. Ciencias Technol. e Desenvolvimento, Univ. Azores, 9502 Ponta Delgada, Sao Miguel, Azores, Portugal AB: Polyphenols in green tea were extracted with H<sub>2</sub>O (20 ml) for 15 min at 70degC four times and the combined extracts filtered and freeze dried. A 100 mg portion was dissolved in H<sub>2</sub>O (10 ml) and extracted with chloroform (10 ml), to remove methylxanthines and pigments, followed by ethyl acetate extraction (repeated three more times). The combined ethyl acetate extracts were dried and redissolved in 500 ml H<sub>2</sub>O then analysed on a 4 µm C18 column (15 cm \* 3.9 mm i.d.) at 35degC, with gradient elution (details given) with acetonitrile/ethyl acetate/0.1% phosphoric acid (8.5:2:89.5) and acetonitrile/H<sub>2</sub>O (1:1) and detection at 280 nm. The green tea polyphenols extraction yield was determined and the contents of different green teas compared (results tabulated and presented graphically). The aroma of different green tea samples were compared using SPME-GC-headspace methods (details given).

(53) TI: Determination of chlorophenols in soils using accelerated solvent extraction combined with solid-phase microextraction.

AU: Wennrich\_L, Popp\_P, Moeder\_M

JN: Analytical Chemistry, 2000, Vol.72, No.3, pp.546-551

IS: 0003-2700

DT: Article

NA: lwenn@ana.ufz.de, Interdisciplinary Dept. Urban Landscapes, UFZ Centre for Environ. Res., Leipzig/Halle Ltd, 04318 Leipzig, Germany

AB: A method for the determination of chlorophenols in soil samples using accelerated solvent extraction (ASE) with H<sub>2</sub>O as the solvent combined with SPME and GC-MS has been developed. Important ASE parameters, such as extraction temperature and time, were optimized using a spiked wetland soil. The effect of small amounts of organic modifiers on

the extraction yields was studied. An extraction temperature of 125degC and 10 min extractions performed three times proved optimal. Two ASE-SPME procedures with and without an organic modifier (5% acetonitrile) were evaluated with respect to precision and detection limits (LOD). The reproducibility of replicate H2O extractions/SPME determinations (n = 6) was in the range 7-20% RSD for the nine chlorophenols investigated. LOD values in the low-ppb range were achieved for all chlorophenols. The ASE-SPME procedure presented here was applied to the determination of chlorophenols in soil samples taken from contaminated areas near Bitterfeld, Germany.

(54) TI: Comments on "adsorption versus absorption of polychlorinated biphenyls onto solid-phase microextraction coatings". Letter.

AU: Vaes\_WHJ, Mayer\_P, Oomen\_AG, Hermens\_JLM, Tolls\_J

JN: Analytical Chemistry, 2000, Vol.72, No.3, pp.639-641

IS: 0003-2700

DT: Article

NA: w.vaes@voeding.tno.nl, Dept. Food and Non-Food Anal., TNO, 3700

AJ Zeist, Netherlands

AB: The authors argue that the method used by Yang et al. (Anal. Chem., 1998, 70, 1866; J. Chromatogr., A, 1998, 800, 257) was inadequate to determine poly(dimethylsiloxane)/H2O partition coefficients of highly hydrophobic substances for the following two reasons. Firstly the phase ratio (Vwater/VPDMS) was too low for the determination of high partition coefficients (e.g. 1 000 000) that can be expected in case of an absorption process. Secondly the measurement of only the SPME-bound fraction, while assuming the remaining part to be completely dissolved, makes the approach highly susceptible to experimental artifacts. Consequently, it is concluded that the partition coefficients of Yang et al. should not be regarded as evidence for the adsorption theory.

(55) TI: Response to comments on adsorption versus absorption of polychlorinated biphenyls onto solid-phase microextraction coatings. Reply.

AU: Hawthorne\_SB, Yang\_Y, Grabanski\_CB, Miller\_DJ, Lee\_ML

JN: Analytical Chemistry, 2000, Vol.72, No.3, pp.642-643

IS: 0003-2700

DT: Article

NA: Energy and Environ. Res. Center, Univ. North Dakota, Grand Forks, ND 58202, USA

AB: The authors reply to Vaes et al. (Anal. Chem., 2000, 72, 639) who challenged the conclusion that adsorption rather than absorption is taking place when PCB are extracted by SPME using poly(dimethylsiloxane) coated fibres (Yang et al., Anal. Chem., 1998, 70, 1866; and J. Chromatogr., A., 1998, 800, 257). The authors argue that adsorption rather than absorption mechanisms remain the best interpretation of their results.

(57) TI: Application note - Analysis of industrial waste water.

BN: Chrompack Application Note 1451-GC

DT: Technical Report

AB: According to a Chrompack application note, waste water (2.5 g) was mixed with 2.5 g potassium carbonate and subjected to SPME in the headspace mode for 30 min at 50degC. Splitless desorption was applied with pressure programming from 50-300 kPa in a large

bore liner (0.75 mm i.d.) to give an optimal peak shape for low-boiling components. The desorbed analytes were separated by GC on a fused-silica column (25 m \* 0.15 mm i.d.) coated with CP-Sil 5 CB (2 µm), operated with temperature programming from 40degC (held for 2 min) to 250degC at 10degC/min, H2 carrier gas (50-300 kPa) and FID (300degC). A fast separation of 35 components, including methanol, acetone, benzene, styrene and toluene, was achieved within 18 min. A chromatogram is shown.

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(59) TI: Source identification of underground fuel spills by solid-phase microextraction/high-resolution gas chromatography/genetic algorithms.

AU: Lavine\_BK, Ritter\_J, Moores\_AJ, Wilson\_M, Faruque\_A, Mayfield\_HT

JN: Analytical Chemistry, 2000, Vol.72, No.2, pp.423-431

IS: 0003-2700

DT: Article NA: Dept. Chem., Clarkson Univ., Potsdam, NY 13699-5810, USA

AB: Solid-phase microextraction (SPME), capillary column gas chromatography, and pattern recognition methods were used to develop a potential method for typing jet fuels so a spill sample in the environment can be traced to its source. The test data consisted of gas chromatograms of 180 neat jet fuel samples representing common aviation turbine fuels found in the United States (JP-4, Jet-A, JP-7, JPTS, JP-5, JP-8). SPME sampling of the fuel's headspace afforded well-resolved reproducible profiles, which were standardized using special peak-matching software. The peak-matching procedure yielded 84 standardized retention time windows, though not all peaks were present in all gas chromatograms. A genetic algorithm (GA) was employed to identify features (in the standardized chromatograms of the neat jet fuels) suitable for pattern recognition analysis. The GA selected peaks, whose two largest principal components showed clustering of the chromatograms on the basis of fuel type. The principal component analysis routine in the fitness function of the GA acted as an information filter, significantly reducing the size of the search space, since it restricted the search to feature subsets whose variance is primarily without differences between the various fuel types in the training set. In addition, the GA focused on those classes and/or samples that were difficult to classify as it trained using a form of boosting. Samples that consistently classify correctly were not as heavily weighted as samples that were difficult to classify. Over time, the GA learned its optimal parameters in a manner similar to a perceptron. The pattern recognition GA integrated aspects of strong and weak learning to yield a "smart" one-pass procedure for feature selection.

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(62) TI: Application of solid-phase micro-extraction for the rapid analysis of chlorinated organics in breast milk. AU: Rohrig\_L, Meisch\_HU

JN: Fresenius' Journal of Analytical Chemistry, 2000, Vol.366,

No.1, pp.106-111

IS: 0937-0633

DT: Article

NA: Zentrum Umweltforschung, Univ. Saarlandes, 66125 Saarbrücken, Germany

AB: The method presented here allows the monitoring of persistent organochlorine compounds in breast milk using the solid phase microextraction technique (SPME) and gas chromatography with electron capture detection (GC-ECD). It describes the determination of hexachlorobenzene (HCB), alpha-, beta- and gamma-hexachlorocyclohexane (HCH), dichlorodiphenyltrichloroethane (DDT) and its derivatives, and some important congeners of the polychlorinated biphenyls (PCB). Also included are more polar substances such as tri-, tetra- and pentachlorophenols, which can be analyzed simultaneously with the aforementioned less polar compounds without the need of a derivatization for the determination of the phenolic compounds. The reproducibility of the results is very good down to the lower mug/l-region. The method is very fast and of low cost compared to the classic extraction and determination procedures. We had already developed a method for the determination of chlorinated organics in whole blood using the new solid-phase micro extraction technique (cf., Belardi and Pawliszyn, Water Pollut. Res. J. Can., 1989, 24, 179), combined with GC/ECD determination. This proved to be a fast and low-cost alternative to the established sample pretreatment techniques to the established sample pretreatment techniques that are commonly used. These include solvent extraction (liquid-liquid extraction, LLE) or separation on solid materials, such as silica gel (solid phase extraction, SPE). So the idea of using SPME sampling for the determination of pesticides and related compounds in breast milk seemed attractive. We succeeded in developing an SPME method for an easy separation of the same spectrum of chlorinated organics as described by Rohrig et al., (Fresenius' J. Anal. Chem., 1998, 361, 192) from breast milk samples, followed by GC with ECD detection.

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(64) TI: Determination by solid-phase microextraction-gas chromatography-mass spectrometry of polycyclic aromatic hydrocarbons in bitumen fumes during road laying.

AU: Agozzini\_P, Avellone\_G, Boscaino\_G, Miceli\_S

JN: Journal of Mass Spectrometry, 1999, Vol.34, No.12, pp.1383-1384

IS: 1076-5174

DT: Article

NA: agozzini@unipa.it, Dipt. Chim. e Technol. Farm., Univ. Palermo, 90123 Palermo, Italy

CO: Presented at 17th Informal Meeting on Mass Spectrometry, held in Fiera di Primiero, Italy, 23-27 May 1999

AB: Bitumen (5 g) was placed in a vial sealed with a silicone-rubber septum with internal PTFE seal and heated for 30 min at 120 or 160degC in a constant-temperature oil bath. Then the headspace gases were sampled for 10 min with use of an SPME fibre coated with a 100-mum thick film of polydimethylsiloxane and the exposed fibre was subsequently analysed on a Varian SATURN 3 GC-MS system equipped with a Restek RTX-5MS column (30m\*0.25 mm i.d.; 0.25 mum film thickness). The SPME fibre was desorbed for 5 min at 250degC, He was used as carrier gas at a head pressure of 76 kPa and the column was temperature-programmed from 150degC (3 min hold) to 200degC at 5degC/min and, after a 10 min hold at 200degC, the temperature was increased to 300degC at 10degC/min and held at the final temperature for 7 min. The analytes were detected by EI or CI (with CH4 as reagent gas) MS by scanning in the range m/z 40-400 with a scan time of 1 s. PAH

detected in fumes produced by heating two types of bitumen, with different penetration grades, at 120 and 160degC are listed.

