Automated Clean-up of QuEChERS Extracts for GC-MS and LC-MS

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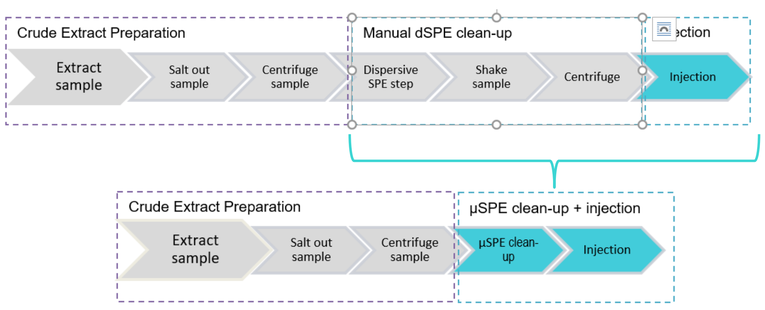
PAL SYSTEM Ingenious sample handling

Introduction

Since the first publication in 2003 the QuEChERS extraction method for pesticides in food (Anastassiades 2003) became a worldwide standard. While the extraction step is well standardized (AOAC 2007.1; CEN 15662), the clean-up of the resulting extracts from different matrices is still in active discussions.

A well proven solution for a unified clean-up for all food matrices with only one cartridge type has been established with the use of µSPE cartridges (Hayward 2016).

Figure 1: µSPE QuEChERS unified extracts clean-up workflow vs. manual dSPE clean-up



The Solution

Unified clean-up using micro solid phase extraction

The application of a mini-column solid phase extraction instead of the dispersive SPE (dSPE) maintains the high potential of the QuEChERS extraction. It allows high sample throughput and recovery for a large number of pesticides in different food matrices even those with high lipid contents (Morris 2014, 2015).

The µSPE clean-up Workflow

Using miniaturized SPE cartridges

The application of µSPE employs miniaturized SPE cartridges in dimensions of 33 mm height x 8 mm diameter. 45 mg Sorbent, dwell volume is approx. 80 µL.

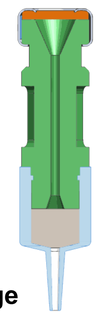


Figure 2: µSPE cartridge

Syringe load and elute

Unlike classical SPE using a vacuum manifold, the flow rates applied to the µSPE cartridge can be precisely controlled with a liquid syringe on a PAL RTC autosampler (Hayward 2016) in the following way:

The µSPE cartridge is sealed by a septum allowing the autosampler syringe to push the raw QuEChERS extracts through the sorbent bed. The syringe works as a LC pump. Low flow rates of approx. 2 µL/s in the load and elution steps are used for sharp analyte/matrix separation.

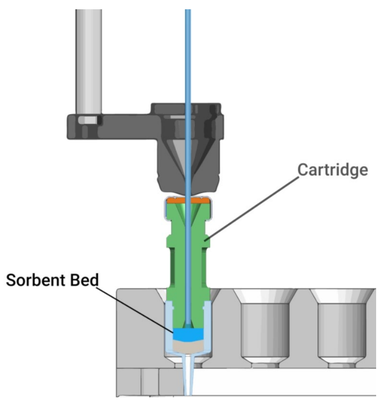


Figure 3: µSPE cartridge with syringe acting as a pump

No concentration step required

The miniaturization of the clean-up step to a microliter scale solid phase extraction (µSPE) prevents the typical dilution by SPE elution thus avoids an additional evaporation step and reduces solvent use.

The pesticides fraction is eluted only in a small volume of a few 100 µL for direct injection into GC-MS or LC-MS.

References

Bruce D. Morris and Richard B. Schriner, Development of an Automated Column Solid-Phase Extraction Cleanup of QuEChERS Extracts, Using a Zirconia-Based Sorbent, for Pesticide Residue Analyses by LC-MS/MS, J. Agric. Food Chem. 2015, 63, 5107−5119, DOI: 10.1021/jf505539e.

Steven J. Lehotay, Lijun Han, Yelena Sapozhnikova, Automated Mini-Column Solid-Phase Extraction Cleanup for High-Throughput Analysis of Chemical Contaminants in Foods by Low-Pressure Gas Chromatography-Tandem Mass Spectrometry, published with open access at Springerlink.com, Chromatographia (2016) DOI 10.1007/s10337-016-3116-y.

M. Hayward, J. Ho, Automated Chromatographic Solid-Phase Extraction Using an Autosampler, American Laboratory (2016) posted online Sep 01, 2016.

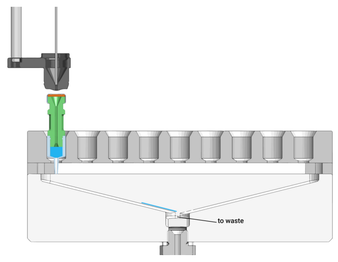
H.-J. Hübschmann, G. Böhm, R. Bolliger, Automated micro-SPE Clean-up of QuEChERS Extracts for Multi-Residue Pesticide Analysis, Poster at the LAPRW 2017, San Jose, Costa Rica, Poster Number: 2071

The Automated Clean-up Workflow

The starting point of the automated clean-up workflow is the centrifuged extract of the standardized QuEChERS extraction.

