36th International Symposium on Capillary Chromatography

Školení účastníků projektu
„Inovace a rozvoj výuky ekoinovací v bakalářských oborech akreditovaných na FTOP VŠCHT Praha“

Jméno školícího: prof. Ing. Václav Janda, CSc.

Datum školení: 14.6.2012

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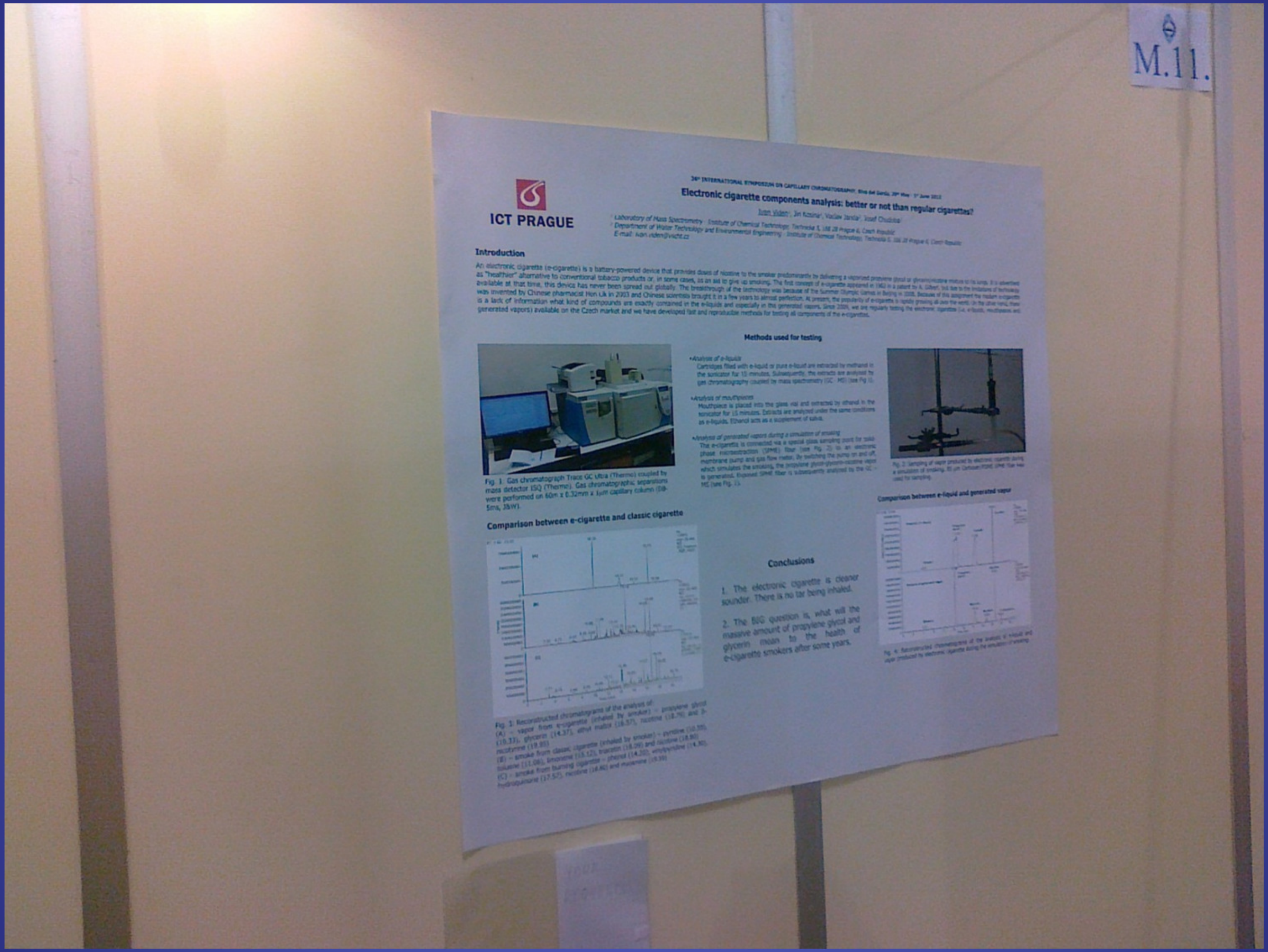
EVROPSKÁ UNIE
Evropský sociální fond
Praha & EU: Investujeme do vaší budoucnosti
VŠCHT PRAHA

Navštívená konference: 36th International Symposium on Capillary Chromatography

- Termín: 27.5.-1.6.2012
- Místo konání: Riva del Garda, Itálie
- Organizátor: Chromaleont S.r.L., prof. Luigi Mondello, spin-off organizace University of Messina
- Historie: již 36. symposium věnované tématu separačních analytických metod; pravděpodobně největší ve světě

Odborné zaměření akce

- Počet účastníků: 350
- Akce je věnována analytickým chemikům; všem, kteří se zabývají vývojem a aplikací separačních metod, jako je chromatografie a elektromigrační metody, včetně předúpravy vzorků a detekčních systémů.



ICT PRAGUE

Electronic cigarette components analysis: better or not than regular cigarettes?

Introduction

Methods used for testing

Comparison between e-cigarette and classic cigarette

Comparison between e-liquid and generated vapor

Conclusions

1. The electronic cigarette is cleaner sounder. There is no tar being inhaled.

2. The BIG question is, what will the massive amount of propylene glycol and glycerin mean to the health of e-cigarette smokers after some years.

Tematické zaměření akce

Názvy sekcí:
A. Fundamentals
B. Column Technology
C. Sample Preparation
D. Sampling Systems
E. Capillary GC
F. (Micro) Liquid Chromatography
G. Supercritical Fluid Chromatography and Extraction
H. Electromigration Methods
I. Instrumentation and Automation
J. Coupled and Multidimensional Techniques
K. Comprehensive Techniques – Gas Phase Separations
K*. Comprehensive Techniques – Liquid Phase Separations
L. Trace Analysis
M. Environmental Applications
N. Energy, Petrochemical and Industrial Applications
O. Biomedical and Pharmaceutical Applications
P. Analysis of Natural Products, Food, Flavours and Fragrances
Q. Microfabricated Chips

36th International Symposium on Capillary Chromatography

- Z hlediska cílů návštěvy konference byly pro nás prioritní sekce C, L a M, částečně i sekce N
- K novinkám pořád ještě patří 2D GC a vícedimenzionální GC
- Co je z toho významné pro FTOP
  - Instrumentace dostupná na FTOP pro environmentální analýzu není úplně nejmodernější, ale na druhou stranu není úplně zastaralá
  - Environmentální analýza je odrazovým můstkem pro všechny ekologicky zaměřené předměty
  - Potvrdila se správnost myšlenky zavést nový obor Analytická chemie životního prostředí (akreditace proběhla 2010)

C 02 AUTOMATED SAMPLE PREPARATION USING A DIGITAL SYRINGE WITH EMBEDDED SPE CAPABILITY

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Popular sample preparation techniques such as solid phase extraction (SPE) , can be time consuming and expensive whether implemented on automated platforms or performed manually. Furthermore, method development to improve the efficiency of the sample preparation process is often difficult and time prohibitive, with long evaporation steps throughout the process. A key objective for all laboratories is to implement efficient sample preparation where there is a real need to improve productivity and minimize waste. While the introduction of automated and semi-automated platforms into the laboratory for sample preparation enables improved efficiencies, they generally come at a significant cost. We have combined the advantages of automation and SPE using a digital syringe with an embedded SPE cartridge which not only improves efficiencies but virtually eliminates solvent use and waste. The miniaturized format is ideal for small valuable samples such as biological extracellular fluids.
We demonstrate through a number of applications the advantages in combing the automation of a hand held, digital syringe with miniaturized SPE sorbent embedded in the needle of the syringe. Method development is rapid and inexpensive, enhancing laboratory workflow, while increasing the accuracy and reproducibility of the SPE process.

