


GERSTEL

ISC 2008 Münster

Newsletter

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News from GERSTEL GmbH & Co. KG · Eberhard-Gerstel-Platz 1 · 45473 Mülheim an der Ruhr · Germany · Phone +49 (0) 208 - 7 65 03-0 · gerstel@gerstel.com

September 2008

Improving performance, selectivity and stability

In many application areas, the determination of pesticides in food for one, extensive sample preparation and sample clean-up is necessary in order to reach the required limits of determination and in order to keep the analysis system sufficiently stable for a routine analysis environment. Method development and sample preparation for GC/MS and LC/MS analysis is not a trivial matter. In recent years, opinions have been voiced that samples can be introduced to the analysis system without any or much preparation and compounds detected and quantitated without prior separation. Especially LC/MS or LC-MS/MS systems, it was said, would handle the matter nevertheless. Even with highly sophisticated mass spectrometers, however, a high level of matrix background can significantly influence system stability and can produce significant analytical interferences. The benefits of automated sample preparation and automated matrix elimination for the determination of pesticides by GC/MS and LC/MS are demonstrated in the material shown in this GERSTEL Newsletter, which is published for ISC 2008 in Münster.

About GERSTEL

GERSTEL develops, produces and supports solutions that include automated sample preparation and sample introduction for GC/MS and LC/MS. The available techniques include automated Solid Phase Extraction (SPE), which can be performed in combination with Standard Addition, Derivatization, and Eluate Concentration with or without Keeper Solvent. Additionally, in combination with a GC or GC/MS system, Automated Liner Exchange (ALEX) enables automated matrix elimination in the GC, allowing the user to dramatically reduce the amount of effort going into sample preparation, for example by using the QuEChERS sample preparation method for the determination of pesticides in non-fatty fruits and vegetables.

Sample preparation is performed simultaneously with the LC/MS or GC/MS run of the preceding sample enabling highest possible productivity and system utilization. The system is controlled through the GERSTEL MAESTRO software in stand-alone mode or fully integrated with the Agilent Technologies GC/MS or LC/MS software. One method and one sequence table controls the complete system.

Pesticide analysis EZ

When the sample matrix no longer matters

Application specialists from TeLA GmbH, Germany, have developed a new method that dramatically simplifies LC/MS determination of pesticide levels, providing high-quality results independent of the sample matrix and complexity.

The standard QuEChERS method enables rapid sample preparation for determination of pesticides in fruits and vegetables. The main benefit of this sample preparation method is that the overall analysis is less time-consuming and less error-prone than more traditional approaches. The limits of QuEChERS are encountered whenever samples with more complex matrices need to be analyzed, such as garlic, onion, artichoke or avocado with high fat content. This can lead to problems with interferences, that can especially influence quantification unless further clean-up steps are performed.

The GERSTEL SPE system provides an excellent solution, enabling reliable and rugged analysis independent of the sample matrix. The system was previously used successfully for the determination of aflatoxins, chloramphenicol and malachite green in foods (please see articles in the LC/MS special issue).

Raw sample extracts were automatically loaded onto standard SPE cartridges and cleaned. A new cartridge was used for every sample to eliminate cross-contamination. Automated SPE clean-up as described in this article took around 20 minutes to complete. Apart from the first sample, the SPE process was performed during LC/MS or GC/MS analysis of the preceding sample, ensuring that the SPE step was performed without increasing the overall analysis time.

Sample clean-up using SPE contributed not only to the ruggedness of the method, it also improved reproducibility and linear-



The SPE LC-MS/MS system used by the TeLA scientists for the pesticide multi-residue method consisting of an Agilent Series LC 1200 and a GERSTEL SPE system mounted over an Agilent 6410 MS/MS Triple Quad.

GERSTEL VENDOR SEMINAR

Monday, September 22, 2008
12:45 - 13:45, Auditorium 2 (HS 2)

Sample Prep Automation for analysis of food contaminants
Automated SPE and sample preparation for improved detection limits and reproducibility in HPLC-MS determination of assorted contaminants in food

Dr. Norbert Helle, TeLA GmbH, Bremerhaven

Automated sample preparation and cleanup in pesticide analysis
Dr. Eike Kleine-Benne, GERSTEL GmbH & Co. KG

ity, among other things. Orange oil samples were cleaned up using a slightly modified SPE method. Recovery for various pesticides in this difficult matrix ranged from 70 to 90 % while recoveries from fruit and vegetable samples were mainly in the range from 80 to 100 %.

