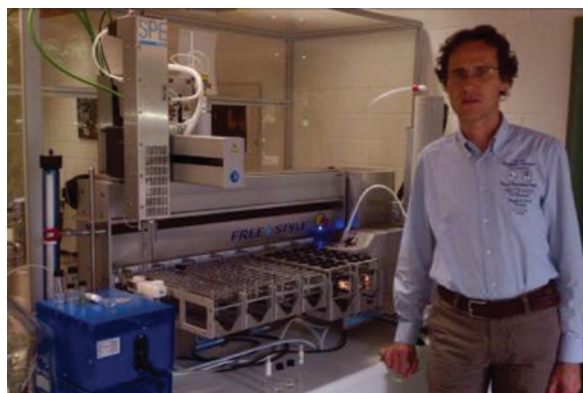


Robotic System in Combination with Pre-packed Glass Columns Reduces Labour and Increases Throughput

Until 2012, the clean-up of soil, sediment and biota samples with silica gel or Florisil was performed manually at the Dr. Nowak Limnological Institute. Despite standardised sample preparation methods, larger deviations could often not be avoided in this time-consuming process. One factor, which influenced this was columns still having to be filled by hand, a procedure in which variations could not be fully excluded. Approximately two years ago, the Institute started using the FREESTYLE™ robotic system developed by LCTech GmbH for automated sample preparation; and since 2013 in combination with the ready-to-use Florisil glass columns. In a fully automated process, sample extracts are loaded onto the ready-to-use columns, the substances to be analysed are directly eluted into the connected evaporation unit and, if required, subsequently subjected to gel permeation chromatography (GPC). In this way, errors can be avoided and work time can be saved: Currently, up to 150 samples per week are analysed by the Institute. Instead of expending significant time in sample preparation, the staff can now focus more strongly on measurements and evaluations.

“Now we are in a position to analyse 100 to 150 samples per week. Previously, in order to achieve this, a member of staff would have had to work exclusively on sample preparation all day long”

“Since 2010, we have used the LCTech GPC system for the clean-up of sediment and biota samples. Before conducting a GPC clean-up, solid-phase extraction with silica gel or Florisil is necessary. Consequently, in 2012, we decided to purchase the FREESTYLE™ system, which is able to automate these steps,” explains Dr. Thomas Brandsch, Head of Department for Residue Analysis at the Dr. Nowak Institute in Ottersberg. Apart from sediment and biota samples, soil samples are also processed using the FREESTYLE™, all of which are examined for polycyclic aromatic hydrocarbons (PAH) and chlorinated hydrocarbons (organochlorine pesticides and PCBs). The measurement is then carried out by means of GC-MS.



Since 2012, the FREESTYLE™ robotic system has been used by the Dr. Nowak Institute. Soil, sediment and biota samples are processed with this system and tested for polycyclic aromatic hydrocarbons (PAHs) and chlorinated hydrocarbons (organochlorine pesticides and PCBs). The measurements are subsequently undertaken with a GC-MS.

Fully automated clean-up overnight

Before introduction of the FREESTYLE™, the necessary clean-up steps had to be done by hand. “It was quite a time consuming task and despite standardised sample preparation, the results often depended on which employee had performed the task,” explains Brandsch. In addition, the glass columns were filled in the Institute and inevitably subjected to fluctuations. With the FREESTYLE™ and

pre-packed columns, the manual work is reduced to placing the sample extract into the unit and to filling solvent into the reservoir. “The rest is automatic, which saves working time and helps to avoid mistakes,” says the expert analyst.

The test sample is freeze-dried prior to the clean-up in the robotic device and then extracted using an ASE system. The volume of the extract is reduced until it can be filled into a sample vial and placed into the FREESTYLE™. In the system, the extract is then loaded onto a ready-to use column and eluted in the evaporation unit. Here, the volume is reduced to a final volume as defined in the method, then transferred to a sample vial. “Generally, the clean-up runs overnight that by the next morning, the purified extracts are available for subsequent processing,” explains Brandsch. The sample weight is matched to the sensitivity of the GC-MS system, such that an aliquot of the purified sample extracts can be directly transferred into a GC vial and measured without further concentration. In biota samples and highly matrix-contaminated sediment samples, identifiable by their intensive staining, the extract will be also subjected to a GPC clean-up, which runs analogously: The extract to be cleaned is placed in a sample vial into the FREESTYLE™, cleaned, concentrated to a defined end volume and then transferred into a vial. Both clean-up steps can run automatically in succession.

