

Application of Ultra-Sensitive UPLC/MS/MS in Reducing Matrix Effects of Complex Food Samples During Multi-Residue Pesticide Analysis

GOAL

To utilize the ultra-sensitive detection of pesticides to reduce matrix effects in complex food samples.

BACKGROUND

One of the biggest challenges in ensuring the safety of our food supplies is measuring hazardous ultra-trace components in the presence of a highly complex sample matrix. For the analysis of pesticides in food matrices, the increased use of liquid chromatography systems coupled with tandem quadrupole mass spectrometers has allowed progress in reducing the problems caused by the sample matrix. However, difficulties remain when trying to discriminate against matrix components that exhibit similar physiochemical properties.

Problems caused by matrix can include disruption to chromatography, increased chemical noise, and ion suppression. In complex matrices, such as spices, these problems can be found in combination, making determinations of pesticide residue concentrations very difficult.

In addition to problems caused by the sample matrix, there are also pesticides that by nature are more difficult to analyze using LC/MS/MS due to a poor (relative) response factor. Successful analysis of these compounds to the regulatory concentration limits is difficult when considering the practicality of increasing sample amount and the balance of extracted matrix concentration. A much more practical solution is to use a more sensitive instrument to maximize performance at these required concentrations.

With Xevo TQ-S, ultra-sensitive analysis allows detection of the most difficult compounds and limits the impact of the sample matrix.

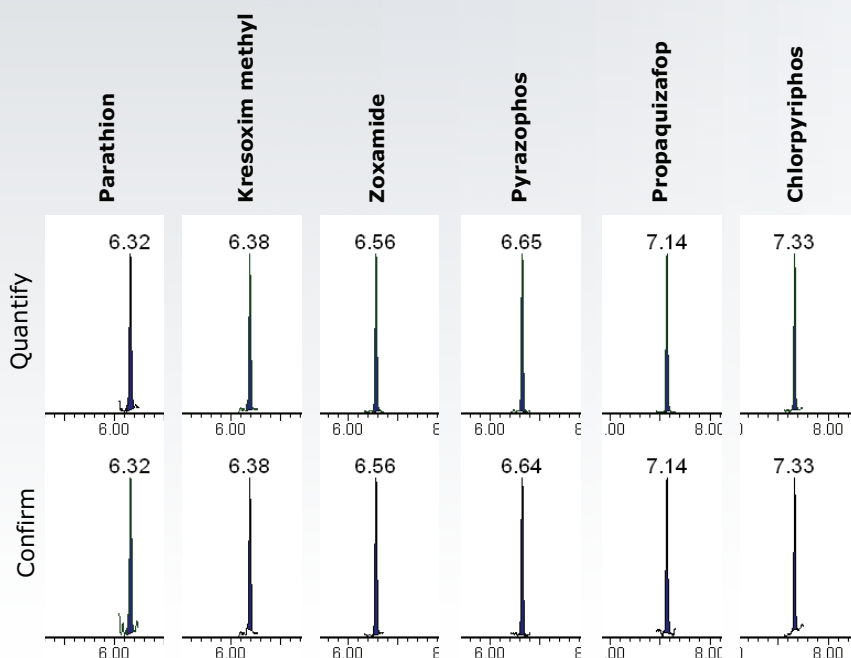


Figure 1. Avocado spiked to 0.005 mg/kg with extracted MRM chromatograms for a selection of pesticides. Both quantitative and confirmatory transitions are detected. This concentration equates to 10 times below the European MRL except zoxamide, which is 4 times below.

In this technical brief, we describe the use of Waters® Xevo™ TQ-S for the ultra-sensitive detection of multiple pesticide residues in food samples, and the application of that sensitivity to matrix reduction strategies.