(72) 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, 2000, Vol.12, No.1, pp.25-32

IS: 1040-7685

DT: Article

NA: Dept. Quim. Anal., Nutricion y Bromatol., Fac. Quim., Univ.

Santiago Compostela, 15706 Santiago de Compostela, Spain

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 mum 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.

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(84) TI: Solid Phase Microextraction: A Practical Guide.

AU: ScheppersWercinski\_SA

PU: Marcel Dekker, New York, NY, USA

IS: 0-82-477058-7

DT: Book

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.

CN: extraction, micro-, solid-phase (SPME), theory and applications of, book

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(139) 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, 1999, Vol.50, No.3-4, pp.247-252

IS: 0009-5893

DT: Article

NA: School Pharm., Univ. Oslo, 0316 Oslo, Norway

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 (30m\*0.25mm 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.

MX: methylenedioxyamphetamine (42542-10-9) analysis of impurities in, by SPME-GC and SPME-GC-MS

CN: chromatography, gas (GC); mass spectrometry (MS); extraction, micro-, solid-phase (SPME), of impurities, from methylenedioxyamphetamine, polydimethylsiloxane-divinylbenzene-coated silica fibres as, for analysis by GC and GC-MS

(140) TI: Optimization of the SPME device design for field applications.

AU: Mueller\_L, Gorecki\_T, Pawliszyn\_J

JN: Fresenius' Journal of Analytical Chemistry, 1999, Vol.364,

No.7, pp.610-616

IS: 0937-0633

DT: Article

NA: janusz@sciborg.uwaterloo.ca, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

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 (30m\*0.25mm i.d.) coated with SPB-5 (1 µm). H<sub>2</sub> was used as the carrier gas at 30 psi with a ramped oven temperature from 40-100degC.

AN: organic compounds, volatile, detmn. of, in environmental materials, by GC, SPME in

MX: environmental materials, detmn. of VOC in, by GC, SPME in

CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), of VOC, from environmental materials, field applications of (141)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, 1999, Vol.18, No.8, pp.557-568

IS: 0165-9936

DT: Article

NA: Dept. Quim. Anal. Quim. Org., Univ. Rovira I Virgili, 43005 Tarragona, Spain

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).

AN: pollutants, detmn. of organic, in environmental materials, by SPME, review

MX: soil, detmn. of organic pollutants in, by SPME, review

waters, natural, detmn. of organic pollutants in, by SPME, review air, detmn. of organic pollutants in, by SPME, review environmental materials, detmn. of organic pollutants in, by SPME, review

CN: extraction, micro-, solid-phase (SPME), in detmn. of organic pollutants, in environmental materials, review; chromatography, gas (GC); chromatography, liquid, high-performance (HPLC)

(142) 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, 1999, Vol.50, No.3-4, pp.167-172

IS: 0009-5893

DT: Article

NA: Lab. Ind. Toxicol., Dept. Clinical Med., Nephrol. and Health Sci., Univ. Parma Med. School, 43100 Parma, Italy

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 (30m\*0.25mm i.d.) coated with HP-5MS (0.25 µm), operated with temperature programming from 45degC (held for 3 min) to 120degC (held for 1min) 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%.

AN: hydrocarbons, aromatic, detmn. of mono-, in blood and urine, for environmental monitoring of air, by GC, SPME in

MX: blood, detmn. of monoaromatic hydrocarbons in, for environmental monitoring of air, by GC, SPME in urine, detmn. of monoaromatic hydrocarbons in, for environmental monitoring of air, by GC, SPME in air, monitoring of monoaromatic hydrocarbons in, by analysis of blood and urine, by GC, SPME in

CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), of monoaromatic hydrocarbons, from blood and urine, adsorbents for, polydimethylsiloxane-coated silica fibres as, for detmn. by GC

(143) 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, 1999, Vol.852, No.2, pp.589-595

IS: 0021-9673

DT: Article

NA: erosen@mail.zserv.tuwien.ac.at, Inst. Anal. Chem., Vienna Univ.Technol., 1060 Vienna, Austria

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. <=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).

AN: organic compounds, sulfur, detmn. of, in air, by GC-AES, sampling in

MX: air, detmn. of sulfur organic compounds in, by GC-AES, sampling in

CN: sampling, of air, for sulfur, adsorbents for, Nafion membrane dryers and ozone scrubbing material as, for detmn. by GC; chromatography, gas (GC); spectrometry, atomic-emission (AES)

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

AU: Lesellier\_E

JN: Analisis, 1999, Vol.27, No.4, pp.363-368

IS: 0365-4877

DT: Article

NA: LETIAM, IUT Orsay, 91400 Orsay, France

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 CO<sub>2</sub> 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<sub>damax</sub>. 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.

AN: hydrocarbons, polycyclic aromatic, detmn. of, in water, by SFC,

SPME in

MX: waters, natural, detmn. of PAH in, by SFC, SPME in

CN: extraction, micro-, solid-phase (SPME), of PAH, from water; chromatography, supercritical-fluid (SFC)

(145) 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, 1999, Vol.50, No.3-4, pp.155-160

IS: 0009-5893

DT: Article

NA: Inst. Natl. Environ. Ind. et Risques (INERIS), 60550 Verneuil en Halatte, France

AB: Fifteen glycol ethers (GE; listed) in water were extracted by SPE on 75 µm Carboxen SPME fibre (specific surface area 1200m<sup>2</sup>/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<sup>2</sup> = 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%.

AN: glycol ethers, detmn. of, in water, by SPME-GC

MX: waters, natural, detmn. of glycol ethers in, by SPME-GC

CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), of glycol ethers, from water, adsorbents for, carboxen-polydimethylsiloxane-coated silica fibres as, for detmn. by GC

(146) 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, 1999, Vol.33, No.18, pp.3254-3259

IS: 0013-936X

DT: Article

NA: ramos@chem.vu.nl, Dept. Anal. Chem. Toegepaste, Free Univ. Boelelaan, 1081 HV Amsterdam, Netherlands

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 H<sub>2</sub>O. 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 N<sub>2</sub> 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<sup>2</sup>=0.973) with published octanol-H<sub>2</sub>O partition coefficients demonstrating the applicability of the method for adsorption equilibrium studies.

AN: pesticides, detmn. of, in soil, by filtration-SPME-GC-MS

MX: soil, detmn. of pesticides in, by filtration-SPME-GC-MS

CN: filtration; extraction, micro-, solid-phase (SPME); chromatography, gas (GC); mass spectrometry (MS)

(147) TI: Determination of VOC contamination in borehole sediments by headspace-SPME-GC analysis.

AU: Dermietzel\_J, Strenge\_G

N: Fresenius' Journal of Analytical Chemistry, 1999, Vol.364, No.7, pp.645-647

IS: 0937-0633

DT: Article

NA: Dept. Hydrogeol., UFZ Centre. Environ. Res. Leipzig-Halle, 06120 Halle/Saale, Germany

AB: Sediment (5-20 g) was shaken with 50-500 ml Ar purged H<sub>2</sub>O 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.

AN: organic compounds, volatile, detmn. of, in borehole sediments, by headspace SPME-GC

MX: sediments, detmn. of VOC in borehole, by headspace SPME-GC

CN: extraction, micro-, solid-phase (SPME); chromatography, gas (GC)

(148) 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, 1999, Vol.364, No.7, pp.625-630

IS: 0937-0633

DT: Article

NA: Energy and Environ. Res. Centre, Univ. North Dakota, Grand Forks, ND 58202-9018, USA

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.

AN: pyrethrins, detmn. of, by SPME-GC-MS

MX: pesticides, detmn. of pyrethrins in, by SPME-GC-MS shampoo, detmn. of pyrethrins in, by SPME-GC-MS

CN: extraction, micro-, solid-phase (SPME); chromatography, gas (GC); mass spectrometry (MS)

(149) 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, 1999, Vol.71, No.13, pp.2417-2422

IS: 0003-2700

DT: Article

NA: cnerin@posta.unizar.es, Dept. Anal. Chem., Centro Politecnico Superior, Univ. Zaragoza, 50015 Zaragoza, Spain

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 1min) 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 H<sub>2</sub>O 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.

AN: pesticides, organochlorine, detmn. of, in food simulants, by GC, optimization of SPME in pesticides, organophosphorus, detmn. of, in food simulants, by

GC, optimization of SPME in

MX: foods, detmn. of pesticides in simulants of, by GC,

optimization of SPME in

CN: extraction, micro-, solid-phase (SPME), of organochlorine and organophosphorus pesticides, from food simulants, optimization of, in detmn. by GC; optimization, of SPME of pesticides from food simulants, in detmn. by GC; chromatography, gas (GC)

(150) 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, 1999, Vol.849, No.1, pp.293-297

IS: 0021-9673

DT: Article

NA: qaenol@urv.es., Dept. Quim. Anal. i Quim. Org., Fac. Enologia Tarragona, Univ. Rovira i Virgili, 43005 Tarragona, Spain

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.

AN: thiols, detmn. of, in wine, by headspace SPME-GC sulfides, detmn. of, in wine, by headspace SPME-GC disulfides, detmn. of, in wine, by headspace SPME-GC

MX: wine, detmn. of disulfides, sulfides and thiols in, by headspace SPME-GC CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), of disulfides, sulfides and thiols, from wine, adsorbents for, Carboxen-poly(dimethylsiloxane)-coated silica fibres as, for detmn. by GC

Subject: spme2000-2 Date: Tue, 19 Sep 2000 12:56:20 +0100 (BST)From: BIDS RSC service <bids\_rsc@beta.bids.ac.uk>To: carlopez@matematicas.udea.edu.co, carlopez@matematicas.udea.edu.co

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Database: Analytical Abstracts

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(1) TI: Nonequilibrium solid-phase microextraction for determination of the freely dissolved concentration of hydrophobic organic compounds: matrix effects and limitations.

AU: Oomen\_AG, Mayer\_P, Tolls\_J

JN: Analytical Chemistry, 2000, Vol.72, No.13, pp.2802-2808

IS: 0003-2700

DT: Article

NA: a.oomen@ritox.vet.uu.nl, Environ. Toxicol. & Chem., Res. Inst. Toxicol. (RITOX), Utrecht Univ., 3508 TD Utrecht, Netherlands AB: Solid-phase microextraction (SPME) has recently been applied to measure the freely dissolved concentration, as opposed to the total concentration, of hydrophobic substances in aqueous solutions. This requires that only the freely dissolved analytes contribute to the concentration in the SPME fiber coating. However, for nonequilibrium SPME the sorbed analytes that diffuse into the unstirred water layer (UWL) adjacent to the SPME fiber can desorb from the matrix and contribute to the flux into the fiber. These processes were described as a model.