Easy- to- operate, flexible robotic system

“The operation of the FREESTYLE™ requires initial programming of the methods. Thereafter, its inclusion in the daily routine is intuitive and very clear,” says Brandsch. In the system software, the user can first create the desired processing methods for the samples. In this way, the design of very complex procedures is also possible: “Either individually installed modules can be selected, or different functions can be combined within one method, which is the Dr. Nowak Institute preference,” explains Michael Baumann, General Manager of LCTech. “The respective modules - SPE, Evaporation and GPC – are simply activated via the software.” In this way, comprehensive sequence lists can be created using different procedures for unattended operation around the clock, day and night.

The basic method structure for an arbitrary method can be defined

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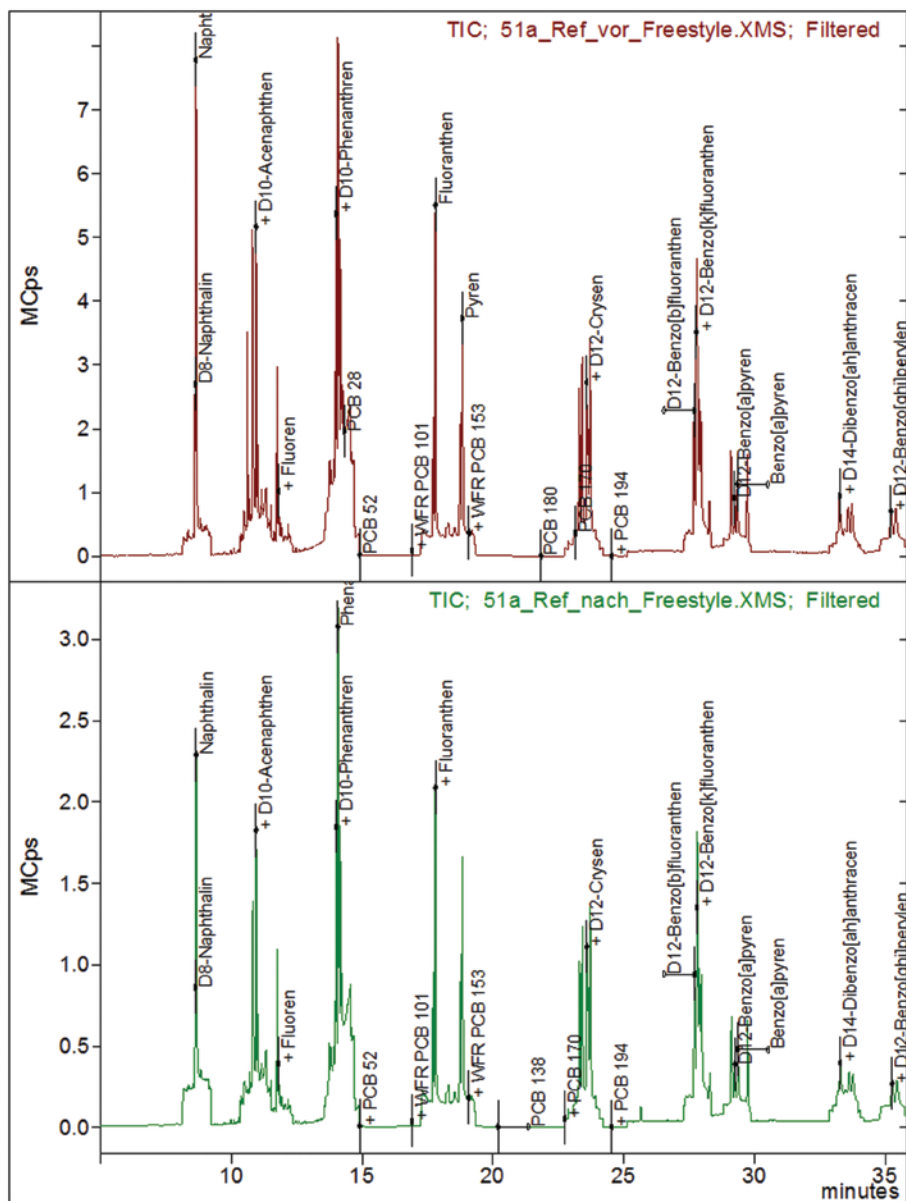
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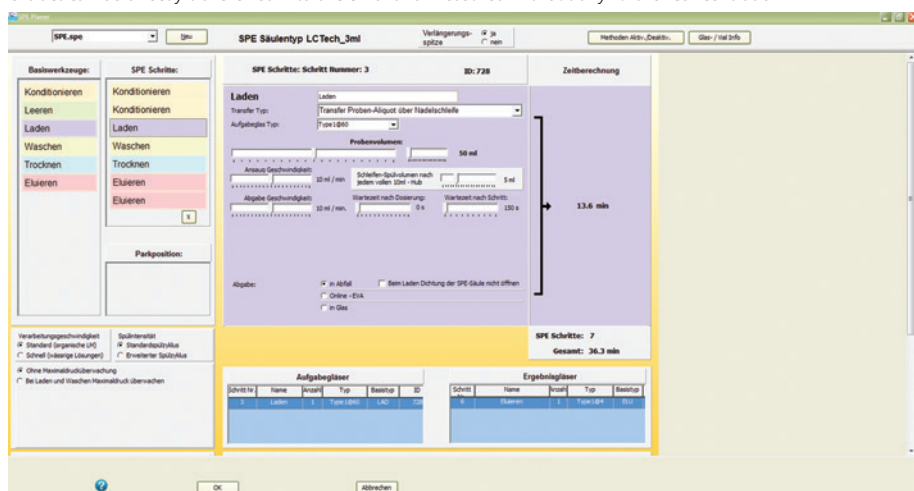
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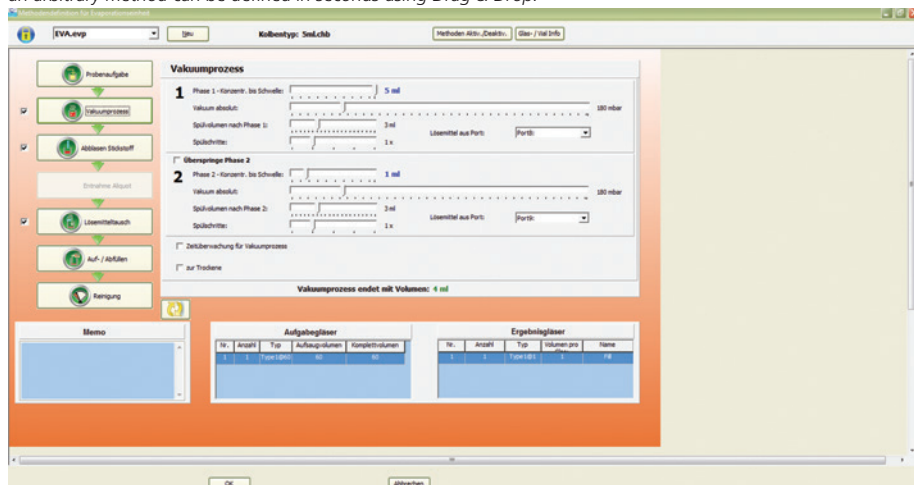
in seconds using Drag and Drop. Within a selected analysis step, parameters, such as volume or velocities, can be adjusted simply by moving bars in the display. "Even the entire concentration process of the sample in the evaporation module can be configured freely within the given conditions," says Baumann. Once a method is saved, it can be routinely called up with a few mouse clicks by any laboratory staff member in order to set up a sample list. "It is important to us that all sample transfers are documented and so, if in doubt, can be traced back," explains Brandsch.