THE APPROACH

The Waters DisQuE™ Dispersive SPE Kit and ACQUITY UPLC® System, coupled with Xevo TQ-S were applied to the high sensitivity determination of multiple pesticide residues in grape, avocado, marjoram, and ginger. Typical reversed-phase conditions were used for the ACQUITY UPLC separations with formic acid modified mobile phase to aid positive ion electrospray. Multiple reaction monitoring (MRM) mass spectrometer parameters were generated by the Waters Quanpedia™ Database for the simultaneous determination of 86 pesticides. Xevo TQ-S was operated with RADAR™, an information-rich acquisition approach that allows you to track your target analytes with extreme precision in MRM mode, while simultaneously scanning the background for all other components.

For a laboratory to report results around the European Maximum Residue Limits (MRLs) it is imperative that high quality measurements are possible even for the most challenging compounds. Comfortable quantitation and confirmation below target MRLs allow high confidence in the analytical data produced, if samples are encountered that are close to the limit. Figure 1 shows quantitative and confirmatory MRM chromatograms for a selection of pesticides (including some challenging compounds) in avocado spiked at 0.005 mg/kg. This concentration is equivalent to 10 times below the European MRL, except zoxamide, which is four times below.

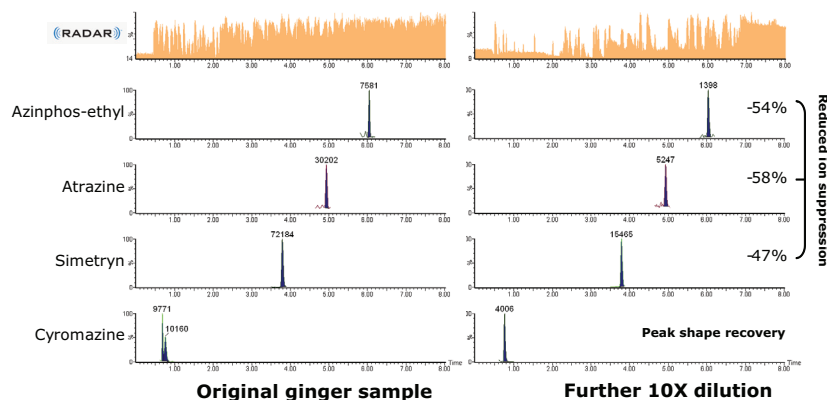


Figure 2. RADAR full scan chromatograms with selected pesticide MRM chromatograms for a ginger sample (0.1 g/mL matrix) at 0.01 mg/kg and a subsequently 10 times diluted sample.

For particularly challenging matrices the mass of the sample taken for extraction can be limited for practical reasons, as well for controlling the complexity of the co-extracted matrix. Some matrices require ten times less sample to be extracted which means that higher sensitivity is required from the analytical system to compensate.

A good example of this problem is the food commodity ginger. The matrix complexity is very high, even when using relatively low matrix concentrations (0.1 g/mL as opposed to the usual 1 g/mL using QuEChERS). The matrix is complex enough that problems with pesticide chromatography and ion suppression can be observed. However, with ultra-sensitive detection, complex samples can be diluted to reduce matrix problems while maintaining detection. Figure 2 shows RADAR full scan chromatograms with selected pesticide MRM chromatograms for a ginger sample at 0.01 mg/kg and a subsequently 10 times diluted sample. Matrix reduction is clearly visible in the RADAR traces and as a consequence, ion suppression is reduced and peak shape recovery is possible.

SUMMARY

The ultra-sensitive pesticide analysis capability of Xevo TQ-S minimizes the impact of more challenging matrices that laboratories may face. This extra sensitivity also allows a better chance of detecting the most challenging compounds in these matrices. In addition, large multi-residue methods are possible without compromising required detection levels. This can ultimately lead to continuous improvement in sample turnaround, quality, and performance that translates to business activities reliant on the laboratory.

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Waters Corporation
34 Maple Street
Milford, MA 01757 U.S.A.
T: 1 508 478 2000
F: 1 508 872 1990
www.waters.com