A Europe-wide round robin was successfully passed. A vegetable sample (zucchini) had to be analyzed for 185 different pesticide residues. Out of 46 laboratories,

TeLA using the GERSTEL SPE was among the 12 that managed to correctly identify and quantify the analytes thus meeting the round robin requirements and passing the test.



Pesticide Analysis of non-fatty foods with reduced sample preparation

Recently, a new multiresidue method for analysis of fruits and vegetables was presented (QuEChERS, Quick Easy Cheap Effective Rugged Safe) [1]. In comparison with previously used methods, QuEChERS enables rapid sample preparation for determination of pesticides. In one example eight samples were prepared in less than 30 minutes. The main benefit of QuEChERS is that it provides a less time-consuming and a less error-prone analysis. Unfortunately extracts obtained following this procedure often contain high levels of matrix that cause problems in the GC-analysis due to liner contamination. When dirty extracts are injected to the GC, liner contamination is quickly experienced, affecting the recovery of many pesticides. As an example, 20 injections of 5 μ L of a standard solution in bell pepper matrix in a baffled empty liner (deactivated) were performed in a study. The peak area trend of three different pesticides was presented. For endosulfan sulfate and chlorthalonil a decrease in peak areas was observed within this series of samples. This can be explained by increasing matrix contamination of the liner leading to analyte loss. For dichlorphos the peak areas obtained increased within the sequence. This effect is described in literature as "matrix – induced chromatographic response enhancement" [2]. This is experienced when matrix components cover remaining active sites in the chromatographic system leading to higher response for sensitive analytes.



GERSTEL Automated Liner EXchange ALEX

When analyzing extracts obtained using the QuEChERS-method, a liner exchange is required after 10 to 15 runs for difficult vegetables such as bell pepper. For this reason, an automated liner exchange system is required in order to perform the analysis on a routine basis with a reasonable sample throughput.

The GERSTEL Automated Liner EXchange (ALEX) enables routine GC analysis of samples containing large amounts of matrix or other solid residue. ALEX replaces the GERSTEL CIS inlet liner at user-defined intervals, eliminating the need for time-consuming clean-up steps during sample preparation. ALEX is an add-on module for the GERSTEL MultiPurpose Sampler (MPS). Control of the complete system is provided by the MAESTRO Software, which can be operated fully integrated with the Agilent Technologies ChemStation. One integrated method and one sequence list operate the complete system including GC and MSD.

References

- [1] M. Anastassiades, S. Lehotay, D. Stajnbaher and F. Schenck: **Fast and easy multiresidue method employing acetonitrile extraction/partitioning and „dispersive solid-phase extraction“ for the determination of pesticide residues in produce.** J AOAC Int 86(2) (2003) 412-31.
- [2] M. Anastassiades, K. Mastovska, S.J. Lehotay: **Evaluation of analyte protectants to improve gas chromatographic analysis of pesticides.** J. Chromatogr. A 1015 (2003) 163-184.

For more information:

AppNote 4/2006

(<http://www.gerstel.com/p-gc-an-2006-04.pdf>)

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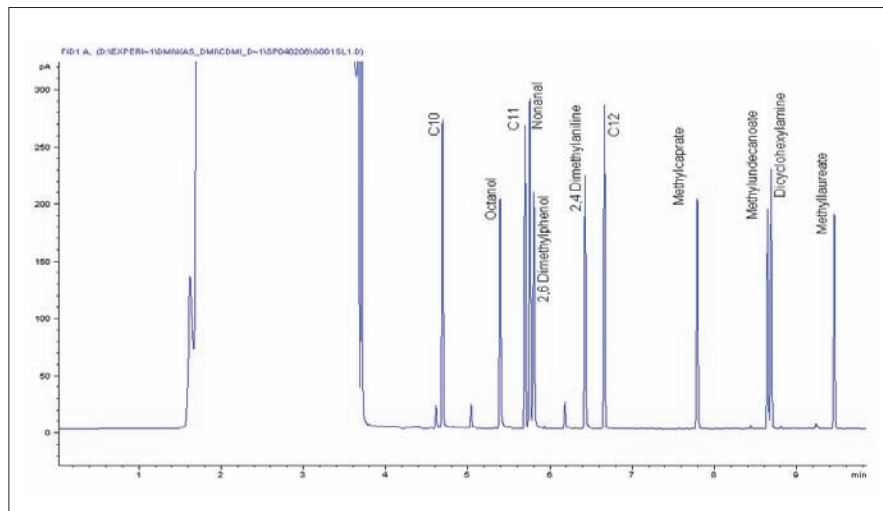