The sample weight is matched to the sensitivity of the GC-MS systems, such that an aliquot of the purified sample extract can be directly transferred into a GC vial and measured without any further concentration.



In the FREESTYLE™ system, the user can set up the required processing methods. The basic method structure for an arbitrary method can be defined in seconds using Drag & Drop.



Within a selected analysis step, parameters, such as volume or velocities, can be adjusted simply by moving bars. Even the entire concentration process of the sample in the EVaporation module can be configured freely within the given conditions – this is an option that did not exist before in automated sample preparation.

Better clean-up with less material

For solid phase extraction, the Nowak Institute uses the LCTech Elufix™ pre-packed glass columns filled with 2 g Florisil and a layer of sodium sulphate.

The columns can be easily removed from the packaging and inserted into the FREESTYLE™. The manual on-site cleaning and filling of reusable columns is consequently fully eliminated. "Through the tighter packing of the same amount of material in the ready-to-use columns, a better cleaning effect is achieved than with using the hand-filled columns. In addition, a sharper separation between the target analyte and contaminants is obtained," says Brandsch. As a result, larger fluctuations no longer occur. In addition, the sodium sulphate ensures that no residual water enters the Florisil column, whereby the reproducibility is further increased.

"LCTech examines each Florisil batch for its quality in accordance with the ISO method and the result is documented with a batch certificate, which makes the columns also suitable for certified laboratories," says Baumann. Instead of a comparatively narrow Luer outlet, a large column outlet with a nearly 5 mm diameter ensures that the eluate is discharged freely and quickly. However, a reusable adapter can be employed for the use of a Luer connection should this be required.

The Elufix™ columns are available with 1, 2, 4 or 10 g activated Florisil and 1 or 2 g anhydrous sodium sulphate. "We will also fill the columns according to individual customer specifications," says Baumann. Compared to plastic, the use of glass as an inert material benefits the process and many other applications in that it excludes both interaction with the analyte and other contaminations. For example, the release of impurities from plastic such as phthalates is avoided.



The sample extract is placed into the FREESTYLE™, then loaded fully automatically onto the pre-packed Elufix™ column and eluted in the evaporation unit. Here, the volume is reduced to an end volume as defined in the method and then transferred into a sample vial.



The Elufix™ columns are available filled with 1, 2, 4 or 10 g activated Florisil and 1 or 2 g anhydrous sodium sulphate. Manual cleaning and filling of reusable columns can now be omitted owing to the use of the LCTech system.

Constant sample throughput

Meanwhile, automation and the use of pre-packed columns achieve a constant sample throughput by the Institute. Not only nights, but also weekends and holidays can be easily included as available productive time to avoid falling behind with the sample processing. "Now we are in a position to analyse 100 to 150 samples per week. Previously, in order to achieve this, a member of staff would have had to work exclusively on sample preparation all day long," says Brandsch. Now, the lab staff can do the sample preparation alongside more demanding tasks, such as measurements and evaluations.

In the meantime, the Institute uses the ready-to-use glass columns for various analytical methods that require a column mediated clean-up in addition to their use in the FREESTYLE™. Columns are available including the inert materials. Alternatively, "self-fill" glass columns are offered in two sizes - 105 and 180 mm - where manual cleaning is also omitted. Suitable filling materials include Florisil and various other sorbents, such as alumina or silica gel.



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"With the FREESTYLE™ and ready-to-use columns, we can analyse 100 to 150 samples per week. Previously, in order to achieve this, a member of staff would have had to work exclusively on sample preparation all day long," says Dr. Thomas Brandsch, Head of the Residue Analysis Department at the Dr. Nowak Institute.



"LCTech examines each Florisil batch for its quality in accordance with the ISO method and the result is documented with a batch certificate, which makes the columns also suitable for certified laboratories," explains Michael Baumann, LCTech General Manager.

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The Dr. Nowak Institute was founded in 1971 by Dr. Karl-Ernst Nowak as the Limnological Laboratory in Fischerhude near Bremen.

Today, the family business that is now led by the second generation extends its service offering into the fields of environment and food and feed.

Around 40 employees work currently at the Dr. Nowak Institute, which is divided into various departments including the areas of wet chemistry, heavy metal analysis, residue analysis, microbiology, ecotoxicology, aquatic ecology and applied limnology. In the residue analysis department, for example, water, sediment, soil, waste and air are analysed in accordance with the current standardized and accredited procedures. Amongst others, samples are tested for priority substances in accordance with the EU-WFD for PAH, PCBs and other organochlorine compounds, such as chlorine alkanes and chlorophenols. They are also tested for volatile hydrocarbons, such as BTX, VOC and vinyl chloride, as well as for perfluorosurfactants, polybrominated diphenyl ethers and plant pesticides or treatment agents, such as AMPA, CKW and glyphosate. Samples and test items are not only analysed but also evaluated in the context of existing legislation or client-specific requirements.

The Institute, which is located in Ottersberg, focuses mainly on samples from northern Germany, however, the Institute also processes specialist samples from all over Germany and around the world.

The LCTech GmbH has headquarters in the Bavarian Dorfen, where products and methods for the preparation and analysis of food, feed and environmental samples have been developed and distributed since 1998. The product range includes semi- and fully automated sample preparation systems and consumables for the analysis of contaminants and residues. The company, which has over 40 employees, is represented through an extensive distributor network worldwide and acts as exclusive distributor for PICKERING Laboratories in Europe, Africa and the Arab countries. Numerous public and private laboratories in food and feed analysis, pharmaceuticals and research are among the customers of LCTech.

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