Experimentally, an equilibrated and disconnected headspace was used as a reference for the freely dissolved concentration. The expected contribution of desorbed analytes to the uptake flux was measured for PCB no. 52 in a protein-rich solution, while it was not measured in a matrix containing artificial soil. On the basis of the present study, a contribution of desorbed analytes to the uptake flux is expected only if (1) the rate-limiting step of the uptake process is diffusion through the UWL, (2) the concentration of the sorbed analyte is high, and (3) desorption from the matrix is fast. AN: organic compounds, extraction of, from chyme, soil and water, by SPME gamma-HCH (58-89-9) biphenyl, 2,2',5,5'-tetrachloro- (35693-99-3) biphenyl, 2,3',4,4',5-pentachloro- (31508-00-6) biphenyl, 2,2',4,4',5,5'-hexachloro- (35065-27-1) biphenyl, 2,2',3,4,4',5,5'-heptachloro- (35065-29-3)

MX: chyme, extraction of organic compounds from, by SPME waters, natural, extraction of organic compounds from, by SPME soil, extraction of organic compounds from, by SPME

CN: extraction, micro-, solid-phase (SPME), of organic compounds, from chyme, soil and water

(2) TI: In-tube solid-phase microextraction coupled to capillary LC for carbamate analysis in water samples.

AU: Gou\_Y, Pawliszyn\_J

JN: Analytical Chemistry, 2000, Vol.72, No.13, pp.2774-2779

IS: 0003-2700

DT: Article

NA: Guelph-Waterloo Centre for Graduate Work in Chemistry, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada AB: Recently, the on-line sample preparation technique, in-tube solid-phase microextraction (SPME), was successfully implemented with a Hewlett-Packard 1100 HPLC system for analysis of carbamates in water samples. This paper describes the coupling of in-tube SPME to capillary LC and explores its utility as a sample preparation method in that format, relative to conventional LC. The Hewlett-Packard HPLC system was upgraded to a capillary LC system using commercially available accessories from LC Packings. The combination of in-tube SPME with a capillary LC system was expected to build on the merits of both in-tube SPME and the capillary LC to generate a sensitive method with an easy, effective, and efficient sample preparation. Due to the relatively large effective injection volume of the in-tube SPME technique (30-45 µl), on-column focusing was employed in order to achieve good chromatographic efficiency. Excellent sensitivity was achieved with very good method precision. For the carbamates studied, the RSD of retention time was between 0.5 and 0.8% under 4 µl/min microgradient conditions. The RSD of peak area counts was between 1.5 and 4.6%. The detection limits for all carbamates studied were less than 0.3 µg/l and, for carbaryl, just 0.02 µg/l (20 ppt). Compared with the conventional in-tube SPME/LC method, the LODs were lowered for carbaryl, prothion, methidathion, diazinon, chlorpyrifos, and barban, by factors of 24, 45, 42, 81, 62, and 56, respectively. The optimized method was successfully applied to the analysis of carbamates in surface water samples.

AN: carbamic acid esters, detmn. of, in water, by SPME-LC carbaryl (63-25-2) prothion (122-42-9) methidathion (2032-65-7) diazinon (2631-37-0) chlorpyrifos (101-21-3) barban (101-27-9)

MX: waters, natural, detmn. of carbamic acid esters in, by SPME-LC

CN: extraction, micro-, solid-phase (SPME), coupled with LC, in detmn. of carbamates, in water; chromatography, liquid (LC), coupled with SPME, in detmn. of carbamates, in water

(3) TI: The comparison of toluene determination between headspace solid-phase microextraction and headspace methods in glue-sniffer's blood and urine samples.

AU: Kim\_NY, Park\_SW

JN: Journal of Forensic Sciences, 2000, Vol.45, No.3, pp.702-707

IS: 0022-1198

DT: Article

NA: Dept. Forensic Sci., Natl. Inst. Sci. Investigation, Seoul 158-097, South Korea

AB: Headspace SPME with use of a replaceable extraction fibre coated with 0.1 mm of polydimethylsiloxane is recommended. The exposed fibre was inserted into the GC injection port, and toluene was determined on an HP-5MS 5% diphenyl/95% dimethylsiloxane column (30 m \* 0.25 mm i.d.; film thickness 0.25 µm) operated at 60degC with He as carrier gas (1 ml/min) and selected-ion monitoring at m/z 91 and 65 (88 and 58 for 1,4-dioxane as internal standard). The calibration graph was linear for 0.01-10 µg/ml of toluene (cutoff value 0.1 µg/ml).

AN: toluene (108-88-3) detmn. of, in blood and urine, by GC-MS, headspace SPME in

MX: blood, detmn. of toluene in, by GC-MS, headspace SPME in urine, detmn. of toluene in, by GC-MS, headspace SPME in CN: chromatography, gas (GC); mass spectrometry (MS); extraction, micro-, solid-phase (SPME), headspace, of toluene, from blood and urine, for detmn. by GC-MS; headspace analysis

(4) TI: Automated multiple solid-phase microextraction. An approach to enhance the limit of detection for the determination of pesticides in water.

AU: Lipinski\_J

JN: Fresenius' Journal of Analytical Chemistry, 2000, Vol.367, No.5, pp.445-449

IS: 0937-0633

DT: Article

NA: SOFIA GmbH, 12489 Berlin, Germany

AB: A method was developed to decrease the limit of detection (LOD) for pesticide residue analysis in water using multiple SPME. To enhance the absolute amount transferred to the GC column an enrichment step is integrated in the SPME/GC-analysis. A series of several extraction and desorption steps are performed and the analytes are trapped at the front of the cold GC column before the GC analysis is started. The parameters mainly influencing this enrichment are the equilibrium time, the slope of the adsorption time/peak area profile at its start, the number and the duration of the extraction steps. The role of these parameters was investigated.

AN: pesticides, detmn. of, in water, by GC, automated SPME inchloroneb (2675-77-6) tecnazene (117-18-0) trifluralin (1582-09-8)benfluralin(1861-40-1) bromoxynil(1689-84-5) dicloran (99-30-9) propachlor (1918-16-7) gamma-HCH (58-89-9) quintozone (82-68-8) fluchloralin (33245-39-5) terbacil (5902-51-2) chlorothalonil (1897-45-6) bromocyclen (1715-40-8) vinclozolin (50471-44-8) alachlor (15972-60-8) bromacil (314-40-9) metolachlor (51218-45-2) aldrin (309-00-2) chlorthal-dimethyl (1861-32-1) trichloronate (327-98-0) chlozolinate (72391-46-9) isodrin (465-73-6) metazachlor (67129-08-2) heptachlor expoxide (1024-57-3) triclosan (3380-34-5) endosulfan (115-29-7) endosulfan sulfate (1031-07-8) pp'-TDE (72-54-8) pp'-DDT (50-29-3) tetrasul (2227-13-6) bromopropylate (18181-80-1) benzene, hexabromo- (87-82-1) mirex (2385-85-5) prochloraz (67747-09-5 MX: waters, natural, detmn. of pesticides in, by GC, automated SPME in CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), automated multiple, of pesticides, from water, for detmn. by GC; automation, of SPME, of pesticides, from water, for detmn. by GC

(5) TI: Determination of methylmercury by solid-phase microextraction inductively coupled plasma mass spectrometry: a new sample introduction method for volatile metal species.

AU: Mester\_Z, Lam\_J, Sturgeon\_R, Pawliszyn\_J

JN: Journal of Analytical Atomic Spectrometry, 2000, Vol.15, No.7, pp.837-842

IS: 0267-9477

DT: Article

NA: zoltan.mester@nrc.ca, Inst. Natl. Measurement Standards, Natl.

Res. Council Canada, Ottawa, ON K1A 0R9, Canada

AB: Direct coupling of solid-phase microextraction (SPME) with inductively coupled plasma mass spectrometry (ICP MS) is described for methylmercury speciation. A thermal desorption interface, consisting of a heated, glass-lined splitless-type GC injector, was placed directly at the base of the torch to minimize the length of transfer line. This arrangement provides for fast desorption and high sample introduction efficiency. Direct liquid immersion and headspace extraction of methylmercury was studied, including the effect of temperature and time on extraction efficiency. For clean solutions, immersion sampling SPME provided good sensitivity that was linear over two orders of magnitude

whereas headspace sampling showed 15% lower sensitivity, but a linear range of more than three orders of magnitude. The detection limit for headspace methylmercury sampling was 0.2 ng/ml. Calibration by the method of additions using direct extraction revealed a severe matrix effect with biological tissue samples, diminishing the methylmercury response 70-fold, whereas that obtained by headspace extraction was statistically indistinguishable from signals generated using matrix free standards. Analytical results showed good agreement between certified and measured values for analysis of National Research Council Canada (NRCC) reference materials, DORM-2, (Dogfish muscle) and DOLT-2 (Dogfish liver).

AN: mercury, methyl- (22967-92-6) detmn. of, in fish, by SPME-ICP MS, interfaces for

MX: fish, detmn. of methylmercury in, by SPME-ICP MS, interfaces for

CN: mass spectrometry, inductively coupled plasma (ICP MS), coupled with SPME, interfaces for, thermal desorption, in detmn. of methylmercury in fish; extraction, micro-, solid-phase (SPME), coupled with ICP MS, interfaces for, thermal desorption, in detmn. of methylmercury in fish

(6) TI: Multiple solid-phase microextraction. AU: Koster\_EHM, deJong\_GJ

JN: Journal of Chromatography, A, 2000, Vol.878, No.1, pp.27-33

IS: 0021-9673

DT: Article

NA: Dept. Anal. Chem. and Toxicol., Univ. Centre Pharmacy, Univ. Groningen, 9713 AV Groningen, Netherlands

AB: Theoretical aspects of multiple solid-phase microextraction are described and the principle is illustrated with the extraction of lidocaine from aqueous solutions. With multiple extraction under non-equilibrium conditions considerably less time is required in order to obtain an extraction yield that is equal to that of one extraction at equilibrium. On the other side, the extraction yield can be increased if multiple extraction is performed with the same total time as is needed for one extraction at equilibrium time. The effect of multiple extraction is strongly dependent on the value of the partition constant and for practical use the length of the desorption time is important. A good agreement between theoretical and experimental data has been obtained. Chromatograms are presented showing the potential of multiple solid-phase microextraction.

AN: lignocaine (137-58-6) extraction of, from phosphate buffer, by

SPME, mathematical models for CN: extraction, micro-, solid-phase (SPME), multiple, theoretical aspects of, mathematical models for; mathematical models, for studying theoretical aspects of multiple SPME

(7) TI: Determination of butyltin compounds in aqueous samples by gas chromatography with flame photometric detector and headspace solid-phase microextraction after in situ hydride derivatization.