C04 VALIDATION AND OPTIMIZATION OF A LIQUID-PHASE MICROEXTRACTION METHOD BASED ON THE SOLIDIFICATION OF FLOATING ORGANIC MICRO DROP FOR TRACE ANALYSIS OF BTEX IN THE WATER SAMPLES

Hakim Faraji

Department of Chemistry - Varamin-Pishva Branch, Islamic Azad University, No 14, Mahdieh 5,3371786837 Varamin, Iran, Islamic Republic ofLiquid-phase microextraction (LPME) based on solidification of a floating organic micro dropfollowed by gas chromatography (flame ionization detection) to pre-concentrate and determinebenzene, toluene, ethylbenzene and xylenes (BTEX) in the aqueous samples has beeninvestigated. The effects of type and volume of extraction solvent, stirring rate, extraction time,temperature and ionic strength in LPME of BTEX were evaluated. The results confirm that theproposed procedure provides an improved accuracy, linear range, limit of detection, limit ofquantification and is very effective for analyzing the BTEX compounds in water samples. Underthe optimized conditions, pre-concentration factor of 149–285 and extraction efficiency of 31-61% were obtained. Repeatability (1.59-5.83%) and intermediate precision (2.05-7.14%)
were in acceptable range. The relative recovery obtained for each analyte in different water samples was higher than 82.3% at three fortification levels with the relative standard deviationof less than 7.5%.

C06 STATE-OF-THE-ART ON SORPTION-BASED METHODS

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In the last years, sorption-based methodologies have been extensively applied for trace analysisof many classes of priority compounds in several types of matrices. Solid phase extraction,solid phase micro-extraction and more recently, stir bar sorptive extraction (SBSE), are some good examples of the mostly used sample enrichment techniques prior to chromatographic or hyphenated analysis. SBSE in particular, operating in the static mode, have been extensively applied, since it is easy to manipulate and presents great advantages over other analytical approaches. Meanwhile, if we focused our attention on SBSE, although great performance has been reported, we still find many analytical limitations. Recently, novel analytical concepts have been proposed such as adsorptive microextraction (AμE) techniques, which represent a great alternative with a wide range of application. The present contribution, will discuss in detail, the state-of-the-art on sorption-based methods, including the novel concepts, configurations, sorbents and potential of application for trace analysis of priority compounds in real matrices.

References:

[1] A.F.P. Gonçalves et al. J. Chrom. Sci. (2012) In press
[2] N.R. Neng et al. Anal. Bioanal. Chem. 402 (2012) 1355
[3] N.R. Neng et al. J. Chromatogr. A 1218 (2011) 6263
[4] N.R. Neng et al. Talanta 83 (2011) 1643
[5] N.R. Neng et al. Anal. Bioanal. Chem. 398 (2010) 3155
[6] N.R. Neng et al. J. Chromatogr. A 1217 (2010) 7303

C10 DISPERSIVE LIQUID–LIQUID MICROEXTRACTION COMBINED WITH CAPILLARY GAS CHROMATOGRAPHY AS A VERY SIMPLE, RAPID AND SENSITIVE METHOD FOR THE DETERMINATION OF BISPHENOL A AND DIETHILSTILBESTROL IN WATER SAMPLES

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2 Department of Chemistry - Vladimir State University, ul. Gor'kogo, 87, 600000 Vladimir, Russia (Russian Fed.)

A new method, termed dispersive liquid–liquid microextraction (DLLME) combined with capillary gas chromatography with electron capture detection, was developed for the determination of bisphenol A (BPA) and diethylstilbestrol (DES) in water samples. Important factors such as the volume and type of extraction and disperser solvent, extraction time and terms of obtaining derivates were studied. An appropriate mixture of acetonitrile (disperser solvent) and tetrachloromethane (extraction solvent) was injected rapidly into a water sample containing BPA and DES. Dispersing of the extract was performed using ultrasound. Analytes were derivatized with trifluoroacetic anhydride catalyzing by triethylamine and determined by means of gas chromatography with electron capture detection. Under optimal conditions recovery is close to 100 %. Limits of detection (0,2 and 0,1 μg/L) were achieved for the determination of BPA and DES in water.

C14 HOLLOW-FIBER LIQUID-PHASE MICROEXTRACTION FOR THE DETERMINATION OF PESTICIDES AND SOME OF THEIR METABOLITES IN SOILS AND WATERS

María Asensio-Ramos, Javier Hernández-Borges, Miguel Ángel González-Curbelo, Bárbara Socas-Rodríguez, Miguel Ángel Rodríguez-Delgado Departamento de Química Analítica, Nutrición y Bromatología - Universidad de La Laguna, Avenida Astrofísico Francisco Sánchez, s/n, 38206 La Laguna, Tenerife (Canary Islands), Spain

Sample preparation constitutes a crucial step in the success of any analytical method, being the current trends focused on the miniaturization and simplification of the processes. In this sense in the last decade, liquid-phase microextraction (LPME) techniques have emerged as a really attractive alternative. They can be carried out in different modes, which can be classified in single-drop microextraction (SDME), dispersive liquid-liquid microextraction (DLLME) and hollow-fiber LPME (HF-LPME). In 1999, HF-LPME was introduced to avoid drop instability in SDME [1]. In it, analytes are extracted into a supported liquid membrane sustained in the pores of a hydrophobic HF and then into an acceptor solution that is placed inside the lumen of the fiber. When the process is carried out in the so-called two-phase mode, the solvent that impregnates the membrane is the same than the one located inside the fiber. In this work, a group of pesticides and some of their main metabolites (2-aminobenzimidazole, carbendazim/ benomyl, thiabendazole, fuberidazole, carbaryl, 1-naphthol and triazophos) were extracted from soils by ultrasound-assisted extraction (USE) and preconcentrated by the use of a two-phase HF-LPME procedure before injection, using 1-octanol as extraction solvent. Separation and quantification was achieved by high-performance liquid chromatography (HPLC) with fluorescence detection (FD). Calibration, precision and accuracy studies of the described USEHF-LPME-HPLC-FD method were evaluated with two soils of different physicochemical properties, reaching good results and LODs in the range 0.001-6.94 ng/g. With the aim of extending the validation, HF-LPME was directly applied to different types of waters (Milli-Q, mineral and run-off) obtaining LODs in the range 0.0002-0.57 µg/L.

Reference:

[1] S. Pedersen-Bjergaard et al. J. Chromatogr. A 1184 (2008) 132

C 16 AUTOMATED STANDARD AND SAMPLE PREPARATION USING THE AGILENT 7696A WORKBENCH FOR GC/MS ANALYSIS OF FAME CONTAMINATION IN JET FUEL

James Mccurry, Anthony Macherone
Gas Phase Division - Agilent Technologies, 2850 Centerville Road, 19808 Wilmington, United States

The Agilent 7696A Sample Prep WorkBench was used to prepare calibration standards and samples for the GC/MS analysis of total FAME in jet fuel using the IP585 method. The WorkBench needed ten times less reagents and standards to achieve better analysis results when compared to manual sample preparation techniques. The GC/MS calibration using WorkBench prepared standards meet all performance criteria without any re-work, saving considerable time in the laboratory. WorkBench prepared jet fuel samples exceeded the method’s precision requirements for several different levels of FAME contamination. The analysis results obtained from the WorkBench samples provided better recovery of the known FAME concentrations compared to the manually prepared samples.

