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Sample Prep efficiently automated

Automated extraction and concentration techniques for comprehensive food packaging migration studies and ultra-trace environmental analysis

Providing the human species with an adequate supply of safe foods still means accepting some risk. Pesticides and other chemicals, for example, are applied to fight harmful insects, plants, fungi and various microorganisms. Year after year such chemicals safeguard our food supply. The drawback: Some of the chemicals contaminate our environment; air soil and water are afflicted. These chemicals may in the end wind up threatening the survival of a multitude of species on our planet.

If improperly handled or applied, the chemicals may also pose a danger to occupational health and safety for agricultural workers and may wind up poisoning the very foods they were designed to protect. Apart from improper application of legally accepted chemical agents, regulators and control labs frequently have cases in which banned substances and chemicals are used in agriculture and, for example, in fish farming.

Another example: Thanks to modern packaging, foodstuffs can be stored and transported under excellent conditions, i.e. in a germ-free environment. The risk of incurring a contamination with viruses, bacteria or fungi can be reduced to an unavoidable minimum with the excellent food packaging materials available today.

The flip side: Packaging materials are rarely inert. Packaged foods, for example, can be contaminated with various chemicals from the packaging material itself. Examples are residual monomers, solvents or plasticizers, among others. Residual solvents can even migrate into foods from printing ink used on labels.

It seems obvious: One hundred percent safety is not achievable. This is of course no reason to bury our heads in the sand – on the contrary. Chemical engineers can contribute by designing production processes properly and analytical chemists can help to ensure that frequent, timely and proper controls are implemented.

Frequent controls and analyses of products in the market are the only solution, especially as the globalized market place makes it ever more difficult to determine the origin of products, their content and the processes by which they were produced. Analyses are required both for consumer safety as well as to safeguard producers against claims brought by consumers.

Current instrumental analysis methods are well developed and matured, but in the field of sample preparation, there is still enormous scope for improvements in both performance and productivity. **Frank David, Ph.D.**, R&D Manager from the Research Institute for Chromatography (RIC) will provide an overview of sample preparation and analyte concentration techniques that can be used to provide efficient determination of food contaminants that enter foods by migration from packaging materials. **Nobuo Ochiai, Ph.D.** will present results from his work on improving productivity and performance in SBSE analysis of aqueous samples in the fields of environmental and food analysis. A wide analyte polarity range is covered and the SBSE technique coupled with GCxGC-TOF for highly productive determination of complex analyte mixtures in complex sample matrices.

40 YEARS ANALYTICAL SOLUTIONS GERSTEL

Eike Kleine-Benne

Invited Speakers

Frank David, Ph.D., Research Institute for Chromatography (RIC), Belgium

Nobuo Ochiai, Ph.D., GERSTEL K.K., Japan

Solutions for Comprehensive Food Packaging Migration Studies

If chemicals from packaging materials migrate into the packaged goods, there could be a potential health risk for those that come into contact with the goods. This is especially true in the case of packaged foods.

The EU has issued various regulations, especially the directives 82/711/EEC, 85/572/EEC and 2002/72/EU, in order to ensure early detection of risks. Analyses are performed in laboratories using food simulants such as aqueous solutions, dilute acetic acid, water-ethanol mixtures and plant oils.

The list of potential migrants is as long as the list of different matrices and as that of the different food additives. Catching all using a single analysis technique is plainly not possible. But there are ways and means to perform the required analyses in an efficient, rugged and highly sensitive manner. Frank David, Ph.D., R&D Manager at the Research Institute of Chromatography (RIC) of Professor Pat Sandra has looked at the matter in greater depth.

The solution lies in automated high-performance sample preparation techniques since sample preparation generally makes up the bulk of the laboratory workload. The RIC is using the GERSTEL MultiPurpose Sampler (MPS) a proven autosampler and sample preparation robot for LC and GC. The MPS enables automation of a long list of sample preparation and analyte concentration techniques. The list starts at the classical headspace technique and continues via solid phase Extraction (SPE) and Solid Phase MicroExtraction (SPME) all the way to Stir Bar Sorptive Extraction (SBSE) using the GERSTEL Twister.