AU: Jiang\_GB, Liu\_JY

JN: Analytical Sciences, 2000, Vol.16, No.6, pp.585-588

IS: 0910-6340

DT: Article

NA: gbjiang@mail.rcees.ac.cn, Res. Center Eco-Environ. Sci., Chinese Acad. Sci., Beijing 100085, China

AB: A method for the extraction and determination of butyltin species in aqueous samples by SPME combined with capillary GC-flame photometric detector (GC-FPD) is described.

The butyltin species was converted to its hydride form by NaBH<sub>4</sub> in a closed headspace vial prior to extraction. A laboratory-assembled SPME device including a fused-silica fibre and a modified microsyringe protection part was used throughout the experiment. The extraction was an equilibrium process that depended on the butyltin species partitioning between the liquid phase and a fibre. When the equilibrium was reached, the fibre was directly transferred to a GC column under the protection of a microsyringe, where the analyte was thermally desorbed inside the heated injector and subsequently separated in a HP-1 capillary column and detected by a laboratory-made FPD using quartz surface-induced Sn emission. The detection limits based on the signal = 3\* baseline noise were 0.2, 0.2, 0.1 and 0.02 mug/l for monobutyltin (MBT), dibutyltin (DBT), tributyltin (TBT) and tetrabutyltin (TeBT), respectively. The recovery ranged from 85-117%. The proposed method has been applied to determination of butyltin species in various aqueous samples.

AN: organotin compounds, detmn. of, in aqueous samples, by GC, headspace SPME in, in situ hydride derivatization in stannane, butyl- (2406-65-7) stannane, dibutyl- (1002-53-5) stannane, tributyl- (688-73-3) stannane, tetrabutyl- (1461-25-2)

CN: extraction, micro-, solid-phase (SPME), headspace, of organotin compounds, from aqueous samples, in detmn. by GC, in situ hydride derivatization in; chromatography, gas (GC), headspace SPME in, in detmn. of organotin compounds, in aqueous samples

(8) TI: Evaluation of solid-phase microextraction in combination with gas chromatography (SPME-GC) as a tool for quantitative bioanalysis.

AU: AbdelRehim\_M, Bielenstein\_M, Arvidsson\_T

JN: Journal of Microcolumn Separations, 2000, Vol.12, No.5, pp.308-315

IS: 1040-7685

DT: Article

NA: Astra Pain Control, 151 85 Sodertalje, Sweden

CO: Presented at the Twenty-First International Symposium on Capillary Chromatography and Electrophoresis, held in Park City, UT, USA, 20-24 Jun 1999

AB: Solid-phase microextraction in combination with capillary gas chromatography and a nitrogen-phosphorus detector as a bioanalysis tool was investigated. Lidocaine (lignocaine) and three of its metabolites were used as model compounds, and human plasma and urine samples were used in this evaluation.

Carbowax-divinylbenzene, polyacrylate, and polydimethylsiloxane fibers were tested. Absorption times were studied for all analytes separately. Carbowax-divinylbenzene fiber gave highest recovery in plasma samples compared to other fibers. Effects of temperature, addition of salt and agitation of the sample were studied. Recovery from plasma was improved by 2-4 times at pH 9 compared to pH 3. This is due to analytes not charged at high pH. Recovery from water was 2-4 times higher than from plasma using Carbowax-divinylbenzene coated fiber. This is due to protein binding of analytes in plasma. Chromatographic selectivity was high and all metabolites were well separated. Calibration graphs were linear for all metabolites in human plasma and urine in the range 0.035-7.7muM for lidocaine and 2,6-xylylidine and from 0.1-3.5muM for glycinexylidide (GX) and monoethylglycinexylidide (MEGX). Precision, measures as relative standard deviation, was less than 15% and accuracy was in the range 80-115%. Limits of quantitation using plasma were 0.035muM (8 ng/ml), 0.035muM (4 ng/ml), 0.100muM (18 ng/ml), and 0.100muM (21 ng/ml) for lidocaine, 2,6-xylylidine, GX, and MEGX, respectively.

AN: lignocaine (137-58-6) detmn. of, and its metabolites, in plasma and urine, by SPME-GC aniline, 2,6-dimethyl- (87-62-7) glycinexylidide (18865-38-8) monoethylglycinexylidide (7728-40-7)

MX: blood plasma, detmn. of lignocaine and its metabolites in, by SPME-GC urine, detmn. of lignocaine and its metabolites in, by SPME-GC CN: extraction, micro-, solid-phase (SPME), of lignocaine and its metabolites, from plasma and urine, adsorbents for, poly(Carbowax-divinylbenzene)-coated silica fibres as, for detmn. by GC; chromatography, gas (GC)

(9) TI: Application of solid phase micorextraction and gas chromatography with electron capture detector for the analysis of chlorinated pesticides in water.

LA: French

AU: Boussahel\_R, Bouland\_S, Montiel\_A, Moussaoui\_KM

JN: Spectra Analyse, 2000, Vol.29, No.213, pp.27-29

IS: 1255-2909

DT: Article

NA: SAGEP, 75014 Paris, France

AB: A Supelco 100 µm poly(dimethylsiloxane)-coated fibre was immersed in a 1 ml sample portion (containing 0.2 mg/l of pentachloroanisole as internal standard) and pre-concentration of analytes was carried out for 20 min in an ultrasonic bath.

The analytes were desorbed at 220degC (2 min) and separated on a PTE5 column (30 m \* 0.25 mm i.d.; 0.25 µm film thickness) with temperature-programming from 100 to 180degC at 10degC/min and, after a 5 min hold, from 180 to 250degC at 5degC/min with a 5 min hold at the final temperature. Helium was used as carrier gas and the analytes were detected by ECD. Calibration graphs were generally linear in the ranges 0.025-40 ppb and limits of detection were in the range 0.005-0.025 ppb.

AN: pesticides, organochlorine, detmn. of, in water, by GC, ECD and SPME in gamma-HCH (58-89-9) heptachlor (76-44-8) op'-DDE (3424-82-6) alpha-endosulfan (959-98-8) pp'-DDE (72-55-9) beta-endosulfan (33213-65-9) op'-DDT (789-02-6) pp'-DDT (50-29-3)

MX: water (7732-18-5) detmn. of organochlorine pesticides in, by GC, ECD and SPME in CN: extraction, micro-, solid-phase (SPME), of organochlorine pesticides from water, for detmn. by GC, ECD in; chromatography, gas (GC), in detmn. of organochlorine pesticides, in water, ECD and SPME in

(10) TI: SPME of explosives before analysis by capillary GC.

LA: French

AU: Mindrup\_RF

JN: Spectra Analyse, 2000, Vol.29, No.213, p.30

IS: 1255-2909

DT: Article

NA: Sample Handling, Supelco, Bellefonte, PA, USA

AB: A poly(dimethylsiloxane)-divinylbenzene impregnated fibre (65 µm) was immersed directly in the sample solution for 30 min.

The analytes were desorbed for 5 min at 250degC and separated on a cyanopropyl column (30 m \* 0.25 mm i.d.; 0.25 µm film thickness) with temperature-programming from 95degC (3 min hold) to 182 degC (held for 4 min) at 8degC/min and then to 250degC (held for 6 min) at 8degC/min. Nitrogen was used as carrier gas at 60 ml/min and detection was

by ECD. Application of the technique is exemplified by the determination of 12 nitroaromatic explosives in water at concentrations of less than 50 ppb.

AN: explosives, detmn. of nitroaromatic, in water, by GC, ECD and

SPME in benzene, nitro- (98-95-3) toluene, 2-nitro- (88-72-2) toluene, 3-nitro- (99-08-1) toluene, 4-nitro- (99-99-0) toluene, 2,6-dinitro- (606-20-2) benzene, m-dinitro- (99-65-0) toluene, 2,4-dinitro- (121-14-2) toluene, 2,4,6-trinitro- (118-96-7) benzene, 1,3,5-trinitro- (99-35-4) p-toluidine, 3,5-dinitro- (19406-51-0) o-toluidine, 3,5-dinitro- (35572-78-2) aniline, N-methyl-N,2,4,6-tetranitro- (479-45-8)

MX: waters, natural, detmn. of nitroaromatic explosives in, by GC, ECD and SPME in

CN: extraction, micro-, solid-phase (SPME), of nitroaromatic explosives, from water, for detmn. by GC, ECD in; chromatography, gas (GC)

(11) TI: Analysis of benzothiazole in Italian wines using headspace solid-phase microextraction and gas chromatography-mass spectrometry.

AU: Bellavia\_V, Natangelo\_M, Fanelli\_R, Rotilio\_D

JN: Journal of Agricultural and Food Chemistry, 2000, Vol.48, No.4, pp.1239-1242

IS: 0021-8561

DT: Article

NA: rotilio@cmns.mnegri.it, Environ. Health Centre "Gennaro Paone", Inst. Ricerche Farmacologiche "Mario Negri", 6600 Santa Maria Imbaro, Italy

AB: Wine (4 ml), containing benzothiazole (I) was placed in a sealed vial with a fused silica fibre exposed to the vapour phase for 15 min at 50degC. Samples were then thermally desorbed and injected directly onto a fused-silica GC column (30 m \* 0.25 mm i.d.) coated with MDN-5S (0.25 µm). Separation was effected with temperature programming from 50degC (held for 1 min) to 150degC (held for 1 min) at 20degC/min then to 280degC at 4degC/min, with He as carrier gas (1 ml/min) and EIMS detection operated in selected-ion monitoring mode at m/z 108 and 135. The calibration graph was linear up to 100 ppb of I with a detection limit of 45 ppt. Results (tabulated) for 12 Italian wine varieties are discussed.

AN: benzothiazole (95-16-9) detmn. of, in wine, by GC-MS, SPME in MX: wine, detmn. of benzothiazole in, by GC-MS, SPME in

CN: chromatography, gas (GC); mass spectrometry (MS); extraction, micro-, solid-phase (SPME), of benzothiazole, from wine, adsorbents for, poly(Carbowax-divinylbenzene)-coated silica fibres as, for detmn. by GC-MS

(12) TI: Sensor analyses of volatile components derived from earth-almond *Cyperus esculentus* L. and carob *Ceratonia siliqua* L.