C17 AUTOMATING SAMPLE PREPARATION FOR THE GC ANALYSIS OF BIODIESEL USING METHOD EN14105:2011

James McCurry, Anthony Macherone

Gas Phase Division - Agilent Technologies, 2850 Centerville Road, 19808 Wilmington, United States

The recently revised European Union method EN14105 describes complex, multistep procedures to manually prepare standards and samples for the GC analysis of glycerol contaminants in B100 biodiesel. The Agilent 7696A WorkBench was successfully used to automated the standard and sample prep of this method while reducing the reagent use and chemical wastes by a factor of ten. Calibration performance of the WorkBench prepared standards exceeded the method requirements. Using a commercial biodiesel sample, the WorkBench was shown to prepare the samples with an extremely high degree of precision that surpassed the method's specifications.

LIQUID-LIQUID EXTRACTION FOLLOWED BY FULL EVAPORATION DYNAMIC HEADSPACE GC-MS (LLE-FEDHS-GC-MS) FOR ANALYSIS OF ODOR COMPOUNDS IN AQUEOUS SAMPLES

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Since certain odor compounds in aqueous samples are present at trace level, analytical methods should include powerful extraction and enrichment steps before GC analysis. However, traditional approaches such as liquid-liquid extraction (LLE) or steam distillation requires large sample amount and tedious sample preparation process. Miniaturized methods such as solid phase microextraction (SPME) and stir bar sorptive extraction (SBSE), which allow extraction and concentration in a single step, provide enhanced sensitivity even with small sample amount because the whole extracted fraction can be introduced into GC by thermal desorption. However, these techniques in both immersion and headspace mode are generally biased toward recovering more hydrophobic compounds in aqueous samples. Recently, we developed a novel headspace technique called "Full evaporation dynamic headspace (FEDHS)" [1] based on full evaporation technique (FET) [2]. FEDHS allows complete vaporization of 100 µL of an aqueous sample, and trapping and drying it in an adsorbent packed tube, while recovering a wide range of odor compounds, and leaving most of the low volatile matrix behind. FEDHS also performs complete vaporization of more than 1 mL of organic solvent extract. In this study, FEDHS-GC-MS was performed for the miniaturized LLE extract, which is corresponding to the original aqueous sample volume of 1 mL. The feasibility and benefits of the proposed method is demonstrated with analyses of key odor compounds, which have hydrophilic and/or semi-volatile characteristic in beverages.

References:

[1] N. Ochiai *et al.* submitted for publication.
[2] M. Markelov *et al.* Anal. Chim. Acta 276 (1993) 235

C 27 EQUIPMENT FOR THE COMBUSTION AND ANALYTICAL DETERMINATION OF HALIDES (CL, F, BR AND I) IN SAMPLES OF FUEL AND COMBUSTIBLE WASTE MATERIALS

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This work is focused to the determination of halides (fluorine, chlorine, bromine and iodine) in the different types of solid fuels. Occurrence of halides in fuels causes formation of halides with the corrosive properties to the inner parts of boiler under the conditions of the combustion process. During the combustion process, especially within the imperfect burning, reactions of the halides with aromatic hydrocarbons should occur with the result of polyhalogenated aromatic compounds, dangerous for humans and generally for the environment. Due to the increasing production and utilisation of solid alternative fuels based on the biomass or waste material, requirements to the analysis for the individual halides content grows. Experimental part of this work describes development of the new equipment for the determination of halides in fuels. Equipment consists from the furnace with the heating possibility up to 1200°C, with two parallel quartz tubes. These tubes lead to the drying container and three consecutive sorption impingers. The method is based on the combustion of the sample under the oxidizing atmosphere in the quartz tube under the defined conditions (oxygen/argon rate, temperature, gas flow, time schedule) with the subsequent absorption of the gaseous products to the absorption solution, based on the hydrogen peroxide and sodium carbonate/sodium bicarbonate solution. This solution is consequently analysed by the ion chromatography method. Testing of the equipment, defining and optimization of conditions of this method for the analytical laboratory practice is a part of this work.

LIQUID-LIQUID EXTRACTION FOLLOWED BY FULL EVAPORATION DYNAMIC HEADSPACE GC-MS (LLE-FEDHS-GC-MS) FOR ANALYSIS OF ODOR COMPOUNDS IN AQUEOUS SAMPLES

Jun Tsunokawa1, Nobuo Ochiai1, Kikuo Sasamoto1, Andreas Hoffmann2
1 GERSTEL K.K., 1-3-1, Nakane, Meguro-ku,, 152-0031 Tokyo, Japan
2 GERSTEL GmbH & Co. KG, Eberhard-Gerstel-Platz 1, 45473 Mülheim an der Ruhr, Germany

Since certain odor compounds in aqueous samples are present at trace level, analytical methods should include powerful extraction and enrichment steps before GC analysis. However, traditional approaches such as liquid-liquid extraction (LLE) or steam distillation requires large sample amount and tedious sample preparation process. Miniaturized methods such as solid phase microextraction (SPME) and stir bar sorptive extraction (SBSE), which allow extraction and concentration in a single step, provide enhanced sensitivity even with small sample amount because the whole extracted fraction can be introduced into GC by thermal desorption. However, these techniques in both immersion and headspace mode are generally biased toward recovering more hydrophobic compounds in aqueous samples. Recently, we developed a novel headspace technique called “Full evaporation dynamic headspace (FEDHS)” [1] based on full evaporation technique (FET) [2]. FEDHS allows complete vaporization of 100 µL of an aqueous sample, and trapping and drying it in an adsorbent packed tube, while recovering a wide range of odor compounds, and leaving most of the low volatile matrix behind. FEDHS also performs complete vaporization of more than 1 mL of organic solvent extract. In this study, FEDHS-GC-MS was performed for the miniaturized LLE extract, which is corresponding to the original aqueous sample volume of 1 mL. The feasibility and benefits of the proposed method is demonstrated with analyses of key odor compounds, which have hydrophilic and/or semi-volatile characteristic in beverages.

References:

[1] N. Ochiai *et al.* submitted for publication.
[2] M. Markelov *et al.* Anal. Chim. Acta 276 (1993) 235

C30 SAMPLE PREPARATION AND INJECTION TECHNIQUES IN COMBINATION WITH GCMS

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Nowadays, a wide variety of sample preparation techniques are available for the analyst to select from in order to analyze volatile organic compounds (VOC) or semi-volatile organic compounds (SVOC) in all matrices. Based on the specific research question and available techniques, a selection has to be made on the most suitable manner in which to analyze the samples. In this work, a selection of non-invasive sample preparation techniques in combination with gas chromatography coupled with mass spectrometry (GCMS) were evaluated. The sample preparation techniques evaluated were headspace (HS), headspace solid phase dynamic extraction (HS-SPDE), headspace solid phase micro extraction (HS-SPME), liquid solid phase micro extraction (L-SPME), stir bar sorptive extraction (SBSE) and purge and trap (PT). In addition, the influence of cryogene refocussing in the beginning of the column was evaluated .
The samples which were used in this research consisted of aqueous references mixtures of different volatile and semi-volatile compounds, as well as real food matrices samples (e.g wine).
When selecting the most suitable technique, criteria such as such as robustness, sensitivity, speed and high-throughput possibilities have to be taken into account. All of the aforementioned methods presented advantages and disadvantages. For instance, PT (offline) was not suitable for high amount of samples, contrary to the other techniques employed in this study.
On the other hand, each technique showed sensitivity for a different range of volatile or semivolatile compounds. SPME (liquid or headspace) appeared to cover the broadest range of volatile compounds, especially when combined with cryo refocusing of the compounds during desorption.

