Novel Automated Sample Clean-up: First Comprehensive Evaluation of Different Filtration Devices for x-y-z Robots

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Abstract

In our study we evaluate the available filtration options for x-y-z robots and some not-yet commercially available filtration devices prototypes with respect to, e.g., filtration efficacy, capacity, ease of application, and compare them to manual (offline) filtration devices such as syringe filters. We support the results by UV and particle size analysis prior and after filtration to showcase the performance of the individual filtration option. Furthermore, the filtration step of routine analysis of plant protection active substances is incorporated in a fully automated workflow of a world-first Thermo Fisher Scientific™ TriPlusRSH SMART expanded autosampler attached to a GC-FID and GC-FID-MS instrument. Amongst the active substances evaluated are Glyphosate and Fosethyl-Al. In total five different filtration options were tested: syringe filters, Thomson filter vials, ITSP-solutions filter cartridges (only available upon special request) and PAL System µSPE filter cartridges. The latter are prototype materials which are currently being introduced to the market.

Introduction

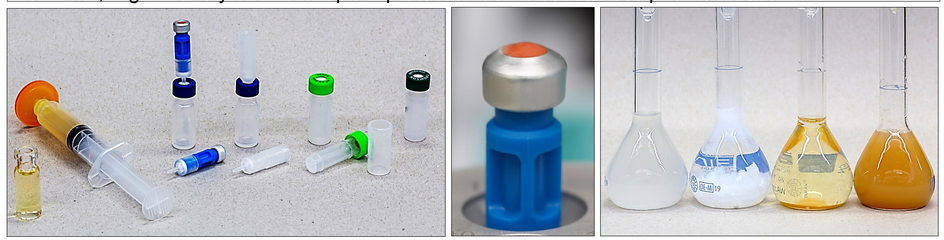
Sample extraction steps, in particular the liquid extraction steps, deliver raw extracts, which carry a large amount of matrix as well. For subsequent chromatographic analysis, the raw extracts need mandatory clean-up from co-extractives and particulate matter. Moreover, apparently clean solutions might carry (solid) impurities which must be extracted prior to analysis, as they might lead to column clogging and high backpressure in HPLC or IC.

Numerous well-established manual sample clean-up methods are available today. Most of them are easily transferable to autosamplers. However, demanding tasks like filtration are more complex to properly automate. Their introduction in the sample treatment workflows carries risks which need to be addressed carefully in terms of analytes retention, sample contamination, additional consumables, time and costs.

Results

All tested filter devices are fully compatible on x-y-z autosamplers. However, the requirements differ in terms of additionally needed hardware parts, which are most in case of syringe filters and filter vials. µ-SPE filters are least demanding in space and equipment.

Graph 1: Left: Depiction of the tested filter devices (syringe filter, ITSP's filter, CTC's µSPE-filter and Thomson filter vials from Thomson and Verex (Phenomenex); from left to right); Middle: Blown-up ITSP-filter; Right: variety of different plant protection active substances prior to filtration.



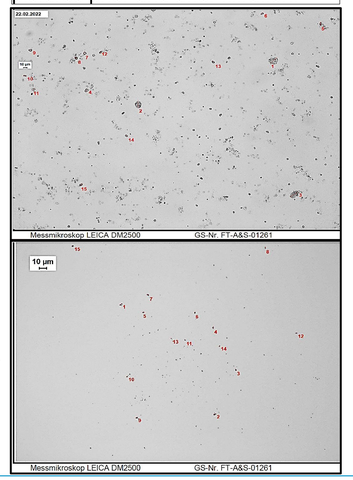
The first test was the filtration efficacy which is best for the syringe filters, as they have the largest filter cross section. The filtration effect of the other devices is comparable and sufficient, as can be seen exemplary in graph 3 for the CTC µ-SPE filters. Due to the high filtration rate, it was not possible to obtain meaningful results by particle size measurements with a Zeta-sizer. Additionally, we conducted turbidity measurements by UV-vis spectroscopy and could semi-quantitively prove the results obtained by the optical light microscope investigations.