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Design

Paura Design, Hagen,
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www.paura.com



GC FID chromatogram of a Grob test mixture (1 μ L splitless) injected into a CIS 4 equipped with the ALEX system

Fast Screening of Pesticide Multiresidues in Aqueous Samples

A method for fast screening of pesticide multiresidues in aqueous samples using dual stir bar sorptive extraction (dual SBSE) - thermal desorption (TD) – fast GC/MS has been developed. Recovery of 82 pesticides – organochlorine, carbamate, organophosphorous, pyrethroid and others – for the SBSE was evaluated as a function of octanol water distribution coefficients ($\log K_{ow}$: 1.7-8.35), sample volume (2-20 mL), salt addition (0-30 % NaCl), and methanol addition (0-20 %). The optimized method consists of a dual SBSE performed simultaneously on respectively a 20 mL sample containing 30 % NaCl and a 20 mL sample without modifier (pure sample). One extraction with 30 % NaCl is mainly targeting solutes with low $\log K_{ow}$ (< 3.5) and another extraction with unmodified sample solution is targeting solutes with medium and high $\log K_{ow}$ (> 3.5). After ex-

traction, the two stir bars were placed in a single glass desorption liner and were simultaneously desorbed. The desorbed compounds were analyzed by fast GC/MS using a Modular Accelerated Column Heater (MACH). The method showed good linearity ($r^2 > 0.9900$) and high sensitivity (limit of detection: < 10 ng/L) for most of the target pesticides. The method was applied to the determination of pesticides at ng/L levels in river water and brewed green tea.

For more information:

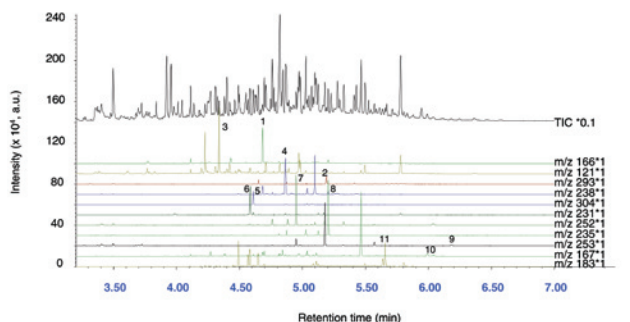
AppNote 12/2006 (<http://www.gerstel.de/p-gc-an-2006-12.pdf>); AppNote 4/2004 (<http://www.gerstel.com/p-gc-an-2004-04.pdf>); AppNote 3/2005 (<http://www.gerstel.com/p-gc-an-2005-03.pdf>)



Automated SPE

GERSTEL has introduced automated Solid Phase Extraction (SPE) for the MultiPurpose Sampler (MPS). The system is based on standard 6, 3 and 1 mL cartridges, using disposable needles for liquid transfer to eliminate carry-over. Users can work on a variety of sample types due to the flexible rinse and elution capabilities offered by the MPS. It is possible to combine automated SPE with sample prep steps and with introduction to LC/MS or GC/MS systems. By performing SPE and chromatography in parallel, productivity can be optimized. The complete system is controlled by the GERSTEL MAESTRO software or integrated with Agilent Technologies ChemStation.

Total ion chromatogram and some representative mass chromatograms obtained by Dual SBSE-TD-LTM GC/MS of a river water sample spiked with 82 pesticides at 500 ng/L level.