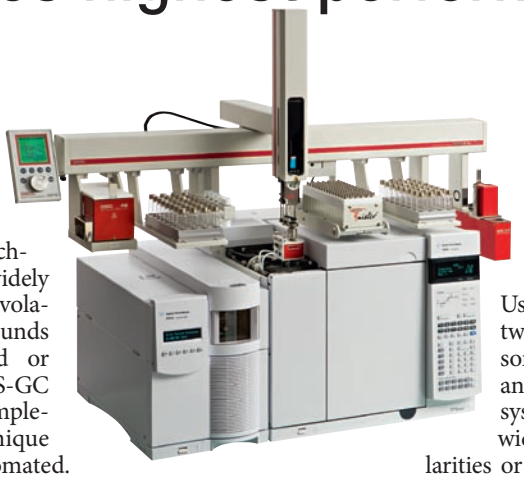
The various sample preparation modules and accessories for the MPS as well as automated liquid sample preparation steps are easily and efficiently controlled using the GERSTEL MAESTRO Software whether stand alone or fully integrated with the Agilent Technologies ChemStation software.

In his lecture at the GERSTEL vendor seminar, Dr. David provides an overview of the approach to studies of food migration from packaging at the RIC.

For more information:
AppNote 1/2007: „Automated Dynamic Headspace Sampling using Replaceable Sorbent Traps“, http://www.gerstel.de/p-gc-an-2007-01.pdf

Comparing automated extraction techniques

Dynamic Headspace (DHS) provides highest performance



Traditional static Headspace gas chromatography (HS-GC) is a technique that is widely used to determine volatile organic compounds (VOCs) in liquid or solid samples. HS-GC is a rugged and simple-to-perform technique that is easily automated. Unfortunately, HS-GC does not provide the level of sensitivity achievable by Headspace-Solid Phase MicroExtraction (HS-SPME)-GC or by Dynamic Headspace (DHS)-GC. GERSTEL set out to compare the performance of these three techniques based on a range of analytes and the following sample matrices: ground coffee, shower gel and cheese. The GERSTEL MultiPurpose Sampler (MPS) ensured that all three techniques were reliably automated.

„Whether for the extraction and concentration of analytes from shower gel, coffee or cheese, the DHS technique won out in all cases while providing quality of results in terms of repeatability equal to the other techniques. DHS is simply more sensitive, providing lower detection limits“, says Eike Kleine-Benne, Ph.D., R&D project manager for GERSTEL.

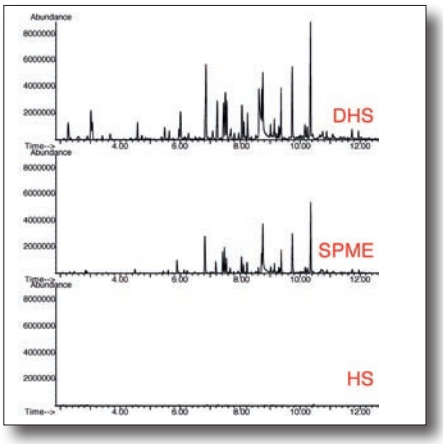
In DHS, equilibrium between the phases is deliberately avoided as analytes are purged away from the sample headspace and trapped on a suitable adsorbent. This means that analytes are more efficiently removed from the liquid, viscous or solid sample and transferred to the analysis system providing a marked improvement in sensitivity and detection limits compared with classical Headspace GC.

DHS is a simple and reliable analytical tool used to concentrate and determine small amounts of analytes from liquid or solid samples. The GERSTEL MPS performs all DHS steps in a reliable and repeatable manner. Samples are placed in standard disposable 20 mL headspace vials and for each sample, a separate adsorbent tube can be used. This means that carry over from sample to sample can be completely eliminated or at least reduced to an absolute minimum.

For the concewtration step, a number of standard adsorbents can be used, such as carbon-based adsorbents, Tenax TA or even PDMS foam sorbent. A selection of pre-packed adsorbent tubes is available.

Additionally, users can pack tubes with adsorbent(s) of their choice. Using a tube with two or more adsorbents for the analysis enables the system to cover a wider range of polarities or a wider boiling-point range.

There are no valves or transfer lines in the Thermal Desorption Unit (TDU) system used to desorb the DHS tubes. This means that loss of analytes is dramatically reduced. An inert gas transfers analytes efficiently from the sample to the adsorbent tube and later from the tube to the directly attached cool trap and on to the GC/MS system.



Coffee: The DHS technique won out in all cases

Compared with SPME, the DHS adsorbent trap provides a much better phase ratio enabling significantly lower detection limits. All steps in the DHS process are selected by mouse-click in the MAESTRO Software (cf. Page 4) and are performed reliably by the MPS.

The steps in the DHS process are intelligently overlapped using the PrepAhead function. This means that the DHS process for a sample is performed during the GC run of the preceding sample for maximum throughput and system utilization. Since only the headspace is purged, there is no risk of foaming and system contamination and the associated instrument down-time for cleaning.

Sequential SBSE and thermal desorption

Take two!