C39 APPLICATION OF A NEEDLE TRAP DEVICE IN SAMPLING AND DETERMINATION OF CHLORINATED VOLATILE COMPOUNDS

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Needle trap device (NTD) with immobilized sorbent inside the syringe, act as an extraction technique which fulfills theserequirements and combines sample preparation and injection into one step. In order to apply NTD coupled with gas chromatography – mass spectrometry in sampling and determination of chlorinated volatile compounds from gaseous samples, a selection of the appropriate sorbents inside the needle trap device, optimization of desorption and sampling conditions as well as validation study for the selected NTD was carried out. Five different sorbents in multibed combinations were selected to compare their extraction efficiency towards dichloromethane, trichloromethane and tetrachloromethane in order to choose the appropriate one. The best results for these compounds were obtained with application of Tenax TA/Carbopack X/ Carboxen1000 NTD needle, both concerning peak areas and RSD values of 1.29 - 3.96 %. For the selected NTD, the best extraction and desorption conditions were optimized. For extraction process, needle was inserted into the gas sampling bag containing the standard sample. Extraction from the Tedlar bag filled with the appropriate concentration of compound was performed with manual pump (with 10 mL volume). Desorption took place in standard S/SL injector and thermal desorption was chosen as the best way of desorption in temperature of 320°C for 1 minute. The selected needle trap device is suitable for analysis of chlorinated compounds in air samples due to the linearity in selected mass range [ng] and good precision results determined as relative standard deviation equal to 5.05 and 3.03 and 6.52 %. Application of needle trap filled with Tenax TA, Carbopack X and Carboxen 1000 resulted in detection limits of few pg values for these three compounds. Reusability of up to 120 times makes it competitive to SPME fibers, which mostly can be used 50 -100 times. Therefore NTD can equally compete with other solventless extraction methods, such as e.g. SPME, in isolation and preconcentration of dichloromethane, trichloromethane and tetrachloromethane from air samples.

C35 COMPARISON OF THE SELECTIVITY OF DIFFERENT SORBENT PHASES IN BAR ADSORPTIVE EXTRACTION (BAμE) FOR THE DETERMINATION OF FUNGICIDES IN REAL MATRICES

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The selectivity of two activated carbons and three polymeric-based phases were tested in bar adsorptive micro-extraction (BAμE) [1] followed by large volume injection-gas chromatographymass spectrometry (LVI-GC-MS), for the determination of ten fungicides (azoxystrobin, difenoconazole, metalaxyl-M, myclobutanil, penconazole, tebuconazole, flusilazole, cyprodinil, procymidone and benalaxyl) used as model compounds in real matrices. Assays performed on 25 mL laboratory water samples spiked at the 0.8 μg/L level, using modified pyrrolidone polymeric phase, yielded recoveries ranging from 100.0 to 107.8 %, under optimized experimental conditions (extraction: 4 h (1,000 rpm), pH 5.5; back-extraction: acetonitrile/ methanol (1:1), 15 min. under sonification). Excellent precision (< 11.5 %) and accuracy and were achieved using intraday and interday repeatability assays. The methodology revealed LODs ranging from 4.0 to 30.0 ng/L and excellent linearity between 0.04 and 1.60 μg/L with determination coefficients higher than 0.9980. Assays performed in real water and wine samples, using the standard addition methodology, showed good sensitivity and excellent correlation coefficients (*r*2 > 0.9956) for tap and ground water, as well as for red and white wine samples. The comparison with other sorption-based approaches is also addressed.

Reference:

[1] N.R. Neng *et al.* J. Chromatogr. A 1217 (2010) 7303

C32 COMBINATION OF DISPERSIVE LIQUID-LIQUID MICROEXTRACTION AND SINGLE DROP MICROEXTRACTION; A NEW AND EFFICIENT TECHNIQUE FOR PRECONCENTRATION OF ACIDIC PHARMACEUTICAL IN WATERS USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Hakimeh Assadi gheshlagh Sofla, Ali Sarafraz
Ferdowsi University of Mashhad, chemistry department, 9177946984 Mashhad, Iran, Islamic Republic of Iran

In the present work new combination of two liquid phase microextraction techniques involving dispersive liquid liquid microextraction and single drop microextraction as a new preconcentration technique was developed for the separation and determination of the acidic nonsteroidal anti-inflammatory(NSAIDs) pharmaceutical compounds namely naproxen, diclofenac and ibuprofen in water samples using high performance liquid chromatography UV- detection. The extraction conditions were optimized and under the optimal conditions, the method showed good linearity range of 0.1-1000 µg.L-1, acceptable reproducibilities, low limits of detection and satisfactory relative recoveries. The developed method was applied for the determination of anti-inflammatory drugs in river and waste water samples.

L01 VOC ANALYSIS IN WATER AT PPQ LEVEL

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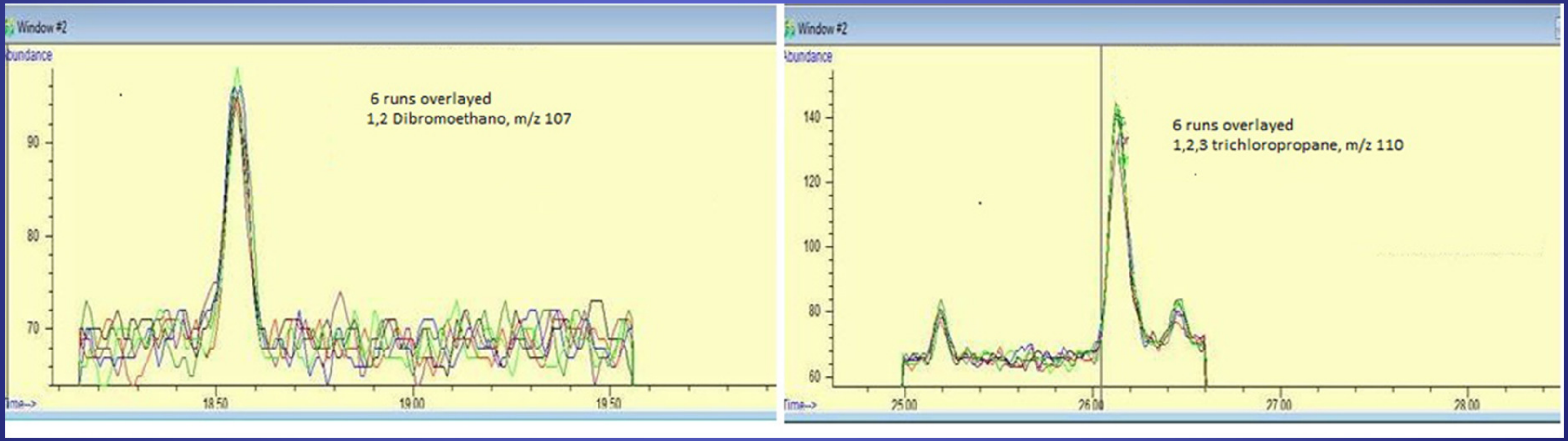
Nowadays, the closed purge and trap technique, coupled with GC-MS, is the unique choice when low ppt level water analysis is required. A very sensitive system has been developed (LOD at 100 ppq), by means of cryofocusing PTV inlet. Both sensitivity and reliability were investigated.



6 runs overlayed
1,2 Dibromoethano, m/z 107

6 runs overlayed
1,2,3 trichloropropane, m/z 110

L02 A SENSITIVE SBSE-TDS-GC/MS-METHOD FOR THE SCREENING OF LEACHABLES FROM PLASTIC PACKAGING MATERIALS IN AQUEOUS SAMPLES

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The use of plastics as packaging material may lead to emission and migration of miscellaneous substances into the packed goods. The amount of migrating substances has to be harmless and they must not interact with the commodity and change its physical-chemical-biological function and effect. Therefore it is necessary to determine substances at trace levels to ensure the product quality and human health.For analyses at trace levels stir-bar sorptive extraction (SBSE) has become a frequently used pre-concentration step, as it is solventless and very sensitive. In combination with thermaldesorption gas chromatography hyphenated to mass spectrometry (TDS-GC/MS) it can be used for such screening tests as mentioned above. However, the high sensitivity of this system can cause blank troubles because of impurities in the used extraction- and dilution-solvents. Even commercially available high purity solvents show blank peaks caused from substances present in the solvent, which can coelute with analytes. Using deconvolution software, multidimensional chromatography and high resolution mass spectrometry one can get these blank peaks under control but in the worst case the impurity of the solvent is the same as the analyte in the sample, which disables drawing conclusions of the compound’s origin. Especially in migration studies for plastic materials particular attention has to be paid to above-mentioned problems as bottled solvents are closed with plastic caps and water purification systems’ pipes are made of plastic materials, too. This work demonstrates a careful experimental design of SBSE-TDS-GC/MS experiments as well as a sophisticated data evaluation in order to obtain reliable results that were successfully applied to migration tests for plastic materials in aqueous simulants where several substances could be identified at ppt- to ppb-levels.

