Automated extraction techniques

**Step by step**

1. Standard 1, 3 or 6 mL SPE cartridges are available equipped with transport adapters for easiest possible automation using the GERSTEL SPE. An added benefit is that the GERSTEL SPE cartridges have practically no dead volume.
2. During conditioning cartridges are prepared for sample loading. Activation steps can be performed by the GERSTEL SPE, in many cases without adding to the overall analysis time.
3. Loading the sample with defined volumes at constant flow rates leads to highly reproducible results even if there are variations in packing density or amount of the solid phase material.
4. Rinsing steps can be performed in order to remove interferences or to introduce a solvent that matches the elution solvent. Up to 12 different solvents can be used, providing unlimited flexibility and enabling effortless implementation of highly complex rinse cycles.
5. Drying the cartridge prior to elution can be helpful in some cases. The GERSTEL SPE lets you automate this intermediate step by mouse click.
6. Retained compounds are eluted from the SPE cartridge into a closed vial via a disposable syringe needle. This eliminates the risk of cross contamination. The obtained eluate can be concentrated by solvent evaporation directly in the collection vial in the "VAP" module.

**Solid Phase Extraction (SPE)**

Solid phase extraction (SPE) is probably one of the most versatile and most frequently used extraction techniques in the field of chemical analysis. SPE is used to eliminate matrix components on the one hand and to extract and enrich analytes on the other. SPE is labor-intensive and time-consuming when carried out by hand, especially when recovery and repeatability leave something to be desired. In contrast, SPE is more relaxed, time-saving, precise and reproducible, if the process is fully automated.

**SmartSPE / μSPE**

SmartSPE or μSPE is a patented microscale solid phase extraction (SBE) technique. SmartSPE / μSPE allows significantly smaller volumes to be handled, which reduces solvent consumption and the time and effort involved in sample preparation. Since SmartSPE / μSPE uses conventional SPE sorbents, existing methods can typically be adapted to the micro-scale approach and implemented.

After conditioning the sorbent, the cartridge is loaded with sample. If necessary, the sorbent bed can be washed to remove matrix components before elution is carried out in the final step using a suitable solvent. Compared to conventional SBE, this process requires significantly less solvent due to the lower quantity of sorbent (10 – 45 mg).

The SmartSPE / μSPE is fully supported by the GERSTEL MultiPurpose Sampler (MPS). Its automation with the MPS not only offers the user the benefits and added value described above. It is also fully integrated with the GERSTEL MassHead functionality and Agilent Technologies MassHunter and ChemStation software. Other GERSTEL modules and sample preparation steps can be used in conjunction with SmartSPE / μSPE. Your daily sequence table is set up just a few mouse clicks – including both sample preparation steps and GC/MS- or LC/MS analysis.

**Sequence by Barcode**

New automation efficiency and QC options have been integrated into the MAESTRO software using the GERSTEL Sample ID (SID) barcode reader. In addition to sample logging and sample ID transfer to the data file and file name, SID enables automated analysis by pre-defined methods as well as automated generation of sequence tables. Single sample mode or Batch Analysis Mode (BAM) is selected by mouse click. Trigger vials can be introduced at user defined intervals, for example, to activate solvent injection(s) for system background checks followed by one or more check standards as part of the QC routine operation. Trigger functions are performed without taking up multiple sample positions in the tray enabling a higher overall analysis throughput while ensuring adherence to set quality control requirements. Multiple options are available for sample verification and in case of a failed calibration, the sample is locked and no further analysis is possible. Data import and export functions enable LIMS or data base synchronization.

**Biomarkers of alcohol consumption**

**Fully automated detection of phosphatidylethanol (PETH) from dried blood spots (DBS)**

By measuring the concentration of alcohol in breath or blood it may be possible to determine whether someone has abstained from drinking alcohol for a longer period of time, as may be required, for example, in connection with drivers’ aptitude tests or detox therapy. In those cases, other methods, including the analysis of specific alcohol biomarkers, are required.