AU: Cantalejo\_MJ

JN: Zeitschrift fuer Lebensmittel-Untersuchung und -Forschung, 1999, Vol.208, No.5-6, pp.373-378

IS: 0044-3026

DT: Article

NA: iosune.cantalejo@upna.es, Dept. Quim. Aplicada, Area Tecnol. Alimentos, Univ. Publica Navarra, 31006 Pamplona, Spain AB: An electronic nose (Aroma Scanner), based on a sensor array of 32 conducting polymer sensors, and GC-MS were compared for the characterization of aroma compounds from carob and earth-almonds (details given). Volatile compounds were isolated from raw and roasted samples by high vacuum

distillation, and the distillate further treated by SPE (polystyrene divinylbenzene) and SPME for GC analysis. Three pattern recognition techniques were applied to the data: principal-components analysis, cluster analysis and neural network analysis. Results showed the Aroma Scanner was most suited to monitoring the earth-almond roasting process. The sensor is a complementary technique to GC-MS.

AN: aroma compounds, characterization of, in carob and earth-almond, by GC-MS and sensory evaluation, chemometrics in, sensors for

MX: carob, characterization of aroma compounds in, by GC-MS and sensory evaluation, chemometrics in, sensors for almond, characterization of aroma compounds in earth-, by GC-MS and sensory evaluation, chemometrics in, sensors for

CN: chemometrics, pattern recognition techniques, in characterization of aroma compounds, in carob and earth-almonds, by GC-MS and sensory evaluation; sensors, for aroma compounds, in carob and earth-almonds, electronic noses, 32 polymer sensor array; chromatography, gas (GC); mass spectrometry (MS); sensory evaluation

(13) TI: Drugs, and other things, from mummies.

AU: Counsell\_D, Lunt\_L, Sutherland\_EL

JN: CAST, Chromatography and Separation Technology, 2000, No.13, pp.6-10

IS: 1461-1236

DT: Article

NA: Univ. Manchester, Manchester, UK

AB: Several analyses of Egyptian mummies are briefly described. Some samples were extracted with polar and non-polar solvents using ultrasonification or Soxhlet apparatus, concentrated under N<sub>2</sub> and analysed by GC-MS on a DB5-MS column (30 m) with temperature programming from 40-200degC at 5degC/min with mass spectra recorded in positive electron impact full scan mode and compared with spectra in other databases. This approach was used to identify C16 and C18 fatty acids, cholesterol, other steroids, triterpenes and phthalates. Opiates, cocaine and nicotine were not detected. Microscale sealed vessel pyrolysis (a useful alternative to solvent extraction) and GC-MS was used in the analysis of resins and bitumin. The presence of polyalkylpolycyclic aromatic hydrocarbons and alkylated monocyclic compounds showed resins more likely to result from plant material than from the mummification process. Lotus flowers were analysed for psychomimetic agents using solvent extraction and SPME followed by GC-MS. This approach revealed the presence of benzyl and cinnamyl alcohols. The merits of the different procedures and the significance of the results obtained are briefly discussed.

AN: drugs, detmn. of, in mummified human body, by GC-MS, sample prep. In biochemical compounds, detmn. of, in mummified human body, by GC-MS, sample prep. in fatty acids cholesterol (57-88-5) steroids terpenes, tri-phthalic acid esters opiates cocaine (50-36-2) nicotine (54-11-5) benzyl alcohol (100-51-6) cinnamyl alcohol (104-54-1) hydrocarbons, polycyclic aromatic

MX: body, human, detmn. of biochemical compounds and drugs in mummified, by GC-MS, sample prep. in CN: chromatography, gas (GC); mass spectrometry (MS); sample preparation, of mummified human body, for detmn. of biochemical compounds and drugs in, by GC-MS

(14) TI: Simple and rapid determination of amphetamine, methamphetamine, and their methylenedioxy derivatives in urine by automated in-tube solid-phase microextraction coupled with liquid chromatography-electrospray ionization mass spectrometry.

AU: Kataoka\_H, Lord\_HL, Pawliszyn\_J

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.4, pp.257-265

IS: 0146-4760

DT: Article

NA: Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada AB: A simple and rapid method for the determination of amphetamine, methamphetamine, and their 3,4-methylenedioxy derivatives in urine samples was developed using automated in-tube solid-phase microextraction (SPME) coupled with liquid chromatography-electrospray ionization mass spectrometry (LC-ESI-MS). 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 stimulants were initially performed by liquid injection onto an LC column to determine spectra. Five stimulants tested in this study gave very simple ESI mass spectra, and strong signals corresponding to  $[M + H]^+$  were observed for all stimulants. The stimulants were well separated with a Supelcosil LC-CN column using acetonitrile/50mM-ammonium acetate (15:85) as a mobile phase.

In order to optimize the extraction of stimulants, several in-tube SPME parameters were examined. The optimum extraction conditions were 15 draw/eject cycles of 35  $\mu$ l of sample in 50mM-Tris hydrochloride buffer (pH 8.5) at a flow rate of 100  $\mu$ l/min using an Omegawax 250 capillary column. The stimulants extracted by the capillary were easily desorbed by mobile phase flow, and carryover of stimulants was not observed. Using in-tube SPME-LC-ESI-MS with selected ion monitoring, the calibration curves of stimulants were linear in the range from 2 to 100 ng/ml with correlation coefficients above 0.9985 ( $n=18$ ) and detection limits ( $S/N = 3$ ) of 0.38-0.82 ng/ml. This method was successfully applied to the analysis of human urine samples without interference peaks. The recoveries of stimulants spiked into urine samples were above 81%.

AN: amphetamine derivatives, detmn. of, in urine, by SPME-LC-electrospray MS, automated systems for amphetamine (300-62-9) methylamphetamine (537-46-2) tenamphetamine (4764-17-4) methylenedioxymethamphetamine (42542-10-9) amphetamine, N-ethyl-3,4-methylenedioxy- (82801-81-8)

MX: urine, detmn. of amphetamine derivatives in, by SPME-LC-electrospray MS, automated systems for

CN: extraction, micro-, solid-phase (SPME), of amphetamine derivatives, from urine, adsorbents for, in-tube polymer-coated silica GC capillaries as, evaluation of polymers for, in detmn. by LC-electrospray MS, automated systems for; chromatography, liquid (LC);mass spectrometry, electrospray (electrospray MS); automated analysis, of amphetamine and derivatives in urine, by SPME-LC-electrospray MS

(15) TI: Determination of phthalate esters in water samples by solid-phase microextraction and gas chromatography with mass spectrometric detection.

AU: Penalver\_A, Pocurull\_E, Borrull\_F, Marce\_RM

JN: Journal of Chromatography, A, 2000, Vol.872, No.1-2, pp.191-201

IS: 0021-9673

DT: Article

NA: marce@quimica.urv.es, Dept. Quim. Anal. i Quim. Org., Univ.

Rovira i Virgili, 43005 Tarragona, Spain

AB: Solid-phase microextraction (SPME) with an 85 µm polyacrylate fiber, coupled to gas chromatography-mass spectrometry was used to determine six phthalate esters and bis(2-ethylhexyl) adipate in water samples. The variables affecting the SPME absorption process were optimized and the method developed was applied to analyze both tap and commercial mineral water samples as well as water from the Ebro river and fishing and industrial ports. For real samples, the linear range in full scan acquisition mode was between 0.02 and 10 µg/l for most compounds, and the limits of detection of the method were between 0.006 and 0.17 µg/l. Commercial water samples contained in recipients which were made from different materials were analyzed, and the influence of the material of the recipients on the concentration of phthalates was evaluated.

AN: phthalic acid esters, detmn. of, in water, by GC-MS, SPME in phthalic acid dimethyl ester (131-11-3) phthalic acid diethyl ester (84-66-2) phthalic acid dibutyl ester (84-74-2) phthalic acid benzyl butyl ester (85-68-7) phthalic acid bis-(2-ethylhexyl) ester (117-81-7) phthalic acid bis-(2-ethylhexyl) ester (117-81-7) phthalic acid dioctyl ester (117-84-0)

MX: waters, potable, detmn. of phthalic acid esters in, by GC-MS,

SPME in waters, natural, detmn. of phthalic acid esters in, by GC-MS, SPME in

CN: chromatography, gas (GC);mass spectrometry (MS); extraction, micro-, solid-phase (SPME), of phthalic acid esters, from water, adsorbents for, polyacrylate-coated silica fibres as, for detmn. by GC-MS

(16) TI: Determination of sulfur compounds in beer using headspace solid-phase microextraction and gas chromatographic analysis with pulsed flame photometric detection.

AU: Hill\_PG, Smith\_RM

JN: Journal of Chromatography, A, 2000, Vol.872, No.1-2, pp.203-213

IS: 0021-9673

DT: Article

NA: phill@becks.de, Brauerei Beck & Co., 28199 Bremen, Germany

AB: A simple and sensitive method for the analysis of volatile and semi-volatile sulfur compounds in beer at trace levels was developed using headspace solid-phase microextraction (SPME) and gas chromatography with pulsed flame photometric detection.

Different SPME fibres were tested and a Carboxen-polydimethylsiloxane coated fibre was found to be the most appropriate. The adsorption and desorption conditions were optimized. The effect of ethanol concentration in the sample on the extraction of analytes was examined. A 60 m non-polar capillary column preceded by a 10 m length of a polar column was found to be capable of separating a wide range of C1-C6 sulfur compounds. The pulsed flame photometric detector enabled increased sensitivity to be obtained over previous methods, such as dynamic headspace followed by conventional flame photometric detection or sulfur chemiluminescent detection, with high-sulfur selectivity. Two sulfur compounds, 2-methyl-1-butanethiol and 3-methylthiophene, were identified in beer for the first time.

AN: organic compounds, sulfur, detmn. of, in beer, by GC, SPME in ketone, methyl 2-thienyl (88-15-3) butane-1-thiol (109-79-5) carbon disulfide (75-15-0) cyclopentanethiol (1679-07-8) disulfide, diethyl (110-81-6) sulfide, diethyl (352-93-2) disulfide, dimethyl (624-92-0) sulfide, dimethyl (75-18-3) trisulfide, dimethyl (3658-80-8) tetrasulfide, methyl (5756-24-1) ethanethiol (75-08-1) thiirane (420-12-2) propionic acid, 3-(methylthio)-, ethyl

ester (13327-56-5) acetic acid, thio-, ethyl ester (926-67-0) methanethiol (74-93-1) propionaldehyde, 3-(methylthio)- (3268-49-3) propanol, 3-(methylthio)- (505-10-2) butane-1-thiol, 2-methyl- (1878-18-8) butane-1-thiol, 3-methyl- (541-31-3) but-2-ene-1-thiol, 3-methyl- (5287-45-furan-3-thiol, 2-methyl- (28588-74-1) propionic acid, 3-(methylthio)-, methyl ester (13532-18-8) butane-2-thiol (513-53-1) propane-2-thiol, 2-methyl- (75-66-1) acetic acid, thio-, methyl ester (1534-08-3) thiophene, 2-methyl- (554-14-3) thiophene, 3-methyl- (616-44-4) propionic acid, 3-(methylthio)- (646-01-5) acetic acid, 3-(methylthio)propyl ester (16630-55-0) pentane-1-thiol (110-66-7) propanethiol (107-03-9) propane-2-thiol (75-33-2) MX: beer, detmn. of sulfur organic compounds in, by GC, SPME in CN: chromatography, gas (GC);extraction, micro-, solid-phase (SPME), of sulfur organic compounds, from beer, adsorbents for, Carboxen-polydimethylsiloxane-coated silica fibres as, for detmn. by GC

(17) TI: Solid-phase microextraction for determining the distribution of sixteen US Environmental Protection Agency polycyclic aromatic hydrocarbons in water samples.

