



The Q19: Automated Clean Up For The Analysis of 700 Pesticides In Difficult Matrices Like Tea And Hops

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Tea, tobacco, spices and hops are the most complicated matrices to analyze for pesticides. In our latest work, we developed a fast SPE-based clean up method, where we used a combination of the commercial push-through mini SPE columns [1] and were able to validate this method for 600 + compounds.

In our current work, we are optimizing the sample preparation with our own SPE columns so that we can now analyze 700 pesticides in difficult matrices with an automated clean-up of the QuEChERS [2] extracts. The developments of our own SPE phase are shown on Poster Nr. PD084.

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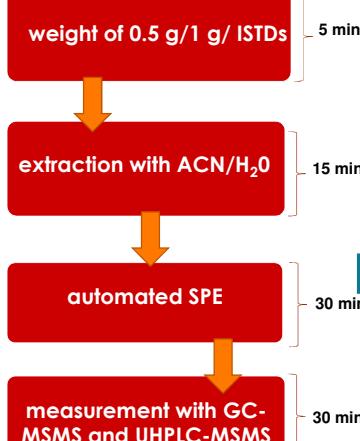
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The new part of our development was the automation of the cleanup of the QuEChERS extracts. Our own poly-layer-SPE columns (see fig. 1) allowed efficient clean up and greater parameter scope than the conventional products. Furthermore we don't need any centrifugation steps anymore. The complete sample preparation is shown at the fig. 1.

We use the LC Tech FREESTYLE SPE module for the automated clean-up, which is more flexible regarding the type of SPE columns than other equipment. Very important was the use of cooled sample racks for the QuEChERS extracts as well as for the cleaned extracts for the measurement. The main single steps of the automated clean up procedure are shown at the fig 2.

Figure 1. The whole sample preparation procedure



The automated sample preparation is much cheaper than all current methods and frees up human resources. The automated cleanup has significantly improved the repeatability of the SPE clean-up. On the other hand it is much slower than the manual method and is only useful in routine work at night or at the vacation time.

Figure 2. Working window (LC TECh) for the programming of the automated sample preparation



Step 0: condition of the SPE column (trays A/B) **5 min**

Step 1: loading of the SPE column (trays A/B) with the prepared QuEChERS extract (tray D), **5 min**

Step 2: SPE procedure and rinsing out of the sample extract in the extract collection 4 ml Vial (Tray C), **15 min**

Step 3: Deviation of the sample extract into the GC- (Tray E) and LC- (Tray F) Vials, **5 min**

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The new clean up method is independent from the human resources, time and is cheaper than all current methods. The same extract can be measured with both kinds of equipment. It is a very good option in the lab, when there is a shortage of human labor or when the number of samples is unexpectedly large. The bottle neck with this method is the standardizing of our own SPE column production and at the relatively low speed of the LC Tech SPE module at this time.

With the clean up on commercial SPE phases, some important pesticide groups like rodenticides and some sulfonylurea herbicides could be only measured with the low recovery rate or were excluded from the scope. With the own SPE column, we can now sure measure these critical compounds.

The new, automated approach was tested and verified with the successful participation in the proficiency tests for tea matrices (FAPAS) and is now in the accreditation procedure. All of our result and also the further developments of our "Q19 method" were published in the recent issue of LABO magazine (Jan/Feb 2018) [3]. We are still optimizing and developing the SPE composition to get the perfect combination of SPE our own and commercially available SPE material.

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[1] A. Romanotto, J. Langner, F. Mütze, Analysis of 600+ pesticides in difficult matrices in 1 hour, 11th EPRW 2016, 24-27 May, Cyprus, Poster presentation

[2] Official collection §64 LFBG: determination of pesticide residues in fruit and vegetables using GC-MS and/or LC-MS/MS after acetonitrile-extraction/distribution and cleaning with dispersive SPE (QuEChERS) (acc. to DIN EN 15662); L 00.00-115; 2014-02

[3] A. Romanotto, J. Langner, K. Speer. Q19 in der Routine: Automatisierte Probenaufarbeitung für das schnelle Pestizidscreening in schwierigen pflanzlichen Matrices, LABO 01-02/2018, www.labode