Optimized Sample Preparation for Multi-residue Pesticide Analysis in Difficult Matrices

Langner, J., Romanotto, A.
PiCA GmbH, Rudower Chaussee 29, 12489 Berlin Germany

**Introduction**
Producers or consumers becoming increasingly concerned about the accurate control of pesticides and other undesirable substances in foodstuffs. Analytical methods have to be fast, easy, recovering a large number of analytes and has to be useful in analyzing difficult matrices like tea, hops and spices.

With the "Q19" method\(^1\) we developed a processing approach, which is more suitable for complicated matrices than the current § 64 methods of the LFBG, QuEChERS and DFG S19\(^{2,3}\) and which is not as time and solvent consuming as modified QuEChERS\(^4\).

After a QuEChERS extraction with sample weights of 0.5-1.0 g, there is a cleanup with a combined mini SPE (PSA, NH\(_2\), MgSO\(_4\) from Agilent and UCT). The resulting extract can be analyzed by LC and GC . With this method, which is validated in accordance with the SANTE criteria\(^5\) we can analyze more than 600 analytes in less than 1 hour.

**Methods and Results**
We optimized the sample preparation to get clean extract with a minimum of materials while expanding the spectrum of analytes. For this purpose we tested a lot of commercially available SPE materials and we checked how analytes got lost during the sample preparation.

1. 2. Miniaturization of the SPE column

Fig. 2. different SPE modules with their extracts (green tea)
(a) Q19-SPE
(b) PiCA-SPE with the same composition as (a)
(c) minimized PiCA-SPE with ½ of the materials in (b)

NH\(_2\), MgSO\(_4\)/PSA

Fig. 3. matrix profiles by post column infusion for Q19-SPE (a) and for miniaturized SPE (c)

2. Optimization of the SPE column

Fig. 4. SPE modules with their extracts (black tea)
(a) Q19-SPE
(d) optimized PiCA-SPE with new composition

similar purity of samples

Fig. 5. matrix profiles by post column infusion for Q19-SPE (a) and optimized PiCA-SPE (d)

similar purity of samples

3. Investigation of the processing steps

Fig. 1. drying of extract with fine and coarse MgSO\(_4\)

(number of analytes)

3. Optimization of the SPE column

Fig. 5. matrix profiles by post column infusion for Q19-SPE (a) and optimized PiCA-SPE (d)

similar purity of samples

number of analytes

In order to optimize the sample preparation regarding material consumption, purity of the extracts and the number of analytes, we identified the steps, in which analytes were lost. Using coarse MgSO\(_4\) for extract drying and combining different SPE materials in an optimized SPE column, we have developed a preparation method 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.

**Conclusion**

We optimized the sample preparation to get clean extract with a minimum of materials while expanding the spectrum of analytes. For this purpose we tested a lot of commercially available SPE materials and we checked how analytes got lost during the sample preparation.

**References**

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. Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed. SANTE /11945/2015