A method for ultra-trace analysis of organic pollutants in aqueous sample was developed using sequential stir bar sorptive extraction and thermal desorption – comprehensive two-dimensional GC – time-of-flight mass spectrometry (Sequential SBSE – TD – GC x GC – TOF-MS). A novel SBSE procedure termed sequential SBSE provides more uniform enrichment over the entire polarity/volatility range for organic compounds at trace level in aqueous sample. Sequential SBSE consists of a SBSE performed sequentially on respectively a 5-mL sample without modifier and a 5-mL sample containing 30 % NaCl using two stir bars. The first extraction with unmodified sample mainly targets the solutes with high K_{ow} (Log/K_{ow} > 4.0), second extraction with modified sample solution (containing 30% NaCl) targets solutes with low and medium K_{ow} (Log/K_{ow} < 4.0).

After extraction the two stir bars are placed in a single glass desorption liner and are simultaneously desorbed for subsequent GC – MS or GC x GC – TOF-MS analysis. Recovery of model compounds consisting of 82 pesticides (organochlorine, carbamate, organophosphorous, pyrethroid, and others) and 6 polycyclic aromatic hydrocarbons (PAHs) for sequential SBSE was evaluated as a function of Log/K_{ow} (1.70 - 8.35).

The recovery of sequential SBSE was compared with those of normal SBSE with or without salt addition (30 % NaCl). Although the recovery for 5 solutes with low K_{ow} (Log/K_{ow} < 2.5) showed less than 80 %, the sequential approach provided very good recovery in the range of 82 to 113 % for the rest of the solutes, while single approaches (with or without salt addition) showed less than 80 % recovery for 23 and 41 solutes, respectively.

Sequential SBSE – TD – GC x GC – TOF-MS method showed good linearity (r^2 > 0.9900) and high sensitivity (limit of detection: < 10 pg/mL) for most of the model compounds. The method was successfully applied to screening and quantification of pesticides at ng/L level in river water and brewed green tea.

Nobuo Ochiai

Automated Liquid Sample Introduction

Direct Thermal Extraction in disposable micro-vial inserts

Automated Tube EXchange (ATEX) has been introduced for the GERSTEL MultiPurposeSampler (MPS) in combination with the GERSTEL Thermal Desorption Unit (TDU). The ATEX option enables the introduction of liquid samples directly into micro-vial inserts used for thermal Desorption / Thermal Extraction in the TDU. Extracted analytes are refocused and concentrated in a Cooled Injection System (CIS) inlet prior to introduction to the GC/MS system. The efficient extraction and concentration ensures highest possible sensitivity and lowest detection limits. The analysis system is kept free of high-boiling contaminants and matrix residue ensuring best possible stability and system uptime.

The ATEX micro-vials can be used for liquid or solid samples. Up to 196 samples can be processed automatically for determination of VOC / SVOC in heavy or involatile matrices. Following the analysis, the sample cup with the remaining high-boiling or solid residue is automatically removed. ATEX helps to ensure maximum uptime and best possible analysis results by keeping involatile or complex matrix material out of the GC/MS system. Standard addition and other liquid phase sample preparation steps can performed automatically by the MPS. The complete system is controlled directly from the GERSTEL MAESTRO Software or integrated with the Agilent ChemStation. Just one method and one sequence table controls the complete process from Sample Introduction through Thermal Desorption to GC/MS analysis ensuring the simplest possible operation.

Frank David, Ph.D.
R&D Manager at the Research Institute for Chromatography (RIC) and currently, part-time Visiting Professor at the University of Ghent, Pfizer Analytical Research Center (PARC).

Nobuo Ochiai, Ph.D.
Application specialist at GERSTEL K.K. in Japan, in Japan, author of more than 15 journal papers, seven of which on SBSE.

Pieter Stoutjesdjik
From the International Sales Department is responslible for Europe, Africa and the Middle East.

Eike Kleine-Benne, Ph.D.
R&D Project Manager at GERSTEL. Dr. Kleine-Benne coordinates R&D projects with external partners such as universities, research institutes as well as private clients and partners of GERSTEL.

Research Institute for Chromatography (RIC)

The Research Institute for Chromatography (RIC) was founded in 1986 by Prof. Dr. Pat Sandra and has been involved in the development and promotion of chromatography know-how from the very start. RIC offers analytical services to industry and to private and governmental laboratories. In collaboration with customers, new methods are developed, evaluated and validated. Very often, the final step of this method development work is the transfer of instrumentation, knowledge and know-how to the customer's laboratory.