AU: Doong\_RA, Chang\_SM, Sun\_YC

JN: Journal of Chromatography, A, 2000, Vol.879, No.2, pp.177-188

IS: 0021-9673

DT: Article

NA: radoong@mx.nthu.edu.tw, Dept. Nuclear Sci., Natl. Tsing Hua Univ., Hsinchu 30043, Taiwan

AB: A SPME procedure has been developed for the determination of 16 US Environmental Protection Agency promulgated PAHs. Five kinds of SPME fibres were used and compared in this study. The extracted sample was analysed by GC with FID or MS. Parameters affecting the sorption of analyte into the fibres, including the sampling time, thickness of the fibre coating, and the effect of temperature, have been examined. Moreover, the feasibility of headspace SPME with different working temperatures was evaluated. The method was also applied to real samples. The 85 µm polyacrylate (PA) and 100 µm poly(dimethylsiloxane) (PDMS) fibres were shown to have the highest affinities for the selected PAHs. The PA fibre was more suitable than the PDMS fibre for the determination of low-ring PAHs while high sensitivity of high-ring PAHs was observed when a 100 µm PDMS fibre was used. The method showed good linearity between 0.1-100 ng/ml with regression coefficients ranging from 0.94-0.999. The reproducibility of the measurements between fibres was found to be very good. The precisions of PA and PDMS fibres were from 3-24 and 3-14%, respectively. Headspace SPME is a valid alternative for the determination of two- to five-ring PAHs. A working temperature of 60degC provides significant enhancement in sensitivity of two- to five-ring PAHs having low vapour pressures (>10<sup>-6</sup> mm Hg at 25degC) (1 mm Hg = 133.3 Pa) and low Henry's constants (>10 atm ml/mol) (1 atm = 1.02 \* 10<sup>5</sup>Pa).

AN: hydrocarbons, polycyclic aromatic, detmn. of, in water, by GC and GC-MS, optimization in, SPME in naphthalene (91-20-3)acenaphthylene (208-96-acenaphthylene, 1,2-dihydro- (83-32-9) fluorene (86-73-7) phenanthrene (85-01-8) anthracene (120-12-7) fluoranthene (206-44-0) pyrene (129-00-0) benz[a]anthracene(56-55-3) chrysene(218-01-9) benz[e]acephenanthrylene (205-99-2) benzo[k]fluoranthene (207-08-9) benzo[a]pyrene (50-32-8) indeno[1,2,3-cd]pyrene (193-39-5) dibenzo[ah]anthracene (53-70-3) benzo[ghi]perylene (191-24-2)

MX: waters, natural, detmn. of PAH in, by GC and GC-MS, optimization in, SPME in

CN: extraction, micro-, solid-phase (SPME), of PAH, from water, in detmn. by GC and GC-MS, optimization in; optimization, of SPME conditions, in detmn. of PAH, in water, by GC and GC-MS; chromatography, gas (GC); mass spectrometry (MS)

(18) TI: Determination of tetraethyllead by solid-phase microextraction-thermal desorption-quartz furnace atomic absorption spectrometry.

AU: Fragueiro\_MS, AlavaMoreno\_F, Lavilla\_I, Bendicho\_C

JN: Journal of Analytical Atomic Spectrometry, 2000, Vol.15, No.6, pp.705-709

IS: 0267-9477

DT: Article

NA: bendicho@uvigo.es, Dept. Quim. Anal. y Alimentaria, Univ. Vigo, 36200 Vigo, Spain

AB: A new procedure that uses a preconcentration system based on solid phase microextraction (SPME) and detection by quartz furnace AAS after thermal desorption from the microextraction fibre has been proposed for the determination of tetraethyllead in gasoline (leaded and unleaded) and water. Three different volatilizers were designed and their influence in the thermal desorption of tetraethyllead was studied. Working tetraethyllead solutions were prepared in 40 ml amber vials and sampling was performed by exposing the SPME fibre to the headspace over vigorously stirred samples for 10 min. The analytical performance characteristics of the proposed procedure were as follows: the detection limit for tetraethyllead was 0.43 ng/ml, with a RSD of 6% for the determination of 10 ng/ml of tetraethyllead (n = 5) while the calibration curve was linear up to 50 ng/ml range. The proposed procedure was finally applied to the determination of tetraethyllead in gasoline and water samples obtaining good agreement with those values obtained by an alternative method that included the direct injection of tetraethyllead via a septum in the heated volatilizer with a gas chromatographic syringe, the tetraethyllead volatilization and its transport by the gas carrier to the quartz furnace, where the analytical signal was observed. This procedure could easily be adapted for the speciation of ionic lead and tetraethyllead.

AN: lead, tetraethyl- (78-00-2) detmn. of, in gasoline and water, by SPME-quartz furnace AAS

MX: water (7732-18-5) detmn. of tetraethyllead in, by SPME-quartz furnace AAS gasoline, detmn. of tetraethyllead in, by SPME-quartz furnace AAS

CN: spectrometry, atomic-absorption (AAS), quartz furnace, coupled with SPME, in detmn. of tetraethyllead in gasoline and water; extraction, micro-, solid-phase (SPME), coupled with quartz furnace AAS, in detmn. of tetraethyllead in gasoline and water

(19) TI: Determination of acetic acid in aqueous samples, by water-phase derivatization, solid-phase microextraction and gas chromatography.

AU: Wittman\_G, vanLangenhove\_H, Dewulf\_J

JN: Journal of Chromatography, A, 2000, Vol.874, No.2, pp.225-234

IS: 0021-9673

DT: Article

NA: wittmann@chem.u-szeged.hu, Dept. Inorg. and Anal. Chem., Jozsef Attila Univ., 6701 Szeged, Hungary

AB: The direct derivatisation of acetic acid with n-hexyl chloroformate and with benzyl bromide in water was evaluated. With n-hexyl chloroformate, acetic acid did not give the n-hexyl acetate derivative, but the reaction of acetic acid with benzyl bromide in aqueous

solution resulted in the formation of benzyl acetate. The derivatisation of acetic acid with benzyl bromide and the headspace solid-phase microextraction (SPME) of benzyl acetate were optimised. Under optimum conditions, the limit of detection for acetic acid was 260nM, and the relative standard deviation of the overall procedure at 0.1mM-acetic acid was 15.6% (n = 10). A linear response was obtained in the concentration range from 5µM to 0.1mM (R<sup>2</sup> = 0.993, n = 6). Although Carbowax-divinylbenzene (CW-DVB)-coated fibres exhibited a higher extraction capacity for benzyl acetate, polyacrylate (PA) was selected, because its mechanical stability was better than that of CW-DVB fibres. Moreover, the relative standard deviation of the SPME was better with PA (1.5%, n = 10 at 10µM) than with CW-DVB-coated fibres (8%,n=10 at 10µM). Thus, a new analytical method for the quantitative determination of micromolar concentrations of acetic acid in the aqueous phase was developed. This method is based on water-phase derivatisation with benzyl bromide, headspace SPME with PA fibres and GC-FID. It was observed experimentally that benzyl alcohol formed by hydrolysis of the reagent affected the fibre-gas phase partitioning of benzyl acetate.

AN: acetic acid (64-19-7) detmn. of, in water, by GC, SPME in

MX: water (7732-18-5) detmn. of acetic acid in, by GC, SPME in

CN: chromatography, gas (GC); extraction, micro-, solid-phase (SPME), of acetic acid, from water, adsorbents for, polyacrylate-coated silica fibres as, for detmn. by GC

(20) TI: Determination of triazines in soil leachates by solid-phase microextraction coupled to gas chromatography-mass spectrometry.

AU: Zambonin\_CG, Palmisano\_F

JN: Journal of Chromatography, A, 2000, Vol.874, No.2, pp.247-255

IS: 0021-9673

DT: Article

NA: Dipt. Chim., Univ. Studi Basilicata, 85100 Potenza, Italy

AB: A solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) method was developed for the evaluation of the leachability order of selected triazines (propazine, terbuthylazine, sebuthylazine, ametryn, prometryn and terbutryn) in soil/sediment samples (organic carbon content ranging from 0.19 to 0.42%), analysing fractions collected from a soil packed microcolumn elution experiments. The procedure is fast, simple, highly sensitive and solvent free. SPME-GC-MS was also employed for the quantitative determination of triazines in the soil leachate, since the method showed good recovery yield. Detection limits were always better than 1 ng/ml. The method was tested on a contaminated landfill top soil.

Prometryn and ametryn were identified through their MS spectra and then quantified. Terbuthylazine was used to assess recovery. Results compared well with those obtained by solvent extraction followed by HPLC-UV detection.

AN: herbicides, triazine, detmn. of, in soil leachates, by SPME-GC-

MS terbutryn (886-50-0) prometryn (7287-19-6) terbuthylazine (5915-41-3) ametryn (834-12-8) sebuthylazine (7286-69-3) propazine (139-40-2)

(21) TI: Speciation of trimethyllead and triethyllead by in-tube solid-phase microextraction high-performance liquid chromatography electrospray ionization mass spectrometry.

AU: Mester\_Z, Lord\_H, Pawliszyn\_J

JN: Journal of Analytical Atomic Spectrometry, 2000, Vol.15, No.6, pp.595-600

IS: 0267-9477

DT: Article

NA: Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

AB: An analytical method has been developed for the determination of the trimethyllead (TML) and triethyllead (TEL) species in aqueous samples. In-tube solid-phase microextraction and HPLC are coupled to a quadrupole mass spectrometer using an electrospray as an ionization interface. The optimization of instrumental parameters is described, including the evaluation of three commercial GC capillaries for the in-tube SPME experiments. Elemental lead-208 (Pb<sup>+</sup>) and molecular forms of TML and TEL (m/z) were monitored simultaneously to provide complete speciation information. Results from the in-tube SPME-HPLC-ESMS experiment indicated that complete separation and detection of TML and TEL can be achieved in under 5 min. Precision is greater than 5% and estimated limits of detection are 11.3 and 12.6 ng/ml respectively for TML and TEL at a solution flow rate of 450 ul/min.

(22) TI: Headspace solid-phase microextraction-capillary gas chromatography-ICP mass spectrometry for the determination of the organotin pesticide fentin in environmental samples.

