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The Pesticide Explorer Collection – Fast Screening and Quantification of Pesticide Residues Using a Comprehensive LC-MS Solution

Susanne Sales[1], Charles T. Yang[2], Michal Godula[1], [1]Thermo Fisher Scientific, Dreieich, Germany, [2]Thermo Fisher Scientific, San Jose, California, USA

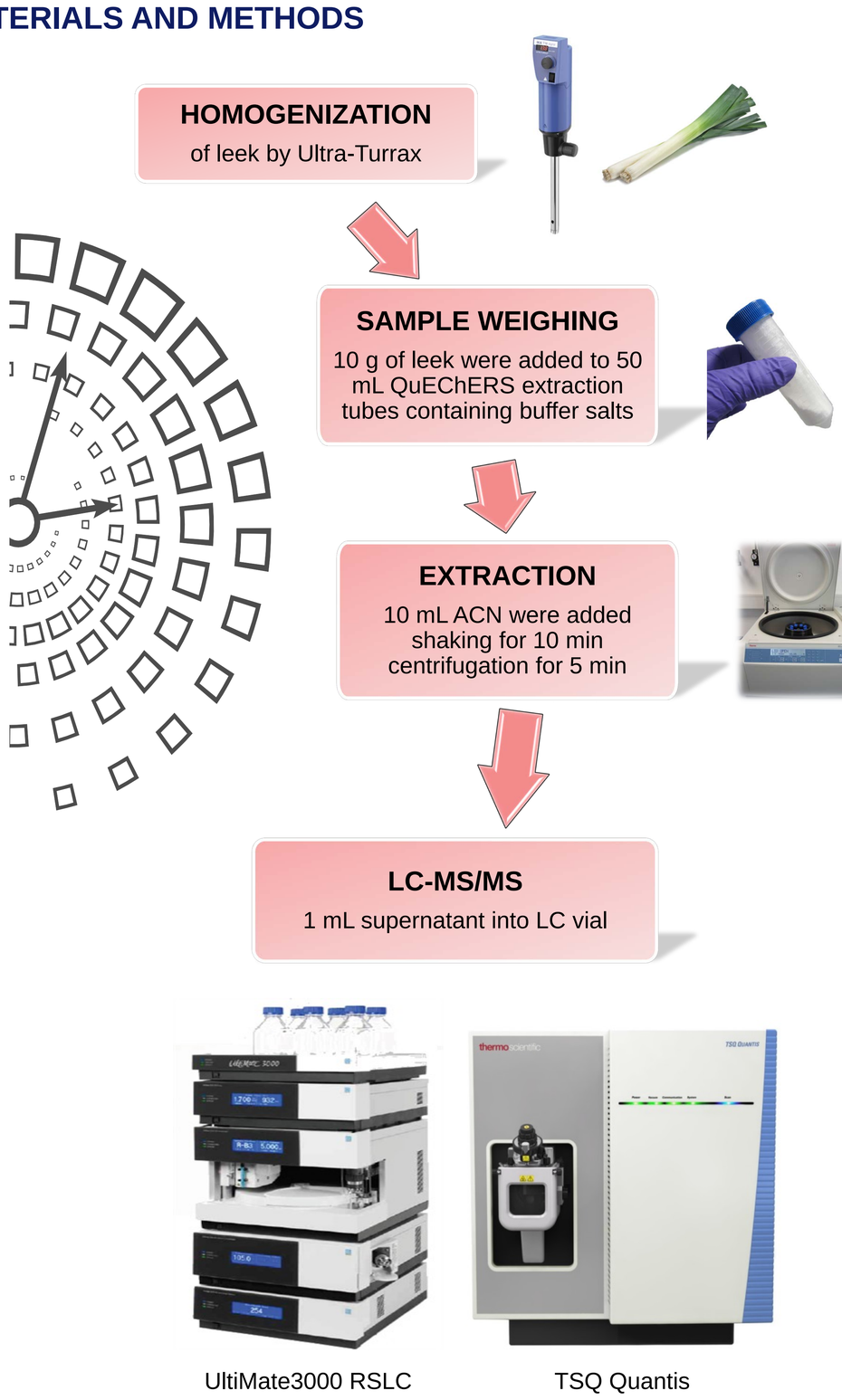
ABSTRACT

Pesticides are chemical or biological agents meant to control, kill or repel plants or animals considered to be pests. Although, pesticides have benefits such as improved productivity and protection of crop losses, some pesticides have drawbacks too such as potential toxicity to humans and other species. – Due to the widespread use of agricultural chemicals in food production and the difficult control of international food chain, people are exposed to low levels of pesticide residues through their diet. Identification and quantification of pesticide residues in foods and food products is therefore an important part of routine food control. Routine pesticide residue analysis remains one of the most challenging tasks in mass spectrometry. Their chemical diversity, the sheer number of potentially used pesticides (>1100), and the wide range of matrices are the key challenges in their analysis. Reliable and validated multi methods using the latest technical developments are therefore required for the analysis of pesticides.