Phosphatidylethanol (PETH) is a promising candidate because it can be detected at sufficient levels in the blood for a long period of time after alcohol has been consumed. However, enzymatic activity in liquid blood samples can cause PETH concentrations to change during storage and transport. PETH analysis from dried blood spots (DBS) is therefore recommended. With the GERSTEL DBS A system, PETH can be detected efficiently, accurately and reliably. All steps in the sample preparation process, including barcode reading, internal standard addition, extraction of a defined sample area from the DBS card, solid phase extraction (SPE) and online LC/MS/MS detection can be performed in a quick, reliable and safe manner, and fully automated from start to finish. More details on the GERSTEL MPS-DBS-LA/MS/MS system are available at the GERSTEL booth No. 62 or send a request to oliver_lerch@gerstel.de.

**Automation**

More efficiency and flexibility in the forensic toxicology laboratory

Automating key processes and routines can increase productivity and performance of your analytical laboratory in a sustainable manner.

**Fully automated detection of phosphatidylethanol (PETH) from dried blood spots (DBS)**

Fully automated detection of phosphatidylethanol (PETH) from dried blood spots (DBS)
In addition to precision and sensitivity of the sample, the volume and the time it takes to achieve measurement results play all a role in analyzing blood. Analysis devices that are simple and easy to use do a lot to improve acceptance among users. Dried Blood Spot (DBS) analysis ticks all the boxes with the MPS-based GERSTEL DBS Autosampler (DBS-A).

Automated Dried-Blood-Spot-Analysis  
What a drop of blood reveals

In addition to SPE workflows, the GERSTEL MultiPurpose Sampler (MPS) can perform fully automated liquid-liquid extraction.

Automated Liquid-Liquid Extraction (LLE)  
Speeding up the workflow

Liquid-liquid extraction is one of the most widely used standard procedures in sample preparation. All relevant steps can be automated with the MPS. The only manual effort required is to load the samples in 2-, 4- or 10 mL vials and place them in the MPS sample trays. The method is conveniently generated by mouse-click. Your daily sequence analysis table is then set up in just a few steps, again, conveniently by mouse-click, and you’re ready to go: the MPS adds an internal standard to the sample, along with extraction buffer and solvent as specified by the user. During the extraction step, the MPS provides efficient agitation for optimal results.

Phases can be separated quickly – thanks to the integrated centrifuge. The MPS removes the relevant phase and transfers it to a clean vial if necessary, a further extraction with fresh solvent can be performed. The MPS can also inject the extract to the analysis system or evaporate it to dryness in the mVAP to reconstitute with an LC-compatible solvent or to add a derivatization reagent.

Whichever steps you follow, you have the choice: the MPS is a flexible and robust tool for the automation of liquid-liquid extraction (LLE) not just due to the wide range of options it affords the user, but because it is astounding simply to control using the MAESTRO software.

Suggested reading


[10] The hematocrit value is determined by near infrared measurement which extracts the sample before analyzing by LC-MS/MS. The automated function was added to the GERSTEL DBS-A, in cooperation with Büchi (www.buchi.com), GERSTEL has the DBS hematocrit value to take and analyzing forensic toxicology and metabolomics is based on its MultiPurpose Sampler (MPS).

In addition to precision and sensitivity of the sample, the volume and the time it takes to achieve measurement results play all a role in analyzing blood. Analysis devices that are simple and easy to use do a lot to improve acceptance among users. Dried Blood Spot (DBS) analysis ticks all the boxes with the MPS-based GERSTEL DBS Autosampler (DBS-A).

Determining DBS hematocrit values automatically

In cooperation with Buchi (www.buchi.com), GERSTEL has implemented automated non-destructive determination of hematocrit values in dried blood spots placed on DBS cards. The automated function was added to the GERSTEL DBS-A, which extracts the sample before analyzing by LC/MS/MS. The hematocrit value is determined by near infrared measurement (NIR) combined with multivariate spectral analysis. The process is fully automated under GERSTEL MAESTRO software, every step is simply and easily set up by mouse-click.
**Automated-Dried-Blood-Spot-Analysis**

**What a drop of blood reveals**

In addition to precision and sensitivity of the sample, the volume and the time it takes to achieve measurement results all play a role in analyzing blood. Analytic devices that are simple and easy to use do a lot to improve acceptance among users. Dried Blood Spot (DBS) analysis ticks all the boxes with the MPS-based GERSTEL DBS Autosampler (DBS-A).