AU: Vercauteren\_J, deMeester\_A, deSmaele\_T, Vanhaecke\_F, Moens\_L, Dams\_R, Sandra\_P

JN: Journal of Analytical Atomic Spectrometry, 2000, Vol.15, No.6, pp.651-656

IS: 0267-9477

DT: Article

NA: Jordy.Vecauteren@rug.ac.be, Lab. Anal. Chem., Ghent Univ., 9000 Ghent, Belgium

AB: The extraction and preconcentration capabilities of headspace solid-phase microextraction (headspace SPME) were combined with the separation power of capillary gas chromatography (CGC) and the low limits of detection (LODs) of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of the organotin compound triphenyltin (TPhT, or fentin) in aqueous solution and in potatoes and mussels after digestion with tetramethyl ammonium hydroxide (TMAH) or KOH-ethanol.

Throughout, tricyclohexyltin (cyhexatin, TCT) was used as internal standard. Derivatization to transform TPhT and TCT into sufficiently volatile compounds was carried out with sodium tetraethylborate at pH 8 (0.2M-ammonium buffer). Headspace extraction was performed for 10-20 min at 75 or 85degC with a 100 mum polydimethylsiloxane fibre. Absorption curves showed that even at high temperature (75degC) no equilibrium conditions were obtained for either compound.

Direct aqueous SPME was also studied, but the sensitivity was 11 times lower at 25degC than observed when using headspace SPME at 75degC). After 2 min desorption of the SPME fibre at 270degC in the GC inlet, the organotin compounds were separated on a capillary column with a polydimethylsiloxane coating and transported into the ICP by means of a home-made heated transfer line. Monitoring of the selenium-120 signal by ICP-MS during the run of GC provided extremely low LODs for fentin in water: 2 pg/l (instrumental) and 125 pg/l (procedure), as well as good repeatability of 8% RSD (n = 10). For mussels and potatoes, concentrations in the range 3-10 and 0.9-3 ng/g (dryweight, as Sn), respectively were found.

(23) TI: Fiber conditioners for solid phase microextraction: design, testing, and application.

AU: Koziel\_JA, Shurmer\_B, Pawliszyn\_J

JN: Journal of High Resolution Chromatography, 2000, Vol.23, No.4, pp.343-347

IS: 0935-6304

DT: Article

NA: Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

AB: This note presents a background on traditional SPME fiber conditioning, the schematics and optimization of operational parameters for the new device, comparison of performance and suggestions for building and operating a simple SPME fiber conditioner device.

(24) TI: Determination of homocysteine by gas chromatography-mass spectrometry following treatment with chloroformates: a comment.

AU: Husek\_P

JN: Journal of Chromatography, B: Biomedical Applications, 2000, Vol.740, No.2, pp.289-290

IS: 0378-4347

DT: Article

NA: phusek@endo.cz, Inst. Endocrinol., 116 94 Praha 1, Czech Republic

AB: Comments are made on the procedure described by Myung et al. (Ibid., 1999, B727, 1) for the GC-MS determination of homocysteine following treatment with chloroformate and use of SPME. The author concludes that the procedure cannot be recommended as a reliable alternative to current methods as there seems to be some erratic conclusions and questionable steps.

(25) TI: Analysis of dichlorobenzenes in urine using gas chromatography and mass spectrometry.

LA: Chinese

AU: Liu\_JT, Hara\_K, Kashimura\_S

JN: Fenxi Huaxue, 2000, Vol.28, No.4, pp.443-445

IS: 0253-3820

DT: Article

NA: Dept. Chem., Basic Med. Sci. Coll., China Med. Univ., Shenyang 110001, China

AB: Urine (0.5 ml) was charged into a 12 ml glass phial, 0.5 ml H<sub>2</sub>O was added and the phial was sealed. A 1 µl portion of 0.05% p-xylene-d<sub>10</sub> (internal standard) in tetraethylene glycol dimethyl ether was injected into the phial and the mixture was heated at 30degC for 2 min. A 100 µm polydimethylsiloxane fibre was inserted for headspace of the phial and the volatile dichlorobenzenes (VDCB) in the headspace were extracted by SPME for 15 min. The fibre was removed and then placed in the sampling port of a GC and the VDCB were desorbed directly onto on a fused-silica capillary column (30 m \* 0.25 mm i.d.) coated with XTI (0.25 µm), operated with temperature programming from 20degC (held for 1 min) to 290degC (held for 6 min) at 30degC/min, with He as carrier gas (flow rate not given) and EIMS detection operated in selected-ion monitoring mode at m/z 146. The o-, m- and p-dichloro VDCB were cleanly separated. Calibration graphs for these compounds were linear, with detection limits of 0.01 mg/l. Such method was applied in the study of patients poisoned by dichlorobenzenes.

AN: benzene derivatives, dichloro-, detmn. of, in urine, by GC-MS MX: urine, detmn. of dichlorobenzene derivatives, by GC-MS CN: chromatography, gas (GC); mass spectrometry (MS)

(26) TI: Use of solid-phase microextraction for the quantitative determination of herbicides in soil and water samples.

AU: Hernandez\_F, Beltran\_J, Lopez\_FJ, Gaspar\_JV

JN: Analytical Chemistry, 2000, Vol.72, No.10, pp.2313-2322

IS: 0003-2700

DT: Article

NA: hernandf@exp.uji.es, Anal. Chem., Experimental Sci. Dept., Univ. Jaume I, 12080 Castellon, Spain

AB: An in-depth study of SPME optimization and application has been made, considering not only aqueous (surface water and ground water samples) but also the more complex soil samples. Seven herbicides widely used in the area of study have been selected including five triazine herbicides (atrazine, simazine, terbumeton, terbuthylazine, terbutryn), molinate, and bromacil.

Linearity range was between 0.1 and 10 ng/ml and the repeatability below 10% when applying the optimized SPME procedure to water samples. Reproducibility was found to be lower than 20% at the 1 ng/ml level, and the limits of determination in environmental water samples using GC/MS (SIM mode) were well below 0.1 ng/ml (values ranging from 10 to 60 ng/l). Extraction of selected herbicides from soil was carried out by microwave-assisted solvent extraction using methanol in screw-capped vials, leading to recoveries over 80% in spiked soil samples at the 5-200 ng/g level. SPME application over methanolic soil extracts required a 10-fold dilution with distilled water. The recommended procedure was found to be fully applicable for quantitative determination of selected herbicides in soils containing low organic matter content with coefficients of variation below or around 10% and limits of determination ranging from 1 to 10 ng/g. Both procedures were applied to real-world surface water and soil samples where several pesticides were detected including atrazine, simazine, terbuthylazine, and molinate.

(27) TI: Trace analysis of ten chlorinated benzenes in water by headspace solid-phase microextraction.

AU: He\_Y, Wang\_Y, Lee\_HK

JN: Journal of Chromatography, A, 2000, Vol.874, No.1, pp.149-154

IS: 0021-9673

DT: Article

NA: chmleehk@nus.edu.sg, Dept. Chem., National Univ. Singapore, Singapore 117543, Singapore

AB: Headspace solid-phase microextraction (SPME), as a simple, solvent-free method, has been applied to the analysis of 10 chlorinated benzenes (CBs) present at trace levels in water samples. An SPME fibre coated with 100 µm thick poly(dimethylsiloxane) was used for extraction. The analytical data exhibited a relative standard deviation (RSD) range of 1.19% (for pentachlorobenzene) to 8.19% (for hexachlorobenzene) for the 10 CBs; the RSD of most compounds was under 6%. The sensitivity of the method was enhanced with agitation and with addition of salt to the sample solutions. With mass spectrometric detection, the limit of detection was below 0.006 µg/l for all 10 CBs after a 30 min

sampling time. The linearity range was 0.02-20 µg/l for the compounds studied. Water samples collected from a reservoir, and from the tap in a laboratory were analysed using the optimised conditions.

(28) TI: Determination of methyl tert-butyl ether in surface water by use of solid-phase microextraction.

AU: Achten\_C, Puttmann\_W

JN: Environmental Science and Technology, 2000, Vol.34, No.7, pp.1359-1364

IS: 0013-936X

DT: Article

NA: eichler@kristall.uni-frankfurt.de, Inst. Mineralogie-Umweltanal., J. W. Goethe Univ., 60054 Frankfurt am Main, Germany

AB: A combination of SPME and GC-MS was applied to determine low concentrations of methyl t-butyl ether (MTBE) in surface waters. The best results were obtained using a cooled 75 µm poly(dimethylsiloxane)/Carboxen fibre at 5degC, a sample temperature of 18-19degC, a NaCl concentration of 25% and an extraction time of 60 min for a sample volume of 1.5 ml. The adsorbed compounds were thermally desorbed at 240degC and determined on a column (50 m) coated with SE-54 (5 µm) and operated with temperature programming from 35degC (held for 1min) to 160degC at 10degC/min and then to 240degC (held for 40min) at 50degC/min, He as carrier gas and MS detection. The detection limit was 10 ng/l MTBE and RSD (n = 10) was 12%. The method was applied to river water and rain samples and MTBE concentrations were 7-160 and 9-70 ng/l, respectively.

(29) TI: Quantitative analysis of acetates in cigarette tobacco using solid-phase microextraction and gas chromatography-mass spectrometry.

AU: Watson\_CH, Ashley\_DL

JN: Journal of Chromatographic Science, 2000, Vol.38, No.4, pp.137-144

IS: 0021-9665

DT: Article

NA: Air Toxicants Branch, Centers Disease Control and Prevention, Atlanta, GA 30341-3724, USA

AB: A method incorporating SPME and GC-MS for the headspace analysis of selected volatile organic compounds present in cigarette tobacco is developed and evaluated. Quantitative information on methyl, ethyl, n-propyl, isopropyl, isopropenyl, vinyl and butyl acetates present in 29 different flavour variants (full, light, and ultra-light) of the top ten selling brands in the United States is presented. The concentrations of the various acetate analytes range from the low nanogram to microgram levels per cigarette. Clear differences are observed in the concentrations of various acetates when comparing the levels in brands from different manufacturers. The SPME technique provides a method that allows high sample throughput, requires little sample preparation and yields useful analytical information. High precision is obtained on multiple measurements of cigarettes from an individual pack, but lower precision levels are observed in general when comparing results obtained on the analysis of cigarettes from different packs of the same brand. The higher pack-to-pack variations may be due in part to product aging with a proportionate amount of evaporative loss of the relatively volatile acetates.

(30) TI: The detection and analysis of ignitable liquid residues extracted from human skin using SPME/GC.