1. Pirimicarb ($\log K_{ow} = 1.70$); 2. Fensulfothion ($\log K_{ow} = 2.35$); 3. Fenobucarb ($\log K_{ow} = 2.79$); 4. Metolachlor ($\log K_{ow} = 3.24$); 5. Diazinon ($\log K_{ow} = 3.86$); 6. Terbufos ($\log K_{ow} = 4.24$); 7. Pendimethalin ($\log K_{ow} = 5.18$); 8. p,p-DDD ($\log K_{ow} = 5.87$); 9. Deltamethrin ($\log K_{ow} = 6.18$); 10. Cyhalothrin 1,2 ($\log K_{ow} = 6.65$); 11. Permethrin 1,2 ($\log K_{ow} = 7.43$);

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To get more information about GERSTEL innovations for GC (GC/MS) and LC (LC/MS), please visit the GERSTEL booth B5!

Automated Dynamic Headspace

Automated Dynamic Headspace (DHS) has been introduced for the GERSTEL MultiPurpose Sampler (MPS) based on newly developed technology.

A DHS station in the MPS is used to efficiently extract and concentrate VOCs from liquid or solid samples placed in standard Headspace vials. The DHS station provides thermostating and agitation as well as purging of the sample headspace with inert gas. Sample temperatures can be selected from 200 °C to as low as 10 °C, enabling control of the amount of water vapour released and ultimately retrapped. The DHS station additionally holds a replaceable adsorbent or sorbent filled tube used for analyte concentration. The temperature of the adsorbent tube during the DHS process can be varied from 20 °C to 70 °C for optimal trapping of the analytes in question. Adsorbent tubes used are standard GERSTEL TDU Thermal Desorption Unit tubes. Following analyte concentration, the tube is automatically transferred to the TDU

for Thermal Desorption and analyte transfer to the GC. Tubes are fitted with individual adapters enabling both automated movement and leak-free sealing during the DHS and Thermal Desorption steps and during storage in the autosampler tray. Automated liquid handling in the MPS can be used for additional sample prep steps. As part of the sample prep procedure, standard addition can be performed automatically for best possible analytical results.

Automated DHS combined with sample introduction to a GC or GC/MS system is performed by the MPS and TDU. Up to 98 samples can be processed automatically in one sequence. Parallel processing of samples enable the DHS process to be performed during the chromatographic run of the preceding sam-

ple for optimal productivity and for highest system utilization.

Sample prep steps are selected by mouse-click from a pull-down menu using the PrepBuilder function of the GERSTEL MAESTRO software.

A graphical scheduler display for the DHS process provides an overview of the time required for each individual step and for the complete sequence of samples for easier planning.

Just one method and one sequence table controls the complete process from DHS through sample prep and sam-

ple introduction to GC/MS analysis. Integrated control ensures a more efficient operation with less risk of error. The GERSTEL MAESTRO Software operates stand-alone or fully integrated with the Agilent Technologies ChemStation Software.



GERSTEL MAESTRO software

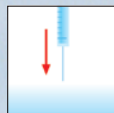
Next generation software for automated sample preparation and sample introduction. MAESTRO optimizes performance and throughput of GERSTEL modules and systems.

- 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 / Multiple Sample Overlap: Automated overlapping of sample preparation and analysis for maximum 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 if the sequence is stopped
- Control of up to 4 systems from one PC
- Real-time monitoring of all modules and parameters
- Interactive on-line help function

Sample Prep by Mouse-Click

The MultiPurpose Sampler (MPS) is an autosampler and sample preparation robot for GC and LC. Sample preparation steps are performed during the analysis of the preceding sample for best possible system utilization and highest sample throughput. Sample preparation steps are performed 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 GC/MS or LC/MS method. Available sample prep techniques are:

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



Action	Method
PREP Vials 1 - 98	
ADD	ADD_1
MOVE	
MIX	MIX_1
MOVE	
INJECT	10µl Flüssig
END	

MAESTRO Software enables Sample Prep by Mouse-Click. All sample preparation steps are conveniently and easily selected from a drop down menu and added to the method. Example:



ADD

Add solvent, internal standard or reagent



MOVE

Move the vial or cartridge



MIX

Agitate or stir and incubate the sample at a set temperature



INJECT

Introduce an aliquot of the sample to the GC or LC system

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