L03 ANALYSIS OF POLYBROMINATED DIPHENYL ETHERS (PBDES) IN COMPLEX MATRICES BY GAS CHROMATOGRAPHY WITH HIGH RESOLUTION-TIME-OFFLIGHT MASS SPECTROMETRY (GC-HRTOFMS)

Sjaak de Koning1, Joe Binkley2, Viatcheslav Artaev2, John Heim2, Mark Merrick2, Kevin Siek2, Dave Alonso2
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Polybrominated diphenyl ethers (PBDEs) are used as flame retardants in a vast number of household consumer products including furniture, upholstery, electrical equipment, electronic devices, and textiles. These compounds can enter the environment through manufacturing process emissions, off-gassing from various products, recycling wastes, and leaching from waste disposal sites. PBDEs have been detected in air, sediments, surface water, fish, and other marine animals. These compounds have been widely used since the 1970s and there is growing concern about their persistence in the environment and their tendency to bioaccumulate in the food chain. Various sample matrices, including sewage sludge, food products, and human breast milk were analyzed by gas chromatography High Resolution Time-of-Flight Mass Spectrometry (GC-HRTOFMS) using novel multi-reflecting TOF technology. This data highlights the advantages of GC-HRTOFMS for the analysis of PBDEs in extremely complex matrices. The ability of HRTOFMS to selectively extract masses at high resolution virtually eliminates background noise allowing both successful screening and trace level detection to be achieved.

L10 A GC/MS-MS vs. GC/HRMS DIOXINS ANALYSIS COMPARISON. SOME CRITICAL CONSIDERATIONS FOR LOW LEVEL, ENVIRONMENTAL SAMPLES

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Official methods for PCDDs and PCDFs (dioxins) analysis are based on high resolution mass spectrometers (HRMS). There are more and more evidences that is possible to perform screening of these pollutants by using an MS/MS approach, but, until now, the major limitation of this approach was due to lower absolute sensitivity of tandem instruments. In this field most of environmental samples show low dioxin contamination levels and sensitivity remains a key feature in this field. Environmental laboratories are requested to certify dioxins concentrations, with the Toxic Equivalent scheme (TEQ), where each dioxin congener concentration is corrected with a Toxic Equivalent Factor (TEF) and added together. Environmental samples often have congeners lower than the IDL, and, in the TEQ concept, appear the Upper , Middle and Lower bound TEQs values. To describe the worst case scenario, the Upper bound, the sum of individual TEQs, including TEQs associated with the LODs when compound is not detected, is used. As technology moves on, new instruments are offered on the market and new valuable possibilities are available to dioxins labs. In this work a critical comparison of the official GC/high resolution MS vs the GC/MS-MS approach is presented. Air and soil samples have been run both on a Thermo MAT 95 Dioxin HR and on a Agilent 7000 QQQ MS, using the same GC method and columns. Preliminary results show that sensitivity differences are still present having HRMS instruments an approximate 50 times lower LOD. Comparative results (middlebound results) show that samples > 0.1 pg TEQ/g differ from HRMS with an average of 20 %, with a 90° percentile of +/- 59% while samples >1 pg TEQ/g maintain the same differences, but the 90° percentile lowers to +/- 1.2%. For these samples the Italian legislation sets a limit of 10 and 100 pg TEQ/g for residential and industrial samples respectively. Data obtained with the MS/MS approach results in an increased specificity, allowing a much easier automation and more reproducibility, between analysts.

M02 DETERMINATION OF GLYPHOSATE AND AMINOMETHYLPHOSPHONIC ACID AND PRESENCE AND REMOVAL IN A DRINKING WATER TREATMENT PLANT

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Glyphosate is one of the most popular pesticides and is used as a broad-spectrum systemic herbicide for the removal of weeds and grasses. It is applied in large amounts over the world, e.g. in 2007 the EPA estimated that in the US almost 100,000 tons of the compound were used. Although it is not a very toxic compound to humans or terrestrial animals (LD50 in rats is 4.3 g/kg bw), it can be hazardous to aquatic life (see e.g. [1]). It has also been associated with malformations in fetuses and pregnant rats. Aminomethylphosphonic acid (AMPA) is the major metabolite, is rapidly formed in soil by enzymatic hydrolysis and is even less toxic. Formation in water, however, is quite slow (half-life > 35 days [2]). Due to the enormous use of glyphosate, it is encountered in surface waters in sometimes high concentrations. In the River Meuse up to 1.1 µg/L was measured in Keizersveer and also in the Albert Channel, from which Antwerpse Waterwerken is extracting water for the production of drinking water, high amounts have been determined. This can be a problem, since both compounds are hardly removed by active charcoal filtration. Although these compounds are less toxic than normal pesticides, the Flemish government states that concentration in drinking water should be not higher than 0.1 µg/L. This poses heavy demands on the analysis of these compounds. Since these compounds have small molecular weights, they are not readily analyzable by LC-MS/MS, but should first be derivatised, e.g. by fluorenylmethylchloroformate (FMOC). A method was established by Hanke et al [3], which was altered by us (derivatization, SPE-sample preparation and analysis by UPLC-LC-MS/MS). A sample campaign was organized to determine where removal took place. The altered method, the results of this campaign and concentrations in surface water will be presented.

References:

[1] J.P. Giesy *et al.* Reviews of Environmental Contamination and Toxicology 167 (2000) 35
[2] W. Kollman *et al.* Interim report of the pesticide chemistry database. Environmental hazards Assessment Program. Department of Pesticide Regulation (1995)
[3] I. Hanke Anal. Bioanal. Chem. 391 (2008) 2265

M03 GC-MS METHOD FOR SIMULTANEOUS ANALYSIS OF DICARBOXYLIC ACIDS AND SUGARS IN ATMOSPHERIC AEROSOL: RESPONSE SURFACE METHODOLOGY FOR OPTIMIZING SOLVENT EXTRACTION.

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Water-soluble organic compounds (WSOCs) are an important group of chemical tracers that may give relevant information on the relative strengths of primary emission sources and secondary photochemical processes on air quality. In fact, they can be primarily emitted into the atmosphere by a multiplicity of sources -- including power plants, vehicular circulation, meat cooking operations and biomass burning -- or secondarily produced by photochemical atmosphere reactions from both biogenic and anthropogenic precursors. This paper describes the development of a GC-MS procedure for the simultaneous analysis of WSOCs in atmospheric aerosols. The extraction operating conditions must be properly selected to achieve the highest yield for several target analytes with a wide range of water solubility. The response surface methodology (RSM) including central composite design (CCD) was applied to optimize solvent extraction for a large number of dicarboxylic acids and sugars. The factors considered were the solvent type (characterized by solvent polarity parameter) and volume (10-20 ml). On the basis of RSM and experimental evidence, the optimum extraction solvent was a mixture of metane:dichlorometane (90:10) using a volume of 10 ml. The optimized procedure provides the low detection limits (lower than 2 ngm-3) and the good reproducibility (RSD lower than 13%) required by environmental monitoring. The developed protocol was extended to 22 target analytes that are relevant chemical markers, i.e., 15 carboxylic acids and 7 sugars. In addition, the suitability of the optimized procedure was verified by application to PM filters collected under different conditions, i.e., different seasons (summer vs. winter), different sampling sites (urban vs. rural), different particle size dimensions (PM2.5 vs. PM1).

