MULTIRESIDUE PESTICIDES ANALYSIS IN FOOD MATRICES

Agilent Ultivo Triple Quadrupole LC/MS System

Figure 1. Agilent Ultivo Triple Quadrupole LC/MS.

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 part per billion (ppb) levels, posing significant challenges to simultaneously screen and quantify hundreds of analytes in complex food matrices.

Here, we demonstrate screening and quantitation for 246 pesticides and metabolites using an Agilent Ultivo Triple Quadrupole LC/MS. The Ultivo is designed to address many challenges faced by routine production labs, especially in environmental and food safety arenas. Innovative technologies within Ultivo allowed us to reduce its overall footprint, while maintaining the performance levels 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, promoting greater uptime. Ultivo reduces user intervention for system maintenance, making system operation and maintenance manageable for nonexpert MS users. Agilent MassHunter Software simplifies data acquisition, method setup, data analysis, and reporting, resulting in the fastest possible acquisition-to-reporting time, thereby increasing lab productivity.

For more information, visit:
www.agilent.com/chem/Ultivo

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Methodology

Sample preparation

There were 246 pesticides detected in matrices using a dynamic MRM (dMRM) method. Orange, avocado, and black tea were chosen to represent most fruits, vegetables, and dried herbs. Ten grams of organic orange/avocado and 2 g of organic black tea were extracted with 10 mL of ACN and EN Extraction Salts (p/n 5982-5650). Dispersive SPE (dSPE) for high pigment (p/n 5982-5356CH) was used on black tea; modified EMR—Lipid was used on avocado, and a PSA-containing kit was used on orange (p/n 5982-5058).

LC and MS parameters

LC parameters	
Column	Agilent Eclipse Plus C18, 3.0 × 150 mm, 1.8 µm
Column temperature	45 °C
Injection volume	2 µL
Mobile phase	A) Water, 0.5 mM ammonium flouride + 4.5 mM ammonium formate + 0.1 % formic acid B) MeOH, 0.5 mM ammonium flouride + 4.5 mM ammonium formate + 0.1 % formic acid
Flow rate	0.45 mL/min
Gradient	Time (min) %B 0 2 0.5 2 1 50 4 65 16 100 18 100 18.1 2
Stop time	20 minutes
Post time	1.5 minutes

MS parameters	
Drying gas temperature	250 °C
Drying gas flow	11 L/min
Sheath gas temperature	350 °C
Sheat gas flow	12 L/min
Nebulizer pressure	40 psi
Capillary voltage	3,500 V(+); 3,500 V(–)
Nozzle voltage	300 V(+); 1,000 V(–)
Delta EMV	200 V(+); 200 V(–)

Results and Discussion

Instrument performance

Technological innovations within Ultivo lead to efficient ion transmission and selection, and excellent fidelity of ion fragmentation in the collision cell. Figure 2 illustrates the outstanding signal responses of 246 pesticides in black tea matrix below MRL.