AU: Almirall\_JR, Wang\_J, Lothridge\_K, Furton\_KG

JN: Journal of Forensic Sciences, 2000, Vol.45, No.2, pp.453-461

IS: 0022-1198

DT: Article

NA: Int. Forensic Res. Inst. and Dept. Chem., Florida Int. Univ., Miami FL, USA

AB: The detection of flammable or combustible liquid residues directly from human skin is described; the analytes include charcoal lighter fuel, diesel fuel, and gasoline. Analytes (10 µl) were deposited on the hands of a volunteer, and extracted at various time intervals; the hand was covered with a nylon bag (17 or 45 \* 30 cm) into which the polydimethylsiloxane-coated (0.1 mm) SPME fibre was inserted and exposed to the headspace for 15 min. The extracted sample was analysed on a column (30 m \* 0.25 mm i.d.) coated with DB-5 ms (0.25 µm) and operated with temperature programming [35degC (held for 1 min) to 70degC (held for 5 min) at 15degC/min, to 195degC (held for 6.5 min) at 4degC/min, then to 270degC (held for 3 min) at 20degC/min], He as carrier gas (1 ml/min) and FID. The effect of residue quantity and sampling were investigated. Gasoline, lighter fuel, and diesel were detected and characterized within 15, 15 and 45 min, respectively. Best recoveries (not stated) were obtained using the smaller bag and by heating the headspace with a lamp for 5 min before SPME sampling. Liquid deposits on skin were recovered up to 3.5 h after exposure, depending on residue type.

(31) TI: Simple and simultaneous analysis of fenfluramine, amphetamine and methamphetamine in whole blood by gas chromatography-mass spectrometry after headspace-solid phase microextraction and derivatization.

AU: Namera\_A, Yashiki\_M, Liu\_JT, Okajima\_K, Hara\_K, Imamura\_T, Kojima\_T

JN: Forensic Science International, 2000, Vol.109, No.3, pp.215-223

IS: 0379-0738

DT: Article

NA: namera@ipc.hiroshima-u.ac.jp, Dept. Legal Med., Hiroshima Univ. School Med., Hiroshima 734-8551, Japan

AB: Blood was mixed with d5-methamphetamine (internal standard) and 1M-sodium hydroxide and the mixture was sealed in a vial. The headspace was sampled for 15 min using a SPME fibre and the analytes were desorbed in the injection port of a GC-MS instrument containing heptafluorobutyric anhydride as derivatizing agent. The analytes were determined on a fused-silica column (30m\*0.25 mm i.d.) coated with Supelco PTE-5 (0.25 µm) and operated with temperature programming from 60-280degC, He as carrier gas (60 kPa) and 70 eV EI MS detection.

The calibration graphs were linear from 0.01-1 µg/g for fenfluramine (I), amphetamine (II) and methamphetamine (III). The detection limits were 5 ng/g for I and III, and 10 ng/g for II. Intra-day RSD (n = 5) were 4.8%, 1% and 4.4% at the 0.05 µg/g level, and 4.6%, 1.4% and 1.5% at the 0.5 µg/g level for I, II and III, respectively. Inter-day RSD (n = 5) were 9.2%, 8.2% and 8.2% at the 0.05 µg/g level, and 5.7%, 2.7% and 1.6% at the 0.5 µg/g level, for I, II and III, respectively.

(32) TI: Frozen storage effects on anthocyanins and volatile compounds of raspberry fruit.

AU: deAncos\_B, Ibanez\_E, Reglero\_G, Cano\_MP

JN: Journal of Agricultural and Food Chemistry, 2000, Vol.48, No.3, pp.873-879

IS: 0021-8561

DT: Article

NA: pcano@if.csic.es, Dept. Plant Foods Sci. and Technol., Inst. Frio, CSIC, 28040 Madrid, Spain

AB: Anthocyanins in frozen raspberries were extracted in 1% HCl in methanol, concentrated then adsorbed onto a C18 Sep-Pak cartridge and the anthocyanins eluted with 0.01% HCl in methanol. After drying the residue was dissolved in 4% phosphoric acid and analysed on a 5 µm ODS-Hypersil column (25cm\*4mm i.d.), with 4% phosphoric acid and acetonitrile as mobile phase (1 ml/min) with gradient elution and detection at 520 nm. Volatiles in the same sample were extracted using headspace SPME (on 100 µm dimethylpolysiloxane-coated silica fibres) for 30 min at 30degC with samples thermally desorbed then injected onto a fused-silica column (50 m \* 0.25 mm i.d.) coated with CP-Sil-5 CB (0.25 µm), He as carrier gas and operated with temperature programming from 50degC (held for 3 min) to 250degC (held for 17 min) at 5degC/min and 70 eV EIMS.

The effects of freezing on anthocyanins and volatiles are tabulated and discussed.

(33) TI: Kinetics of solid-phase extraction and solid-phase microextraction in thin adsorbent layer with saturation sorption isotherm.

AU: Semenov\_SN, Koziel\_JA, Pawliszyn\_J

JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.39-51

IS: 0021-9673

DT: Article

NA: sem@fly.trinititroitsk.ru, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999

AB: The effects of sorbent saturation in thin adsorbent layers have been much overlooked in earlier research and should be taken into account in both the theory and practice of solid-phase extraction (SPE) and solid-phase microextraction (SPME). The adsorption kinetics of a single analyte into a thin adsorptive layer was modeled for several cases of agitation conditions in the analyzed volume. The extraction process in the adsorbent layer was modeled using a Langmuir isotherm approximated by the linear isotherm at low concentrations and by a saturation plateau at concentrations exceeding the critical saturation concentration. Laplace transformations were used to estimate the equilibration time and adsorbed analyte concentration profile for no agitation, practical and perfect agitation in the analyzed volume. The equilibration time may be significantly reduced at high degrees of oversaturation and/or agitation in the analyzed volume. The resulting models indicated that the adsorbent layer becomes saturated at some critical value of the oversaturation degree parameter. The critical value of the oversaturation parameter is affected by both the concentration of the analyte in the analyzed volume and the sorbent characteristics. It was also shown that the adsorption process is carried out via the propagation of the saturation adsorption boundary toward the inner boundary of the adsorbent layer. These new adsorption models should serve as "stepping stones" for the development of competitive adsorption kinetic models for both SPE and SPME,

particularly in cases where fast sampling is used. AN: alkanes, n-, study of kinetics of adsorption of, by SPE and SPME, mathematical models for CN: extraction, solid-phase (SPE), in study of kinetics of adsorption of n-alkanes, mathematical models for; extraction, micro-, solid-phase (SPME), in study of kinetics of adsorption of n-alkanes, mathematical models for; mathematical models, Laplace transformations, for studying the kinetics of adsorption of n-alkanes by SPE and SPME

(34) TI: Use of solid-phase microextraction in the investigation of chemical communication in social wasps.

AU: Sledge\_MF, Moneti\_G, Pieraccini\_G, Turillazzi\_S

JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.73-77

IS: 0021-9673

DT: Article

NA: msledge@dbag.unifi.it, Dipto. Biol. Animale Genetica, Univ. Firenze, 50125 Firenze, Italy

CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999

AB: Solid-phase microextraction has been used to investigate chemical communication in several social wasp species. Using the technique to analyse exocrine gland secretions, we demonstrate that the results are comparable with those obtained with the more classical methods that use solvents, eliminating, in many cases, the shortcomings of these methods in insect pheromone analysis. As a result of its simplicity this technique is very suitable for research on the chemical ecology of social wasps and on insect communication in general.

AN: hydrocarbons, identn. of cuticular, in Parischnogaster sp. and Polistes dominulus gland secretions, by SPME-GC-MS tricosane (638-67-5) tricos-7-ene (52078-57-6) tricos-9-ene (52078-48-5) heptacosane (593-49-7) heptacosane, 5-methyl- (64821-84-7) heptacosane, 9,13-dimethyl-nonacosane(630-03-5)nonacos-1-ene(77046-61-8) hentriacontane(630-04-6) nonacosane,5-methyl-(71868-29-6) nonacosane,7-methyl-(76535-33-6)octacosane (630-02-4) octacosane, 2-methyl- (1560-98-1) tritriacontane, 13,17-dimethyl- (56987-79-2) docotriacontane

(35) TI: Speciation of dimethylarsinic acid and monomethylarsonic acid by solid-phase microextraction-gas chromatography-ion trap mass spectrometry.

AU: Mester\_Z, Pawliszyn\_J

JN: Journal of Chromatography, A, 2000, Vol.873, No.1, pp.129-135

IS: 0021-9673

DT: Article

NA: janusz@uwaterloo.ca, Dept. Chem., Univ. Waterloo, Waterloo, ON N2L 3G1, Canada

CO: Presented at ExTech 1999 Symposium: Advances in Extraction Technologies, held in Waterloo, ON, Canada, 26-30 Apr 1999

AB: A solid-phase microextraction (SPME) method has been developed to determine two methylated arsenic species in human urine samples by GC-MS. The direct extraction of the methyl arsenic compounds by SPME after thioglycol methylate derivatization was studied. Direct extraction with SPME was suitable for the determination of trace levels of dimethylarsinic acid (DMA) and monomethylarsonic acid (MMA) in urine samples. Four different commercial SPME fibers were tested for the extraction of methyl arsenic

compounds, and the best results were obtained using the polydimethylsiloxane coating. The extraction and desorption time profiles of DMA and MMA were determined. The detection limits for DMA and MMA using the SPME-GC-MS method were 0.12 and 0.29 ng/ml, respectively. The method is linear in the 1 to 200 ng/ml range.

(36) TI: Solid-phase microextraction in the determination of methadone in human saliva by gas chromatography-mass spectrometry.

AU: dosSantosLucas\_AC, Bermejo\_A, Fernandez\_P, Tabernerero\_MJ

JN: Journal of Analytical Toxicology, 2000, Vol.24, No.2, pp.93-96

IS: 0146-4760

DT: Article

NA: Dept. Clinical Anal. and Toxicol., Fac. Pharm., Univ. Amazonas, Amazonas, Brazil

AB: Solid-phase microextraction (SPME) with a 100 µm polydimethylsiloxane film fibre was applied to the determination of methadone and 2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine (EDDP) by GC-MS in human saliva and compared with liquid-liquid extraction. A shorter extraction time of 30 min with the fibre was obtained, speeding up the total analysis time. Linearity was found for SPME from 0.05 to 2.0 µg/ml ( $r = 0.9976$  for methadone;  $r = 0.9988$  for EDDP) with precision between 0.7 and 4.3% for saliva spiked with 0.2 and 1.5 µg/ml of methadone and EDDP. The limit of detection using SPME was 0.04 µg/ml for methadone and 0.008 µg/ml for EDDP. Analytical recoveries of SPME and liquid-liquid extraction ranged from 98.8 to 103.6%. The use of deuterated internal standard by both methods have yielded comparable results. Thus, the SPME method is highly accurate, precise, and useful for determination of methadone and EDDP in saliva.