DETERMINATION OF SELECTED ORGANIC CONTAMINANTS IN SOIL BY PRESSURIZED LIQUID EXTRACTION AND GAS CHROMATOGRAPHY TANDEM MASS SPECTROMETRY

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The determination of organic contaminants in soil is a real challenge due to the large number of these compounds with varying chemical and physical properties. This paper presents the development of an analytical method for the simultaneous determination in soil of 40 organic contaminants belonging to different chemical classes: polycyclic aromatic hydrocarbons, polychlorinated biphenyls, polybrominated diphenyl ethers, UV filters, parabens, bisphenols and triclosan. Soil was extracted by pressurized liquid extraction and the extracts, without the need of a clean-up step, were analyzed by gas chromatography-tandem mass spectrometry after in-situ derivatization, in the gas chromatographic system, using BSTFA containing 1% TMCS. The use of matrix-matched calibration was avoided using isotope labeled internal standards to compensate the matrix induced chromatographic response enhancement observed. In the PLE step, two extraction cycles were performed with an ethyl acetate-methanol (90:10, v/v) solution at 80 şC. For the recovery studies, samples were spiked to reach concentrations of 120 ng g-1, 60 ng g-1 or 30 ng g-1 and the recoveries achieved were satisfactory for most of the compounds. A good linearity was obtained in the range 10-200 ng ml-1 with correlation coefficients equal or higher than 0.990 for all the compounds studied. The developed method provided limits of detection from 0.1 to 2.5 ng g-1.
Once the method was optimized and validated it was applied to soil samples collected from 14 agricultural plots. PAHs were the compounds detected at higher concentrations and levels up to 230 ng g-1 of PAHs with high molecular weights were obtained from a soil sample collected at a plot bordering a highway with very heavy traffic. Parabens were also found but at relatively low concentration levels. The most frequently UV filters detected was 2-ethylhexyl salicylate, at level ranging from 17.2 to 43.4 ng g-1. Triclosan was found in eight out of fourteen samples, at relatively low concentration levels (0.8-28 ng g-1).

M 07 SENSITIVITY AND SELECTIVITY: ANALYSING PBDE IN HOUSE DUST SAMPLES WITH THE GC QUANTUM ULTRA IN EI- SRM MODE COMPARED TO NEGATIVE CHEMICAL IONIZATION GC-MS IN SIM

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Polybrominated diphenyl ethers are a class of compounds used as flame retardants in a wide variety of household appliances and furniture. Certain congeners have been completely banned and are currently on the Stockholm convention list for persistent organic pollutants. There is an ever growing concern about the health risks being exposed to these class of compounds with sources of human intake besides ingestion (nutritional) being inhalation of indoor air and house dust. Preparation of the house dust samples was performed using pressurized liquid extraction with an in cell clean up. In this application note, a comparison is made between two analytical techniques; GC-MS-MS and GC-NCI-SIM, evaluating detection limits, repeatability and selectivity improvements in matrix.

M10 A NEW APPROACH FOR THE QUANTIFICATION OF MULTIPLE STEROIDAL COMPOUNDS IN WASTEWATER BY COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY

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Recent studies have demonstrated that hormonally active contaminants are passing through wastewater treatment plants causing serious environmental risk to aquatic ecosystems. In this study, special emphasis was put on the applicability of comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry with advanced software for automated processing of the complex chromatograms and identification of steroidal compounds in wastewater samples. Because there is a lack of available commercial reference materials for most of these compounds, a chemometric model based on partial least squares regression was developed using six steroid standards to assure the reliable quantitation of all the identified compounds. Secondly, special attention was paid to efficient sample preparation of influent and effluent wastewaters including dynamic ultrasound assisted extraction and solid-phase extraction. Different solvents were employed for free and conjugated steroids to elute them from the sorbent into separate fractions, which were then further processed and derivatized for analysis. 40 steroidal compounds were found in influent water (1-700 ng/L) and five compounds in effluent water (1-70 ng/L). Although, effluent water contained only a small amount of solid matter (< 10 mg/L), high concentrations of steroids (100-5000 µg/kg) were found in these particles.

FAST GCMS ANALYSIS OF 60 VOC COMPOUNDS USING HEADSPACE-TRAP SAMPLING

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The analysis of regulated volatile organic compounds in drinking and waste water is usually done with headspace or purge and trap technique using a so called 624 stationary phase with 30 m, 0.25 mm, 1.4 mm according to the EPA method 624. Reducing analysis time (fast GC) but maintaining chromatographic resolution has been successfully applied using narrow bore columns in various fields. However, the results reported were mainly based on liquid injection techniques. In Headspace analysis the transfer of sample from the insert to the column is usually relatively slow as mainly influenced by the gas flow through the insert. Regarding VOC analysis normally small split ratios are used in favour of sensitivity. This leads to a broad initial spatial band of the compounds over the first part of the column and therefore lead to bad chromatographic resolution which is in contradiction to fast GC approaches using narrow bore columns. For the present VOC analysis a film thickness of 1 mm was selected. The minimum inner diameter for such a column with a 624 phase available is 0.18 mm with a lengths of 20 m. The water samples (5 ml) were placed into 20 ml headspace vials and the volume injected was 1ml (split ratio 5:1). A cold trap is mounted at the top of the column directly under the injector in order to cool the first part of the column resulting in refocussing volatile compounds. The trap was cooled by direct liquid nitrogen and surrounds the column so that refocusing takes place at the top of the column. Different cold trap temperatures were selected and the optimum in refocusing was observed at – 150°C by measuring the peak profile at the end of the column in the mass spectrometric detector. As carrier gas Helium and Hydrogen was used. The best separation was achieved with Hydrogen at an average constant linear carrier gas velocity of 90 cm/sec. All 60 VOC peaks are separated in about 8 minutes run time. The peak width regarding one of the most volatiles which is venyl chloride was improved using the trap by more than a factor 50. The Limits of detection in real world samples for benzene and vinyl chloride were 0.01 and 0.002 mg/l, respectively. Using Hydrogen the sensitivity was reduced relative to Helium by a factor of 2 for the first eluting compound up to 5 minutes retention time and about 4 for compounds eluting later.

DETERMINATION OF N-HERBICIDES IN NATURAL WATER USING HPLC AND HPLC/MS ANALYSIS

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By the plurality of the employed pesticides at the moment, the attention is directed to those substance classes which are measurable amounts in ground water and drinking water respectively. The present study deals with the analysis using high performance liquid chromatography-diode array detector (HPLC-DAD) of several s-triazines. In addition to the triazines an analytical method include the determination of phenylurea herbicides have been developped using HPLC/DAD. Paying attention to the influence of the UV light lambda>290 nm and understanding the degradation phenomena, the photodegradation of isoproturon herbicide in water was determined using solution irradiated with a laboratory light source. In this study the rate of the photodecomposition of the herbicide was measured using high performance liquid chromatography-diode array detector (HPLC-DAD). The photodegradation of this product exhibited pseudo-first-order reaction kinetics. Several photoproducts were identified by HPLC/MS/MS. The main photoproduct in water is identified as 4-isopropyl aniline.