Why GERSTEL, in cooperation with the Dutch company Spark Holland, has developed an automated all-in-one solution (DBS-A) that is based on its MultiPurpose Sampler (MPS) and is either coupled to an HPLC-MS/MS system or available as a workstation. This automated process is currently being used in pharmaceutical and anti-doping laboratories, e.g. at the German Sport University Cologne. It goes without saying that the DBS-A system is an attractive solution for a number of other interesting fields, including forensic toxicology and metabolomics research. The MPS transports the DBS card to a camera, where an integrated image recognition software determines its position and evaluates the quality of the dried blood drop (Dried Blood Spot). The MPS places the card in the desorption interface, where a defined volume of the blood drop (partial or complete sample) is desorbed. The analytes (Flow Through Desorption, FT[D]) are transferred to the HPLC system or cleaned-up using a replaceable SPE cartridge as needed.

After transferring the analytes into the HPLC system or collecting extracts using the workstation, the MPS can be performed fully automated liquid-liquid extraction. Dried Blood Spot (DBS) analysis ticks all the boxes with the MPS method for the determination of THC, 11-OH-THC, and free THC-carboxylic metabolites in urine and dried blood spots.

**References**


**Automated Liquid-Liquid Extraction (LL E)**

**Speeding up the workflow**

Liquid-liquid extraction is one of the most widely used standard procedures in sample preparation. All relevant steps can be automated with the MPS. The only manual effort required is to load the samples in 2-, 4-, or 10-mL vials and place them in the MPS sample trays. The method is conveniently generated by mouse-click. Your daily analysis sequence is then set up in just a few steps, again, conveniently by mouse click, and you’re ready to go: the MPS adds an internal standard to the sample, along with extraction buffer and solvent as specified by the user. During the extraction step, the MPS provides efficient agitation for optimal results.

Phases can be separated quickly – thanks to the integrated centrifuge. The MPS removes the relevant phase and transfers it to a clean vial, if necessary, a further extraction with fresh solvent can be performed. The MPS can alsoject the extract to the analysis system or evaporate it to dryness in the mVAP to reconstitute with an LC-compatible solvent or to add a derivatization reagent. Whichever steps you follow, you have the choice: the MPS is a flexible and rugged tool for the automation of liquid-liquid extraction (LLE) not just due to the wide range of options it offers the user, but because it is outstandingly simple to control using the MAESTRO software.

**Suggested reading**


**Determining DBS hematocrit values automatically**

In cooperation with Buchi (www.buchi.com), GERSTEL has implemented automated non-destructive determination of hematocrit values in dried blood spots placed on DBS cards. The automated function was added to the GERSTEL DBS-A, which extracts the sample before analyzing by LC/MS/MS. The hematocrit value is determined by near infrared measurement (NIR) combined with multivariate spectral analysis. The process is fully automated under GERSTEL MAESTRO software, every step is simply and easily set up by mouse-click.
Solid Phase Extraction (SPE)

Solid phase extraction (SPE) is probably one of the most versatile and most frequently used extraction techniques in the field of chemical analysis. SPE is used to eliminate matrix components on the one hand and to extract and enrich analytes on the other. SPE is labor-intensive and time-consuming when carried out by hand, especially when recovery and repeatability leave something to be desired. In contrast, SPE is more relaxed, time-saving, pre- 

standard SPE, based on standard dimension cartridges. However, these methods are unable to determine whether someone has abstained from drinking alcohol for a longer period of time, as may be required, for example, in connection with drivers’ aptitude tests or detox therapy. In those cases, other methods, including the analysis of specific alcohol biomarkers, are required.

