Multi-Residue Pesticide Analysis in Food Matrices Using Ultivo Triple Quad LC/MS

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AOAC 2017
P-W-044

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Introduction

Pesticides are vital to the success of crop production. Regulatory agencies have set maximum residue levels (MRLs) for hundreds of pesticides and their metabolites in foods. Most MRLs are set at low ppb levels, posing significant challenges to screen and quantify hundreds of analytes in complex matrices simultaneously.

In this presentation, we demonstrate the screening and quantitation for ~250 pesticides and metabolites using Ultivo Triple Quad LC/MS (Figure 1).

Ultivo is designed to address many challenges faced by routine production labs, especially in the environmental and food safety arenas. Innovative technologies within Ultivo allowed us to reduce its overall footprint, while conserving the comparable performance level of much larger MS systems. Innovations, such as VacShield, Cyclone Ion Guide, Vortex Collision Cell and the Hyperbolic Quads, not only maximize quantitative performance in a small package, but also enhance instrument reliability and robustness, which promote greater uptime. Moreover, Ultivo reduces the need of user intervention for system maintenance, making the system operation and maintenance manageable for non-expert users. MassHunter Software simplifies data acquisition, method set up, data analysis and reporting, which results in the fastest possible acquisition-to-reporting time, increasing lab productivity.

Figure 1. Ultivo Triple Quad LC/MS

Experimental

Sample Preparation

~250 pesticides are detected using a dynamic MRM (dMRM) method. Orange, avocado, black tea and broccoli were chosen to represent most fruits, vegetables, and dried herbs. 10 grams of organic orange/avocado/broccoli and 2 grams of organic black tea were extracted with 10 mL of ACN and EN Extraction Salts (5982-5650). High pigment dSPE (5982-5356CH) was used on black tea; modified EMR-Lipid was used on avocado; PSA kit was used on orange (5982-5058), and pigment samples EN dSPE was used for broccoli (5982-5256).

LC and Mass Spectrometer parameters

Table 1. LC Conditions

Column	Eclipse Plus C18 3.0 x 150 mm, 1.8µm	
Column temp	45°C	
Injection volume	2 µL	
Mobile phase	A: Water, 0.5mM NH_4F +4.5mM NH_4 formate+ 0.1% Formic Acid B: MeOH, 0.5mM NH4F +4.5mM NH4 formate+ 0.1% Formic Acid	
Flow rate	0.45 mL/min	
Gradient	Time	B%
	0	2
	0.5	2
	1	50
	4	65
	16	100
	18	100
	18.1	2
Stop Time	20min, Post Time 1.5min	

Table 2. MS Parameters

Drying gas temp	250 °C
Drying gas flow	11 L/min
Sheath gas temp	350 °C
Sheath gas flow	12 L/min
Nebulizer pressure	40 psi
Capillary voltage	3500 V(+); 3500 V(-)
Nozzle voltage	300 V(+); 1000 V(-)
Delta EMV	200 V(+); 200 V(-)
Cycle Time	800 ms

Results and Discussion

Instrument Performance

The signal response and reproducibility (500 injections) were outstanding as illustrated in Figure 2. 500 injections were carried out with a fast 5-min LC gradient.

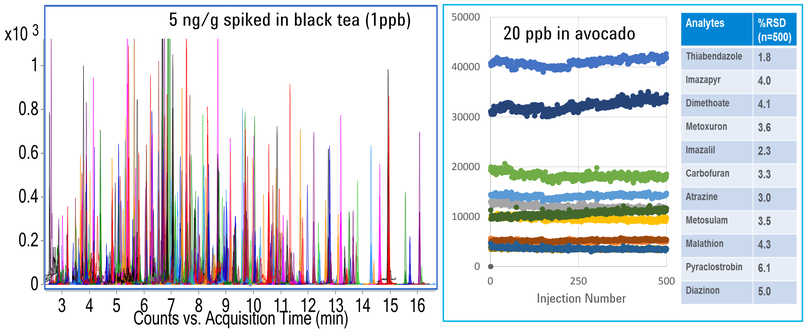

Figure 2. response of 1ppb pesticides in black tea (left) and reproducibility of 20 ppb pesticides in avocado (right)

Sensitivity and Precision

Most of the compounds could be detected at 1 ppb in matrices (corresponding 5 ng/g for black tea) with accuracy of 80-120% for at least 4 replicates. Additional analytes could be detected at higher concentrations (Figure 3). The precision was excellent, with %RSD less than 10% for most of the compounds at the lowest concentration of quantitation.