GERSTEL offers Customer focused solutions for LC/MS and GC/MS

Single vendor solutions for LC/MS and GC/MS from GERSTEL:

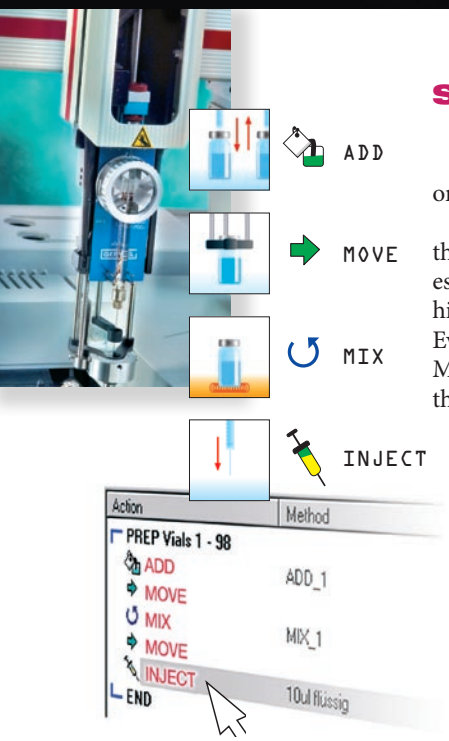
- Automated sample prep and sample introduction
- Integrated software control
- Complete systems focused on your needs
- Application support
- Qualified service: Installation, validation (IQ-OQ/PV), familiarization and training

GERSTEL – your partner for chromatography solutions

GERSTEL MAESTRO Software

Next generation software for automated sample preparation and sample introduction. MAESTRO optimizes performance and throughput of GERSTEL modules and systems.

- Stand-Alone operation or fully integrated in the Agilent ChemStation Software
- One sequence table operates the entire system including LC/MS or GC/MS
- Sample Prep by mouse-click using the PrepBuilder functions
- Scheduler for easy planning
- PrepAhead: Automated overlapping of sample prep and analysis for optimum productivity and throughput
- Priority samples can be added to the system at any point in the analysis sequence
- LOG file and Service LOG file functions ensure traceability
- Automated E-mail notification in case the system is halted
- Control of up to 4 systems
- Real-time monitoring of all modules and parameters
- Interactive on-line help function



Sample prep by mouse click

The MPS is a fully automated autosampler for sample preparation and sample introduction robot for LC and GC.

Sample preparation steps are performed during the analysis of the preceding sample for best possible system utilization and highest sample throughput. Every step is perfomed in a controlled and highly accurate and reproducible manner for best possible results. Every step is selected by mouse-click from a pull-down menu in the MAESTRO software and added to the overall LC or LC/MS method. Available sample preparation techniques are:

- Solid Phase Extraction (SPE)
- Standard addition
- Derivatization
- Extraction and dilution
- Heating, conditioning and mixing
- Twister Back Extraction (TBE)
- Automated Liner EXchange (ALEX)
- Automated Twister (SBSE) desorption and analysis
- Solid Phase Micro Extraction (SPME)
- Thermal Desorption (TDS)
- Dynamic Headspace (DHS)
- Multi Column Switching (MCS)

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45473 Mülheim an der Ruhr, Germany

Editor
Guido Deußing,
ScienceCommunication,
Neuss, Germany
guido.deussing@t-online.de

Scientific advisory board
Eike Kleine-Benne, Ph.D.
eike_kleine-benne@gerstel.de
Oliver Lerch, Ph.D.
oliver_lerch@gerstel.de

Contact
gerstel@gerstel.com

Design
Paura Design, Hagen, Germany
www.paura.com

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GERSTEL

GLOBAL ANALYTICAL SOLUTIONS

GERSTEL GmbH & Co. KG
Eberhard-Gerstel-Platz 1
45473 Mülheim an der Ruhr
GERMANY
☎ +49 208 - 7 65 03-0
🖷 +49 208 - 7 65 03 33
@ gerstel@gerstel.com
🌐 www.gerstel.com

GERSTEL, Inc.
1510 Caton Center Drive
Suite H
Baltimore, MD 21227
USA
☎ +1 410 - 247 5885
🖷 +1 410 - 247 5887
@ info@gerstelus.com
🌐 www.gerstelus.com

GERSTEL AG
Enterprise
Surentalstrasse 10
6210 Sursee
SWITZERLAND
☎ +41 41 - 9 21 97 23
🖷 +41 41 - 9 21 97 25
@ gerstel@ch.gerstel.com
🌐 www.gerstel.de

GERSTEL K.K.
2-13-18 Nakane
Meguro-ku
152-0031 Tokyo
JAPAN
☎ +81 3 57 31 53 21
🖷 +81 3 57 31 53 22
@ info@gerstel.co.jp
🌐 www.gerstel.co.jp

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