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Sample preparation for multi-residue analysis in difficult matrices with an optimized push through SPE

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Introduction

For the analysis of pesticides in food, analytical methods have to be fast and easy, recovering a large number of analytes and they have to be useful in analyzing difficult matrices like tea, hops and spices. With our "Q19" method[1] using combined mini SPE (PSA, NH_2, $MgSO_4$), we developed a processing approach, which is more suitable for complicated matrices than the current § 64 methods of the LFBG, QuEChERS and DFG S19[2,3]. This method is also not as time and solvent consuming as modified QuEChERS[4] . There is always a customers demand for a wider spectrum of analytes. For this purpose we need SPE cartridges for sample preparation which allow a large number of analyte groups to pass through without a reduction of the purification quality. There is only a limited variety of commercially available SPE cartridges. To optimize the clean up procedure of difficult matrices we checked if and which analytes got lost during the sample preparation[5]. In the next step the composition of the used SPE cartridge was optimized.

Methods and Results

We optimized the sample preparation to get clean extracts with a minimum of materials while expanding the spectrum of the analytes. For this purpose we tested which material is responsible for the loss of analytes. After that we combined different commercially available SPE materials to get an optimized SPE cartridge. In addition to the quality of the sample purification, we checked the number of analytes that can be analyzed with the respective composition of the SPE. Every extract was measured by LC-MSMS and GC-MSMS.

1. Influence of certain SPE materials

SPE material	Removed matrix	Retained analytes
Anion exchanger (PSA,NH_2, DEA…)	Sugar, acids	Sulfonylurea, sulfonanilide, coumarine…
Polar materials (SiO_2, diole)	Polar compounds	Sulfonylurea, coumarine…
Non-polar materials (C18, Phenyl…)	Non-polar substances	Thiazole, pyrazole
Carbon (GCB, charcoal)	pigments	Planar analytes, carbamates

2. Testing of diffenerant SPE columns

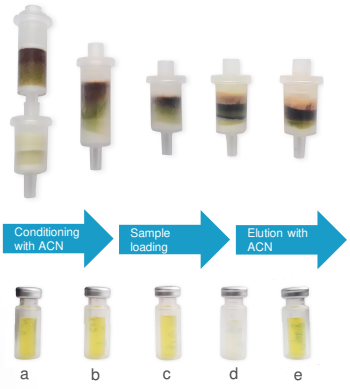


Fig. 1. different SPE modules with their extracts

a) Q19 SPE
b) Q19 selfmade (same composition as (a))
c) ½ Q19 selfmade (½ of (b))
d) PiCA SPE V1 (new composition with GCB)
e) PiCA SPE V2 (optimized composition)

3. Investigation of the sample purity and the analyte spectrum

Fig. 2. matrix profiles LC-MSMS (PCI) and GC-MSMS: Q19 (---) vs PiCA SPE V1 (—) + V2 (—)

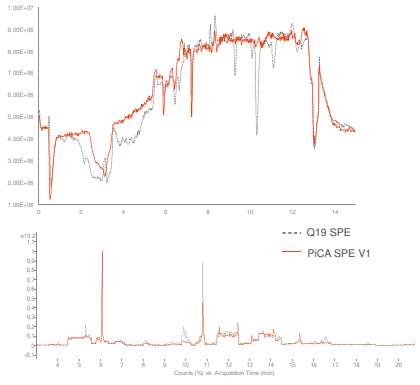
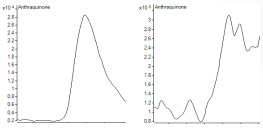


Fig. 3. loss of Antraquinone by using GCB (GC-MSMS)



Purity
No. of analyzed compounds

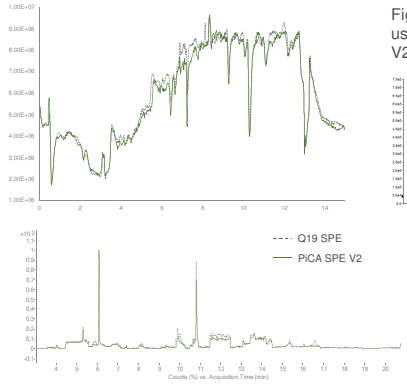
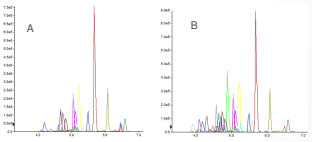


Fig. 4. chromatograms (LC-MSMS) by using the Q19 SPE (A) and the PiCA SPE V2 (B)



Purity
No. of analyzed compounds

Conclusions

In order to optimize the sample preparation regarding purity of the extracts and the number of analytes, we have developed an SPE cartridge which allows a sample preparation resulting in clean extracts with a lower consumption of materials. In addition, we have expanded the range of analytes by > 30 analytes, especially sulfonylureas and rodenticides. In further research we will try to minimize the SPE by reducing the diameter of the cartridges in order to achieve the lowest possible consumption of materials and solvents.

References

[1] 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.labo.de
[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] Official collection §35 LFBG: modular multi-method to determine plant protection substances residues in food (extended new version of DFG method S19), L 00.00-34
[4] Multi-residue Pesticide Analysis in Green Tea by a Modified QuEChERS Extraction and Ion Trap GC/MSn Analysis
[5] J. Langner, A. Romanotto, Optimized Sample Preparation for Multi-residue PesticideAnalysis in Difficult Matrices, poster presentation; EPRW 2018

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