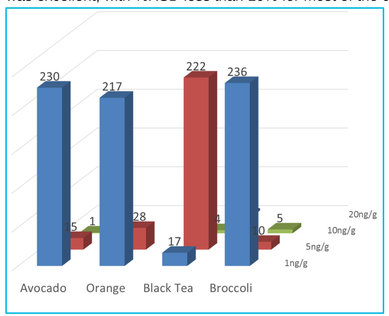

Figure 3. Outstanding sensitivity: Most of compounds could be accurately detected below MRL (1ppb in matrices). Additional compounds could be detected at higher concentrations.

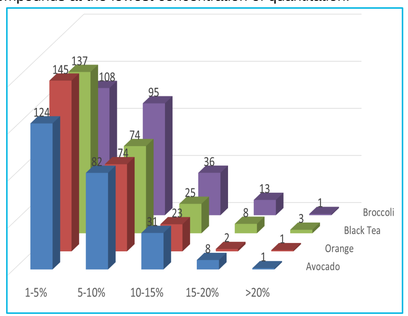

Figure 4. Excellent precision: most of compounds had %RSD <10% at the lowest quantitation concentration (n=6) without any outlier rejection.

Results and Discussion

Real World Samples Analysis: Non-Organic Orange, Avocado and Broccoli

Non-organic orange, avocado and broccoli were processed as organic matrices. Most of the calibration curves had R^2 > 0.99, allowing accurate quantitation of samples. No pesticides could be detected in avocado, while 3 and 7 pesticides were detected in orange and broccoli respectively (Figure 5).

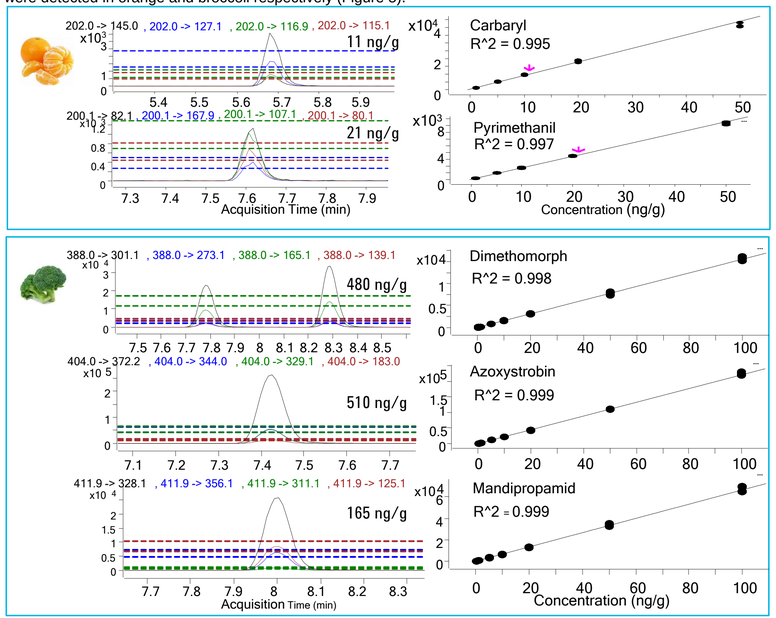

Figure 5. Selected examples of pesticides detected in non-organic orange and broccoli

Conclusions

- Ultivo Triple Quad LC/MS delivers the ultimate performance of an analytical instrument with a minimized footprint.
- Technological innovations within Ultivo afford optimal sensitivity, robust detection and easy maintenance; thereby improving productivity and confidence in results.
- Ultivo provides significant advantages in routine production testing laboratories with enhanced capabilities for non-expert LC/MS users.
- Agilent total workflow solutions that include sample preparation, databases, methods and reporting facilitate fast method development and validation in food safety and environmental analyses.