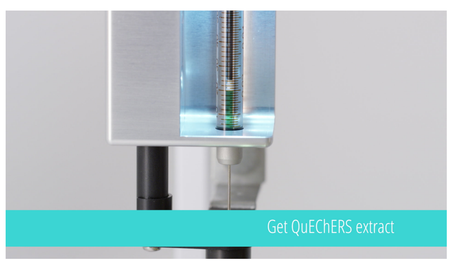
Get conditioning Solvent

Optional



Condition Cartridge

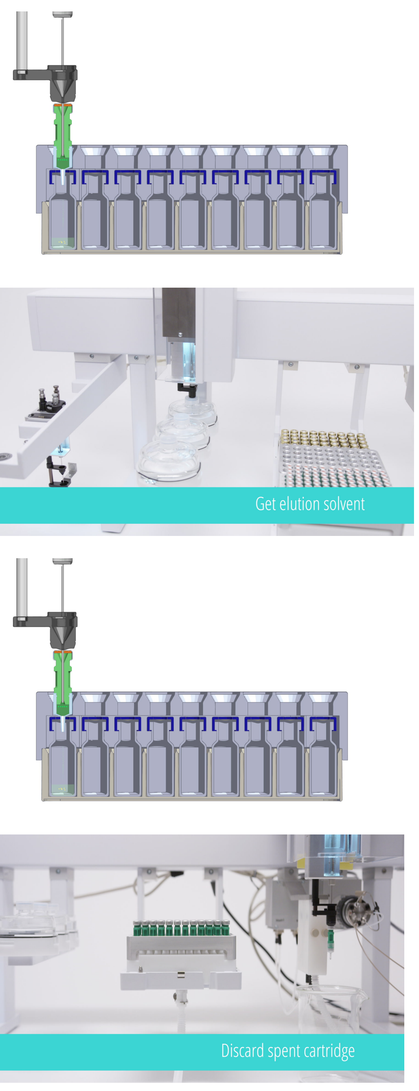
Optional



Get raw extract

150 µL raw extract

Transport cartridge to sample vial



Load sample to cartridge

Load at 2 µL/s

Get elution solvent

Optional

100 µL, 100mM Formate Buffer in MeCN

Elute sample

Optional

Elution at 2 µL/s

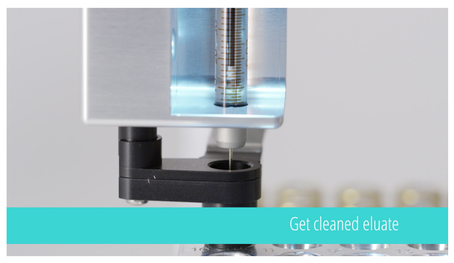
Discard cartridge

Optional

Discard or transport back to cartridge tray

Add 20 µL Analyte Protectant Solution
Add 5 µL ISTD
Add 20 µL MeCN
Mix

Optional



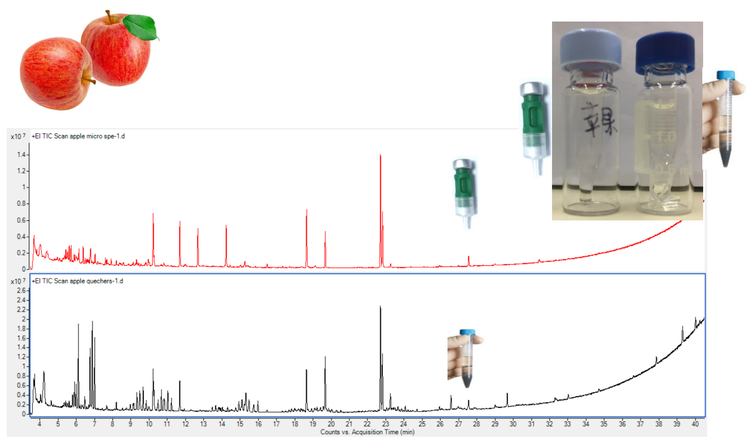
Inject sample

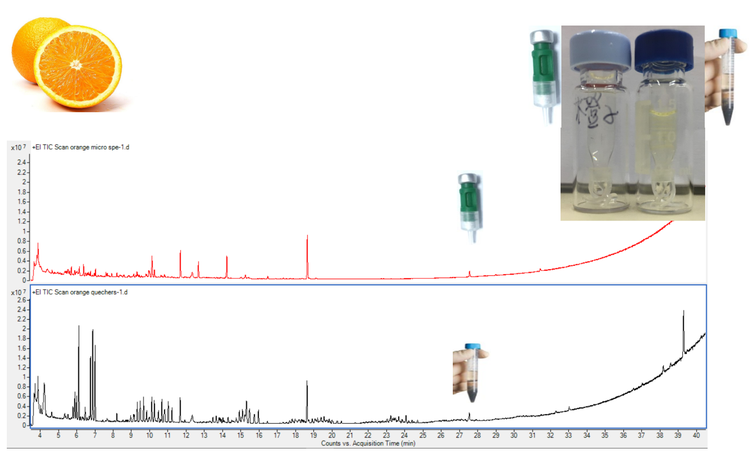
1 µL Sample

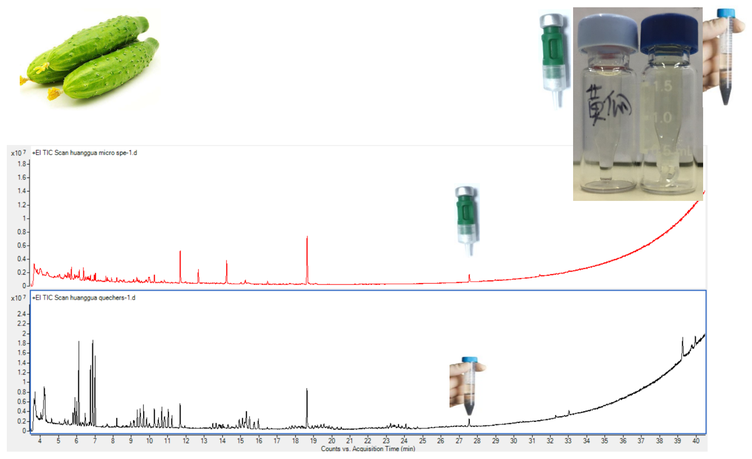
Presented at

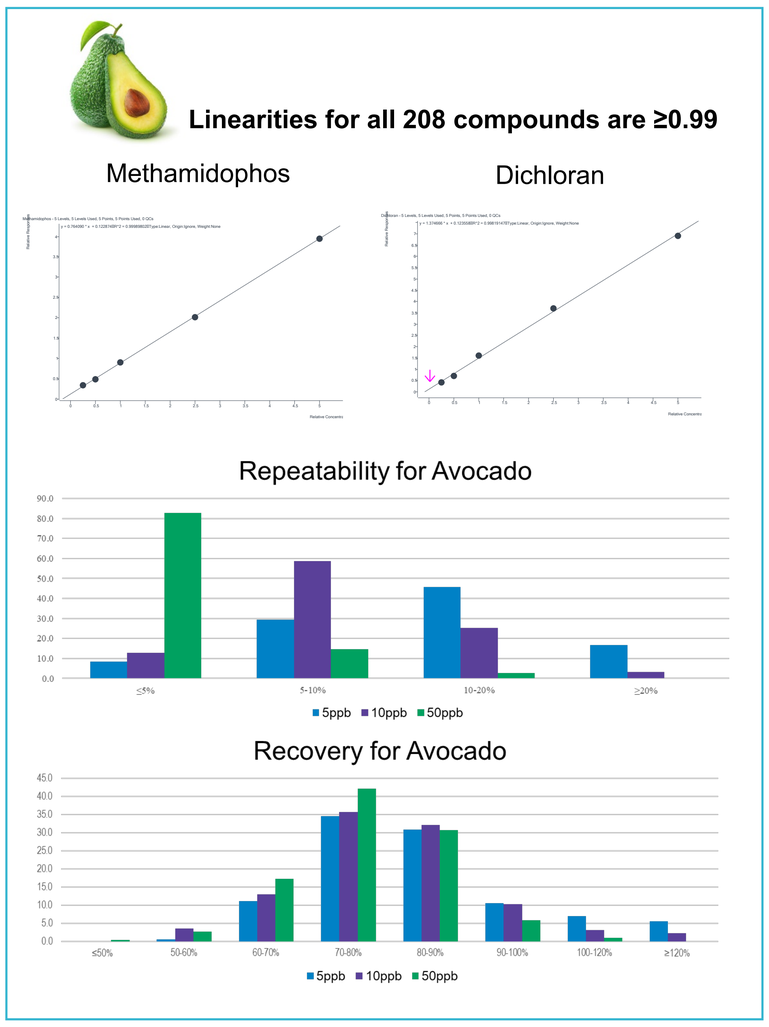
RAFA 2019, Nov. 5-8 2019, Prague, Czech Republic

Reprints of the poster with the full list of references cited will be provided by the author upon e-Mail request to tpreiswerk@ctc.ch









The µSPE Results

One micro-SPE cartridge can serve for all the matrices in food, represented here with simple, complex and high chlorophyll food matrix types.

This clean-up procedure is true Green Chemistry. Significantly less solvent is needed.

Micro-SPE cartridges achieve a significantly better clean-up performance than traditional QuEChERS d-SPE.

The majority of the analyte recoveries are among 70 - 130% with RSDs below 10%, meeting the requirements for pesticide residue analysis in foods.

The automated workflow is highly economical. While the sample sequence is running the PAL starts the preparation of the next sample in parallel to the chromatographic run.

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