M20 AUTOMATED AND HIGH-THROUGHPUT QUANTITATIVE ANALYSIS OF WATER AND SOIL PROFILES BY MULTI-FIBER EXCHANGE-SOLID PHASE MICROEXTRACTION COUPLED TO FAST GAS CHROMATOGRAPHY-ELECTRON IONIZATION/MASS SPECTROMETRY. VALIDATION AND PROFICIENCY TESTING

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The Italian Legislative Decree No 152/2006 implementing Directive 2001/42/EC sets out a series of analytical profiles for water and soil. In previous studies the use of solvents and/or clean-up steps are often reported to extract and eliminate most of the interfering compounds. Accordingly, we developed a procedures where the properties of Head Space (HS)/Direct Immersion (DI)-Solid Phase MicroExtraction (SPME) technique as well as the automation of the preparation procedure by new robotic system called MultiFiber Exchange (MFX), through change of the fibers, allowed a friendly use of Fast GC-MS/EI apparatus with a number of advantages including reduced analyst time and greater reproducibility. As, this increased versatility of MFX allows approaches in fully automated mode, the Authors are interested in: *polycyclic aromatic* hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), phenols [1] and mineral oil hydrocarbons (BAM K010) profiles . The object of this work is obtained all these profiles in fully automated and very short time by use of single GC/MS apparatus with consequently more sensitivity power and high discrimination, than other techniques for routine employed in environmental laboratories. The resulting calibration curves were linear, in the investigated range for all the considerated profiles, with correlation coefficients >0.998. The RSD resulted ≤10%. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance [2]. The automation of the preparation procedure with MFX, that allowed the change of the fiber by robotic device, allowed a friendly use of Fast-MS apparatus with a number of advantages including reduced analyst time both for routine analysis and method development, and greater reproducibility.

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[1] Llompart *et al.* J. Chromatogr. A 963 (2002) 137
[2] M. Thompson *et al.* Pure App.l Chem. 78 (2006) 145

M17 DYNAMIC PURGE & TRAP EXTRACTION OF VOLATILES IN DRINKING WATER AND HIGH SPEED TIME OF FLIGHT DETECTION IN COMPLIANCE TO EPA 524.3

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Analysis of Volatiles Organic Compounds (VOCs) in water is of increasing interest due to critical ground contamination from several sources as gasoline, oil spills and industrial solvents. Especially for drinking water, possible contamination is of great concern for potential human health effect. The U.S. Environmental Protection Agency (EPA) strictly regulates the assessment of drinking water quality trough methods 524.3 in which detection limits and instrumentation requirements are established. Official guidelines require the monitoring of VOCs contaminants in drinking water at progressively lower concentration level and typically the P&T extraction technique is indicated to reach the requested limit of detection. This work is presenting the use of the flexible DANI Dynamic Headspace Sampler (DHS) working in purging mode as viable alternative to classical P&T autosampler, highlighting the several benefits of this approach in terms of extended automation, overlapping incubation time capability and absence of cross-contamination between samples. The extracted VOCs are then analyzed by fast GC-TOFMS, reducing the analysis time down to twelve minutes with substantial gain in sample throughput. The full compliance of the DANI MasterTOFMS to the EPA ion ratio criteria has been validated and proven without the need of dedicated instrument tuning. Default autotuning procedures can be regularly performed before any new calibration sequence. Further advantage of the Time of Flight technology is the availability of the full mass range information and the deconvolution capability which allows to reach the expected sensitivity with proper identity confirmation through library matching. Besides, particular attention has been paid to the most volatiles compounds in terms of peak shape and recovery.

M21 AN OPTIMIZED METHOD FOR DETERMINATION OF ACROLEIN-DNPH IN EXHALED AIR BY GAS CHROMATOGRAPHY – MASS SPECTROMETRY

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Acrolein is an aldehyde, which is introduced to the environment from many sources such as automobile emission, cigarette smoke, heating cooking oil and industrial processes. In cigarette smoke the acrolein is formed from pyrolysis of organic matter. The present study sought to optimize a simple and sensitive method for determination of acrolein-DNPH in exhaled air of smokers and no smokers by gas chromatograph – mass spectrometry using SPME for active sampling. The extraction method of acrolein by SPME was carried out by immersion using polyacrylate (PA) fiber, with DNPH derivatization on-fiber. Some SPME derivatization parameters were studied by a Pareto chart analysis of the effects. It was used a Finnigan Trace GC/MS, equipped with Ion Trap system of Thermo Scientific Inc. (West Palm Beach, USA) and a capillary column 5%-Phenyl-methylpolysiloxane (HP-5 MS) of 30 m long, 0.25 mm i.d. and 0.25 µm film thickness from Agilent Tecnology Inc (Santa Clara, EUA). Using the optimized conditions, some analytical performance was studied to validate the method. The calibration curve was constructed in the range of 94.11 to 205.10 µg/m3 showing R2 = 0.993. The LOD and LOQ were 16.7 µg/m3 and 40.1 µg/m3 respectively. Five replicates at the concentration of 120.7 µg/m3 levels were analyzed on three consecutive days to assess the intermediate precision. The value of RSD obtained was of 10.1%. The application of this new method to real samples was achieved through the analysis of acrolein in exhaled air of smokers and no smokers. The range of values was 0.48 to 1.62 ppb to smoker and 0.35 to 1.43 ppb to no smoker. Both groups presented a large variability in acrolein levels of the breath, indicating that the exposed group was not homogeneous. However, the new method developed in this study allowed to determine the exposition of acrolein present in cigarette smoke.

M22 ROUTINE TRACE LEVEL SCREENING FOR A BROAD RANGE OF VOLATILE AND SEMI-VOLATILE ORGANIC POLLUTANTS IN A RANGE OF ENVIRONMENTAL WATERS USING FULL SCAN GC-MS WITH A 950 COMPOUND TARGET SCREENING DATABASE AND DECONVOLUTION SOFTWARE

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To address the ‘chemical monitoring challenge’ of the Water Framework Directive, the National Laboratory Service (NLS) has developed ‘trace level’ routine screening method for a broad range of volatile and semi-volatile pollutants. This comprehensive ‘one run’ method covers virtually all GC-amenable pesticides as well as hundreds of other organic pollutants which can be identified from a single sample with minimum preparation at ppt levels. Extracted samples are analysed using full scan GC-MS with mass spectral deconvolution and automated quality checking against libraries and retention time locked screening databases to ensure the highest data integrity and quality. This method forms the basis of the UK Environment Agency’s screening protocol for all water types and sample matrices analysed include fresh water, ground water, saline, sewage effluents, trade effluents, leachates and solids/sediments.

M29 INVESTIGATION OF QUECHERS APPROACH FOR THE ANALYSIS OF POLYBROMODIPHENYLETHER IN SEDIMENTS

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Polybromodiphenyl ethers (PBDE) are organobromine compounds that have been intensively used as flame retardants in products such as electronics goods. Due to their endocrine disruptor properties, they have been included in the list of priority substances as defined by the EU WFD. Their monitoring is therefore mandatory to assess their presence in the environment. The PBDE are hydrophobic component so are mostly adsorbed on sediments in aquatic environment. Extractions of PBDE in sediments are usually carried out using Soxhlet or pressurized fluid extraction. If they are efficient, these exhaustive techniques also extract many other interfering components present in the matrix, therefore intensive purification steps are required.
In the aim of reducing preparation sample time, this study focus on the investigation of an alternative technique for extracting PBDE from sediments matrices, and to the associated purification step. Analysis of PBDEs was performed by gas chromatography using programmable temperature injection and mass spectrometry with chemical negative ionization as detection mode. The standard method, ISO 22032, using extraction with pressurized fluid (PFE) and a multilayer column purification is compared with an alternative technique, based on a QuEchers type approach. Optimization for this extraction approach was performed by comparing ultrasonic, vortex or manual agitation. The purification step, using dispersive solid phase extraction, was also investigated with different amount and type of adsorbent tested (C18, alumina, silica, florisil). Several types of sediments including reference material were studied. Overall, with optimum conditions, extraction yield between 80 to 120% were obtained using the alternative technique for most of the various BDE congeners.