A major concern in the application of the different filtration devices is the pressure build-up during the filtration process. In case of the syringe filter, the back-pressure is comparably low due to the large cross section. For the cartridges and filter vials things change dramatically as particles tend to clog the filter and even blow out of the septum (c.f. Graph 1, middle). The sealing by the autosampler syringe is not good in this case.

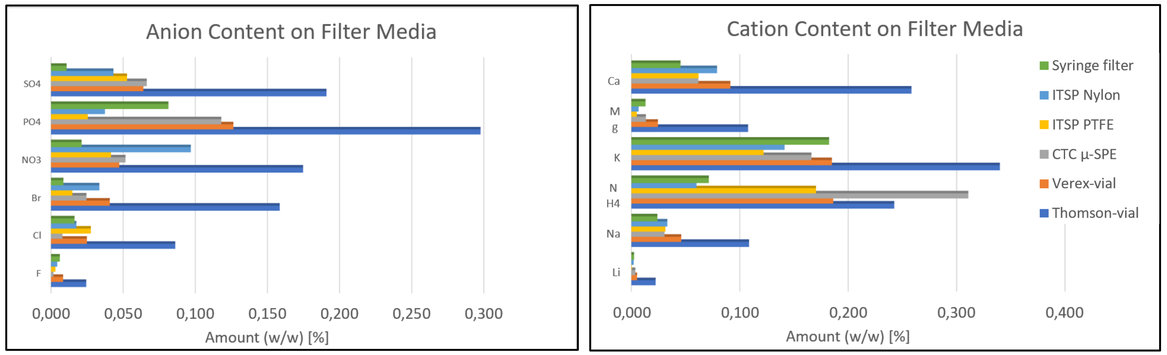
Graph 2: World-first Thermo Fisher Scientific™ TriPlusRSH SMART expanded autosampler attached to a GC-FID and GC-FID-MS instrument.

The newly invented CTC µ-SPE cartridges overcome the back-pressure issue by allowing to employ a pressure of up to 10 bar for sealing of the device. This is advantageous as also the filter vials fail to withstand even low pressures. Moreover, plant protection products are most often high particulate solutions, c.f. Graph 1, right, which would cause higher pressures. Sealing and handling by the autosampler is even worse compared to ITSP-cartridges.

Graph 3: Optical light microscope pictures prior and after filtration.



Graph 4: Comparison of the initial anion and cation payload of the tested filter materials. Those interfere with the analysis of Glyphosate and Fosethyl-Al, as they coelute on the ion chromatogramm.



By investigation of the filtered solutions, we were observing some additional signals in the ion chromatogram, taken by an ICS 1100 system from Thermo Fisher Scientific, which we could relate to an initial "contamination" of the filtration devices. We tested those ions by using a commercially available anion and cation standard. This ready to use standards were filtered through the individual filtration devices. Finally, the "blank" value was subtracted, and the contamination calculated. In total the highest levels of ions were observed the Thompson filter vial with a maximum of 0.3 % (w/w).The other materials showed far less "payload" showing the highest levels for phosphate and ammonium ions.at levels around 0.1 to 0.2% (w/w).

This contaminants can be dealt with in terms of introducing a washing step of the filter in case of the syringe filters, ITSP solution and CTC µ-SPE filter devices. However, filter vials do not allow for such a washing or priming step as they are only designed for a one-time filtration. Moreover, they are limited in the amount of filtrate recovered of about 300µL, which is in some cases insufficient, e.g. when multiple injections from one sample are needed.

Conclusions

Standard syringe filters required the most space on the autosampler due to their large diameter, but delivered the same results compared to their manual use. Thomson filter vials require the same space as filter cartridges, but both require far less space on the instrument The µSPE cartridge convinces in terms of usability and applicability. Contaminant effects could be observed on all the filters, being most prominent on the filter vials. However, syringe filters and µSPE cartridges can easily overcome this with a solvent priming prior to application of the analyte solution. To conclude, the newly designed µSPE filter cartridges were shown to be superior to other well-established filter option available for automation.

Acknowledgment

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