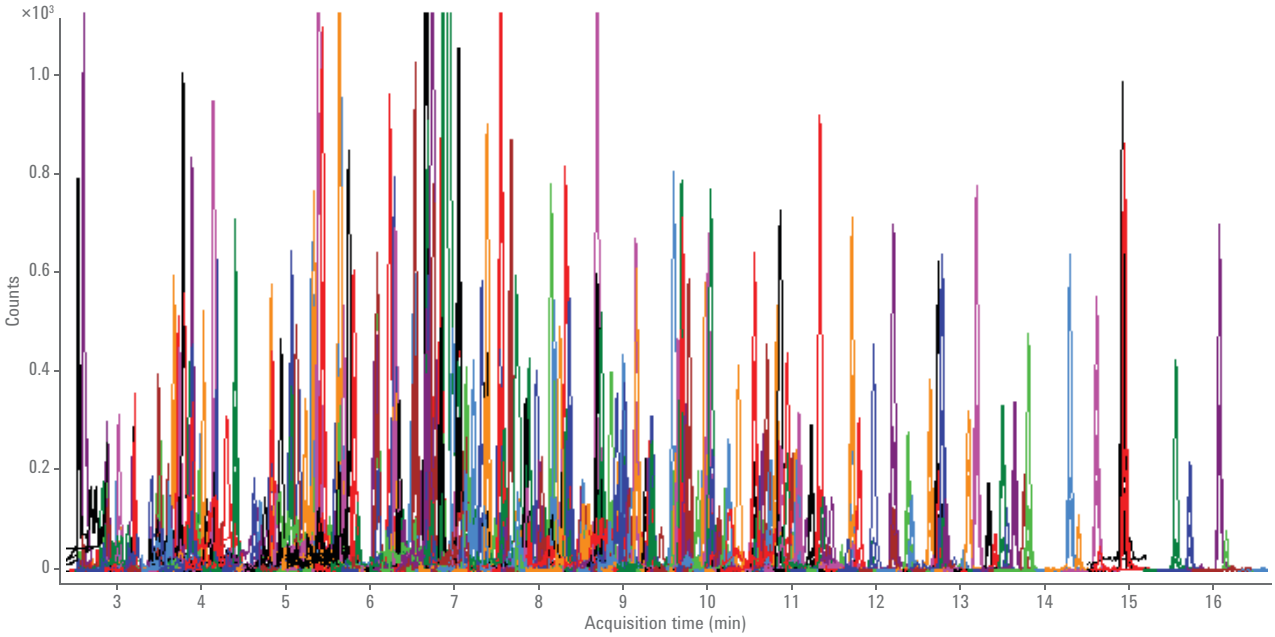

Figure 2. The signal response of the Agilent Ultivo Triple Quadrupole LC/MS for 246 pesticides spiked in black tea matrix at 5 ng/g (1 ppb).

Sensitivity and precision

Most of the compounds could be detected below MRLs, with accuracies of 80–120 % for at least four of six replicates as shown in Figure 3. In orange and avocado matrices, majority of analytes could be accurately quantified at 1 ng/g, with additional analytes detected at higher concentrations. In black tea matrix, majority of the analytes could be accurately quantified at 5 ng/g. Figure 4 shows that the precision was excellent, with %RSDs less than 10 % for most of the compounds at the lowest quantitation levels.

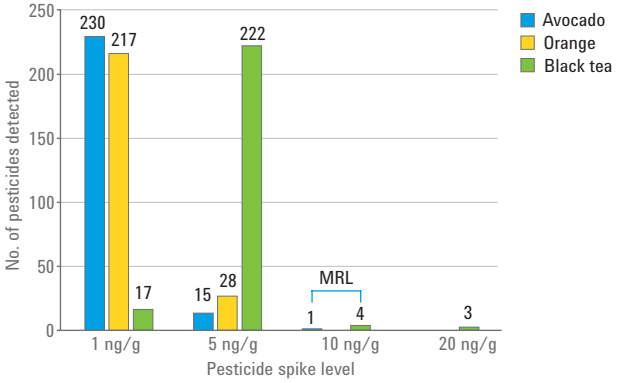

Figure 3. Outstanding sensitivity: most compounds could accurately be detected below MRLs (one compound was not detected in orange due to matrix interference).

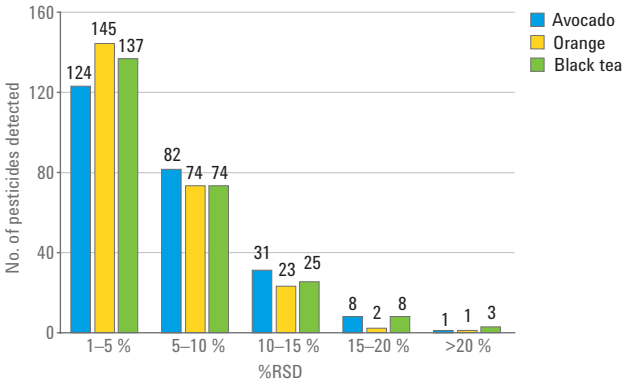

Figure 4. Excellent precision: most compounds had %RSD less than 10 % at the lowest quantitation level (n = 6) without any outlier rejection.