A NEW VALIDATED ANALYTICAL METHOD FOR THE DETERMINATION OF TRIBUTYLTIN IN WATER SAMPLES AT THE QUANTIFICATION LEVEL SET BY THE EUROPEAN UNION

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According to recent directives of the European Union (EU), limits of quantification (LOQ) for the determination of tributyltin (TBT) in surface waters should be ca. 60 pg/L (ppq). This put very stringent requirements on analytical methodologies; definitely when they have to be applied in a routine environment. Stir bar sorptive extraction (SBSE), followed by thermal desorption (TD) and capillary gas chromatography – triple quadrupole mass spectrometry (GC-MS/MS) can provide accurate and precise data at the 2 ng/L level (ppt). For lower concentrations, matrix and reagent interferences together with contamination may provide too high TBT values. A twodimensionalheart-cut GC method was developed to fractionate TBT from interferences. The GC-GC-MS/MS method shows excellent linearity in the range 50 pg/L to 4 ng/L, good repeatability (RSD < 20% at 200 pg/L), and a limit of detection of 11 pg/L. The method performance is demonstrated with two representative samples i.e. a harbor water and a waste water sample.

LE55 EVOLUTION OF THE NEEDLE TRAP TECHNOLOGY

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Needle trap addresses the need for a very simple, but efficient sampling/sample preparation device and procedures prior to separation and quantification steps in the analytical process. The main advantage of the proposed technique over other approaches is easy field and onsite operation with a minimum or no movement of instrumental parts during sampling, no additional extraction steps, straightforward quantitation, and efficient sample introduction to analytical instrumentation such as gas chromatography. The needle trap consists of stainlesssteel bevel tip or side hole needle with about 5 mm packed section near the needle tip. A sorbent is immobilized inside a needle resulting in the development of a needle trap (NT) device. The packing materials included glass wool, PDMS, DVB, Carboxen and Tenax. Construction of this simple and integrated sampling/extraction/sample introduction device was optimized by considering different ways to immobilize a sorbent in the needle, packing single and multiplelayer sorbent beds, and applying different desorption strategies into the GC injector. A carrier gas system is modified or a narrow neck liner was used to minimize the carryover for the needle trap. Breakthrough in the device can be investigated by connecting two NT devices in series. The needle trap performed very well as an exhaustive spot sampler, as well as in a time-weighted average (TWA) operation. The linear velocity of the mobile phase has no influence on the sampling rate of the needle trap. Validation results against the standard NIOSH 1501 method using charcoal tubes for indoor air surveys demonstrated good accuracy for the NT approach. The reproducibility of the NT is about 5% RSD and the detection limits for FID detection and for 25 ml gas sample are about 1 ng/L. The needle trap device has been applied for sampling and analysis of dissolved organics, aerosols and airborne particulate matter from an inhaleradministered drug, spray insect repellant, smokes and tailpipe diesel exhaust. The needle trap device can be used in microextraction, time-weighted average sampling and exhaustive extraction modes. More recently needle trap device has been adopted for autosampler application resulting in a system similar in operation to purge and trap, but substantially miniaturized.

Reference:

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LE57 MINIATURIZED SAMPLE PREPARATION DEVICE DESIGNED FOR ANALYSIS OF VARIOUS VOLATILE ORGANIC COMPOUNDS IN GAS CHROMATOGRAPHY

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Miniaturization of sample preparation techniques has become increasingly important in the research field of separation science because of the possibility of successfully meeting recent requirements, such as significantly enhanced sensitivity and selectivity, and more ecological and economical features. The effective hyphenation of miniaturized sample preparation to microscale separation methods has also been investigated. This approach enables the successful on-line coupling of these techniques without the disadvantages that are typically found in off-line multistep processes used for the analysis of complex mixtures such as environmental and biological samples. Down-sizing of the sample preparation process could make it possible to enhance mass sensitivity and reduce sample size, although the appropriate optimization of the hyphenated system should be considered. Recently, novel sample preparation techniques have been developed with a specially-designed needle extraction device, where a section of the extraction needle has a polymeric coating inside or the needle is packed with particulate materials to ensure the preconcentration of the target analytes. A wide variety of extraction media can be employed, and it is easy to handle during the extraction and desorption processes, which are additional attractive features for automation and for coupling to typical GC instruments on-line. Fiber-packed and particle-packed needle extraction devices have been introduced as the tool of sample preparation of various volatile organic compounds (VOCs), such as aldehydes, ethylene oxide, aliphatic and aromatic solvents and smoking-related VOCs. In this presentation, the development of these miniaturized sample preparation needle devices will be reviewed and several novel applications to real sample analysis, including the applications to human breath analysis and fire investigation, will be introduced.

Autorizace vydavatele sborníku abstraktů k využití sborníku ke vzdělávacím účelům:

From: Luigi Mondello [mailto:lmondello@unime.it]
Sent: Wednesday, June 06, 2012 12:36 PM
To: Janda Vaclav

Subject: Book of abstract
Dear Vaclav,
herewith I authorize you to use the book of abstract of the ISCC and GCxGC meeting for educational purpose.
Best regards
Luigi

Prof. Luigi Mondello

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Scientific Director of “Chromaleont” a spin-off of the University of Messina

Nové trendy výzkumu/vývoje v oboru, které na konferenci/školení zazněly

- Dvourozměrná GCxGC, ev. LCxGC, spojená s TOF detekcí
- Prekoncentrace a příprava vzorku
- Environmentální analýza
- Stopová analýza

Vystavená zařízení

- Symposium bylo spojeno s výstavou přístrojů světově renomovaných firem, jako je Agilent, Leco, Gerstel, Jeol, Thermo a další.

Leco Pegasus® HT TOFMS

Agilent 7200 Q-TOF for GC/MS

Gerstel MPS XL

Poznatky ze symposia se hodí zařadit jako ekoinovace do následujících předmětů fakulty

- Organické technologie – aplikace chromatografických metod pro průmyslovou analýzu, čistší produkce
- Základy zpracování a využití ropy - aplikace chromatografických metod pro průmyslovou analýzu, čistší produkce
- Analýza paliv – chromatografie: fundamentální analytická metoda, včetně ekologických aplikací
- Základy spalovacích procesů – monitoring, čistější emise
- Alternativní paliva v dopravě – korelace složení suroviny x čistější emise
- Alternativní zdroje energie I – korelace složení suroviny x čistější emise
- Alternativní zdroje energie II – korelace složení suroviny x čistější emise
- Úprava napájecích a užitkových vod – sledování kvality vody
- Seminář a laboratoř analytiky prostředí - chromatografie: fundamentální analytická metoda pro analýzu organických látek ve vodách
- Základy zpracování a využití uhlí a plynu - aplikace chromatografických metod pro průmyslovou analýzu, čistší produkce
- Výroba energie z biomasy – kvalita biomasy a odpadů
- Chemie ovzduší – kvalita ovzduší, emise a imise
- Kvalita ovzduší a její kontrola - kvalita ovzduší, emise a imise
- Základy toxikologie a ekologie - chromatografie: fundamentální analytická metoda
- Základy ochrany životního prostředí - chromatografie: fundamentální analytická metoda
- Úprava vody - sledování kvality vody
- Ochrana čistoty vod - sledování kvality vody
- Základy čištění odpadních vod - sledování kvality vody
- Analytika vody - chromatografie: fundamentální analytická metoda
- Hydrochemie - chromatografie: fundamentální analytická metoda

Celkové shrnutí symposia

- Profesně se jedná o největší chromatografickou konferenci na světě
- Co bylo nejzajímavější? Rozvoj GCxGC/TOF