Phosphatidylethanol (PETH) is a promising candidate because it can be detected at sufficient levels in the blood for a long period of time after alcohol has been consumed. However, enzymatic activity in liquid blood samples can cause PETH concentrations to change during storage and transport. PETH analysis from dried blood spots (DBS) is therefore recommended.

With the GERSTEL DBS A system, PETH can be detected efficiently, accurately and reliably. All steps in the procedure are fully automated, including barcode reading, internal standard addition, extraction of a defined sample area from the DBS card, solid phase extraction (SPE) and online LC/MS/MS detection can be performed in a quick, reliable and safe manner, and fully automated from start to finish. More details on the GERSTEL MPS-DBS/MS/LC/MS system are available at the GERSTEL booth No. 62 or send a request to oliver_lerch@gerstel.de.

Biomarkers of alcohol consumption

Automating key processes and routines can increase productivity and performance of your analytical laboratory in a sustainable manner.

Automating sample preparation and introduction depends to a large extent on the right choice of autosampler. The GERSTEL MPS is an innovative X-Y-Z robot that can be used for many sample preparation and introduction tasks for GC/MS and LC/MS. The MPS can be operated integrated with the analytical instrument or as an independent workstation and can be flexibly configured and extended.

Examples of MPS-based application solutions in the field of forensic toxicology are, among others, the determination of tetrahydrocannabinol (THC), cannabinol (CBN) and cannabidiol (CBD) as well as the THC metabolites 11-Hydroxy-THC (THC-OH) and 11-Nor-9-carboxy-THC (THC-COOH) in serum and hair (cf. references on page 3). The MPS automates extraction and purification techniques such as liquid-liquid extraction and solid phase extraction (SPE) and adds internal standard to the sample. The available SPE techniques include standard SPE, based on standard dimension car- 

trigides, m-scale SPE as well as online SPE (GERSTEL SPE®) with exchangeable cartridge that are directly coupled to the LC/MS system for quantitative analyte transfer and optimized sensitivity. The MPS offers mechanical mixing and separation techniques based on mechanical agitation, ultrasonication, filtration, centrifugation. Solutions and liquid extracts can be automatically weighed (weighing option), kept at defined temperature, agitated (PVMIX), evaporated (AVAP) and derivatized (PVDAP) and derivatized. The MPS produces dilution series and calibration standards both accurately and reproducibly. The MPS also automates a wide range of standard techniques, such as Solid Phase Micro Extraction (SPME), Static Headspace (SHS) and Dynamic Headspace (DHS), Stir Bar Sorptive Extraction (SBSE, GERSTEL, Twister) as well as thermal desorption and pyrolysis. A wide range of sample vessels for a variety of different tasks and applications are available. In addition to standard trays with optional temperature control, space-saving cool stacks with trays in drawers allow a maximum number of samples to be stored protected from heat and light.

By measuring the concentration of alcohol in breath or blood it may be possible to determine whether someone has abstained from drinking alcohol for a longer period of time, as may be required, for example, in connection with drivers’ aptitude tests or detox therapy. In those cases, other methods, including the analysis of specific alcohol biomarkers, are required.

Phosphatidylethanol (PETH) is a promising candidate because it can be detected at sufficient levels in the blood for a long period of time after alcohol has been consumed. However, enzymatic activity in liquid blood samples can cause PETH concentrations to change during storage and transport. PETH analysis from dried blood spots (DBS) is therefore recommended.

With the GERSTEL DBS A system, PETH can be detected efficiently, accurately and reliably. All steps in the procedure are fully automated, including barcode reading, internal standard addition, extraction of a defined sample area from the DBS card, solid phase extraction (SPE) and online LC/MS/MS detection can be performed in a quick, reliable and safe manner, and fully automated from start to finish. More details on the GERSTEL MPS-DBS/MS/LC/MS system are available at the GERSTEL booth No. 62 or send a request to oliver_lerch@gerstel.de.

Simple and exact sampling of a 10 μL volume of whole blood onto Dried Blood Spot (DBS) cards. Image: DBS System