Here we present the Pesticide Explorer™ Collection, a complete workflow solution for the analysis of ~ 600 pesticide residues by liquid chromatography-triple quadrupole mass spectrometry. The analysis was performed on a Thermo Scientific™ UltiMate3000™ RSLC system coupled to a Thermo Scientific™ TSQ Quantis™ mass spectrometer. Chromatographic separation was carried out on a Thermo Scientific™ Accucore aQ™ column. The QuEChERS extraction procedure was applied on all investigated samples. Leek representing vegetable with high pigment content was chosen as matrix for validation.

The method was tested according to SANTE11945/2015 requirements. Analytical parameters such as linearity, specificity, LOD, LOQ, precision and accuracy were evaluated using the Thermo Scientific TraceFinder™ 4.1 software. The validation outcome showed satisfactory results since LOQs ≤ 10 µg/kg were reached for a large number of the ~ 600 target compounds in leek. The RSD values for most of the compounds were lower than 20%.

MATERIALS AND METHODS



UltiMate3000 RSLC

TSQ Quantis

Systems:

Thermo Scientific TSQ Quantis Triple Stage Quadrupole MS coupled to a Thermo Scientific UltiMate3000 RSLC system.

LC conditions:

Column: Accucore aQ, 100 mm x 2.1 mm x 2.6 µm
Mobile phase: A: Water + 5mM Ammonium formate & 0.1% Formic acid
B: Methanol + 5mM Ammonium formate & 0.1% Formic acid
Injection volume: 1µl
Flow rate: 300 µl/min
Column temperature: 25° C
Run time: 15 min

TSQ MS conditions:

Source: HESI
Detection mode: t-SRM (> 1700 SRM transitions)

Data analysis:

Thermo Scientific TraceFinder 4.1 software

RESULTS

XIC Overlay

Almost 600 pesticides (corresponding to approx. 1700 SRMs, polarity switching) were measured in a single 15 min run on a TSQ Quantis Triple Stage Quadrupole MS coupled to UltiMate3000 RSLC system (Figure 1) using carefully optimized gradient profile.

Figure 1. Chromatographic separation of 600+ compound on the Accucore aQ analytical column

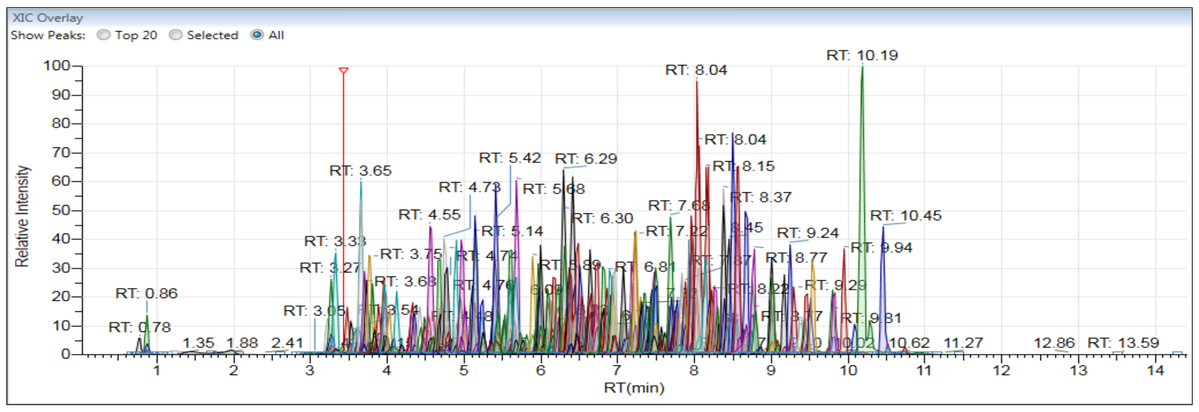


Figure 1 shows an XIC overlay of ~ 600 pesticide residues measured in one run.

Calibration Curve and Related Quantifier and Qualifier Ions

Calibration curves ranging from 100 ppb to 0.5 ppb were performed for all pesticide residues. One quantifier and 2 qualifier ions (where applicable) were measured for each compound. The results for Aminocarb (Figure 2A) and Butocarboximsulfoxide (Figure 2B) are shown.