Real world samples analysis: nonorganic orange and avocado

Nonorganic orange and avocado were acquired from a local market and 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 nonorganic avocado, while three pesticides were detected above MRL in nonorganic orange (Figure 5).

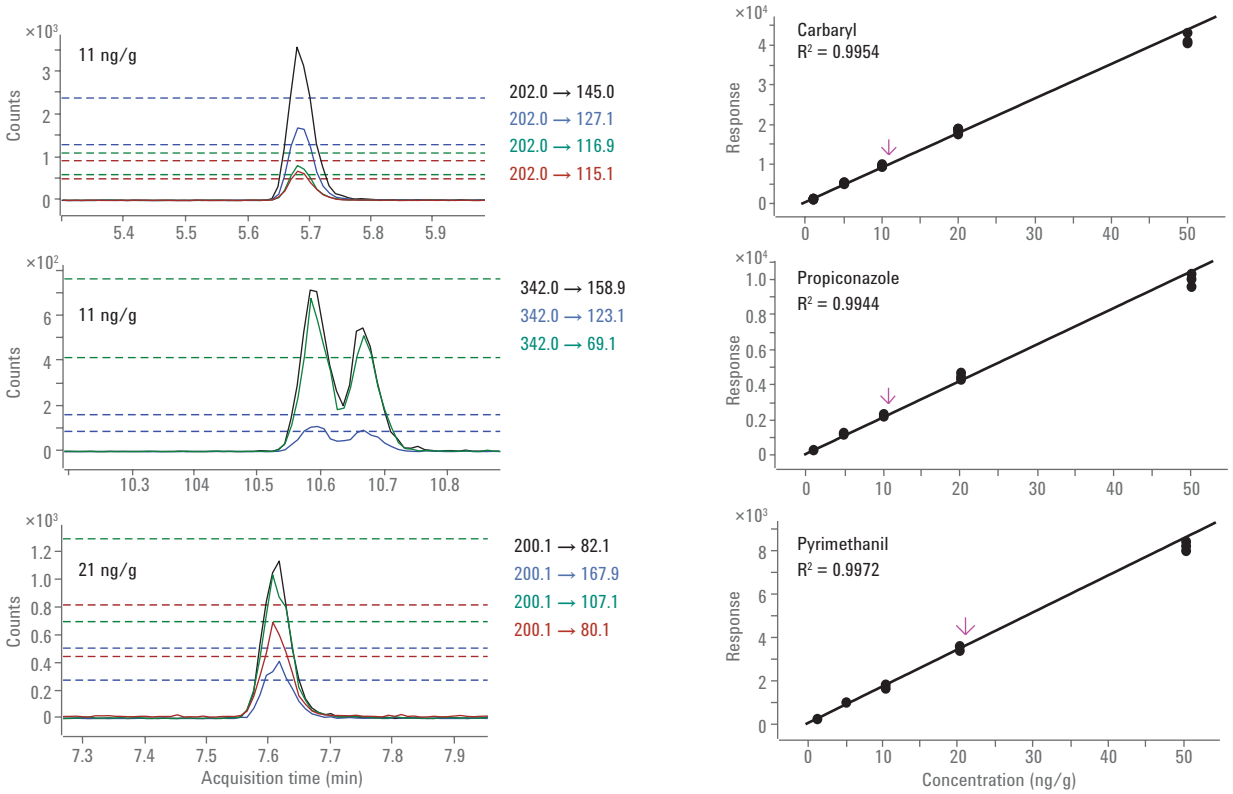

Figure 5. Pesticides detected above MRL in nonorganic orange.

Conclusions

- Agilent Ultivo Triple Quad LC/MS delivers the ultimate performance of an analytical instrument with a minimized footprint.
- Technological innovations within Ultivo ensure 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 nonexpert LC/MS users.
- Agilent offers total workflow solutions that include sample preparation, databases, methods, and reporting facilitate fast method development and validation in food safety and environmental analyses.

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