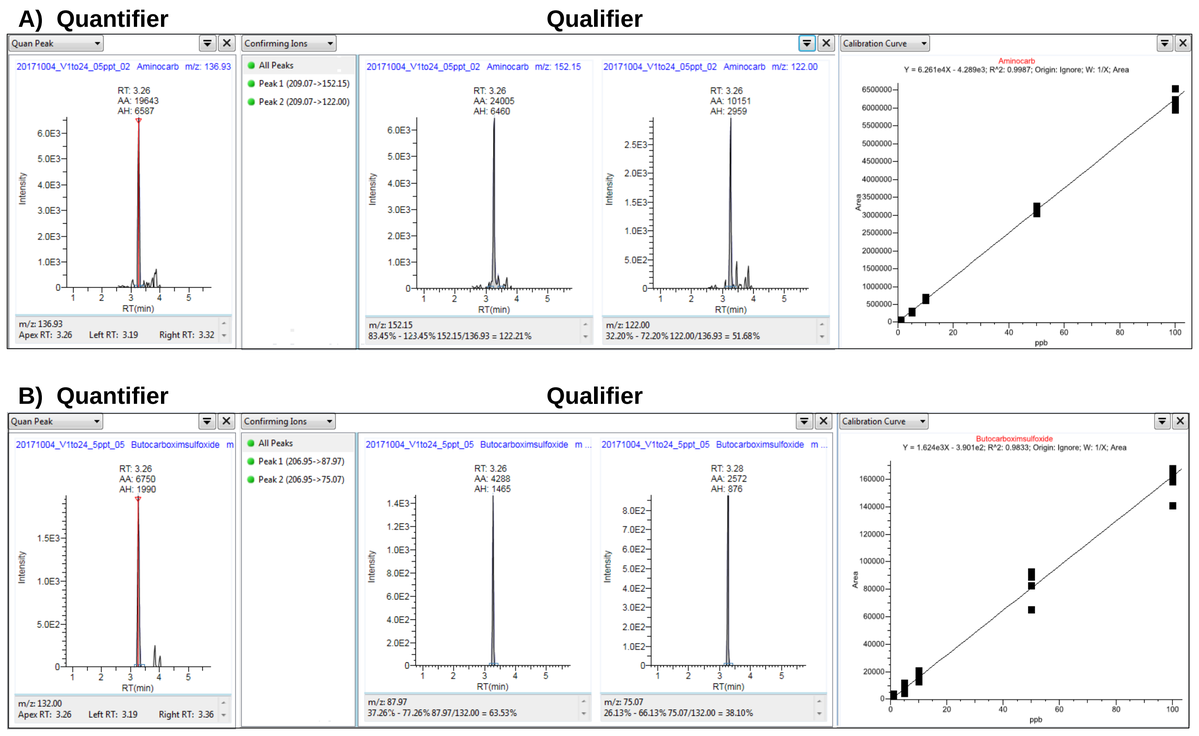


Figure 2. Examples of 2 pesticide residues (Aminocarb (A) and Butocarboximsulfoxide (B)) at LOQ (0.5 mg/Kg and 5 mg/Kg, respectively) and their calibration curves are shown.

LOQs

LOQ values for all compounds measured on the TSQ Quantis™ were determined. Figure 3 shows the % amount of pesticides with LOQs ≤ 10 or > 10 µg/Kg.

- 94 % of the compounds have an LOQ below or equal 10 µg/Kg.
- Only 6 % have an LOQ > 10.
- 2 % of LOQs were above the level of the required MRLs.

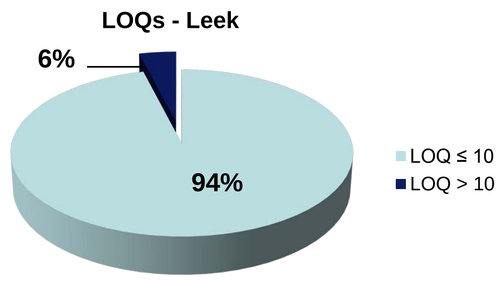


Figure 3. Pie chart showing the % of pesticides with LOQs ≤ 10 or > 10 µg/Kg.

CONCLUSIONS

- The method allows the measurement of ~ 600 compounds in only 15 min.
- The performance for the majority of target compounds complies with current regulatory requirements. Only 2 % of the LOQ values are above the required MRL values.
- The method is available for an easy method transfer.

ACKNOWLEDGEMENTS

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TRADEMARKS/LICENSING

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