

Multi-Residue Pesticide Analysis in Food Matrices Using Ultivo Triple Quad 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 ppb levels, posing significant challenges to screen and quantify hundreds of analytes in complex food matrices simultaneously.

In this presentation, we demonstrate the screening and quantitation for 246 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 for user intervention for system maintenance, making the system operation and maintenance manageable for non-expert MS 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.

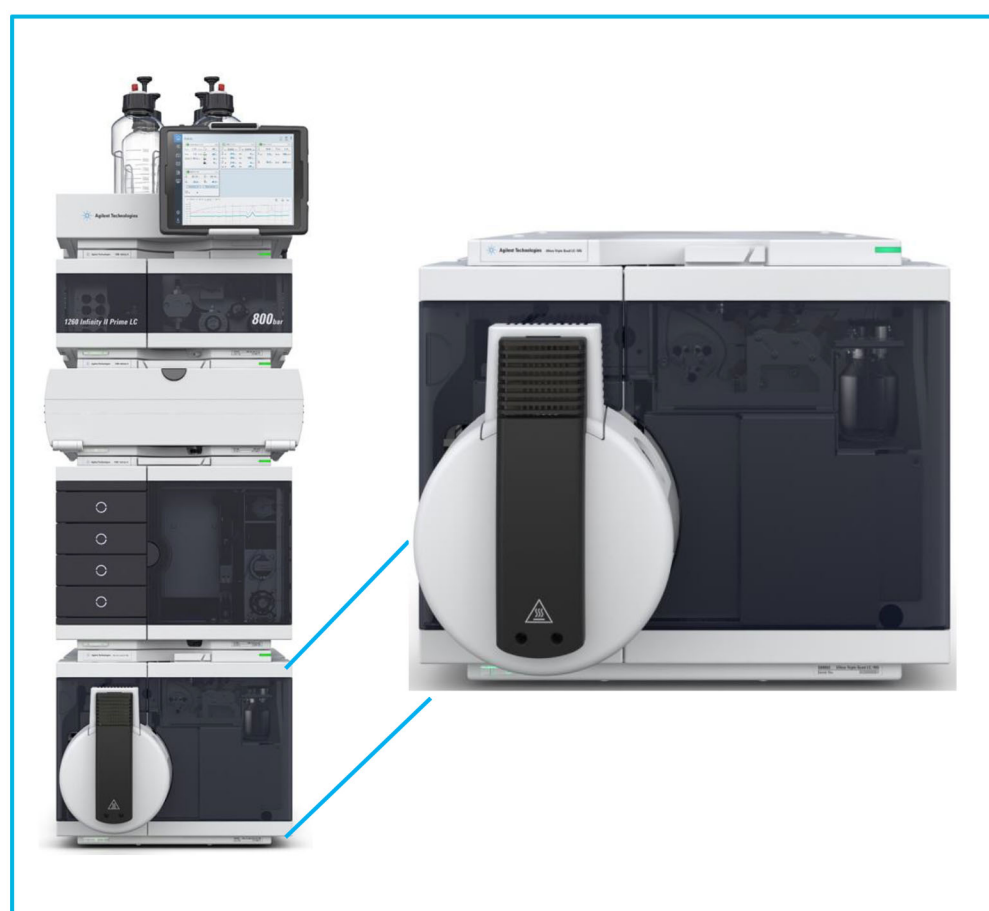


Figure 1. Ultivo Triple Quad LC/MS

Experimental

Sample Preparation

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

LC and Mass Spectrometer parameters

The LC and MS parameters are listed in Table 1 and 2.

Column	Eclipse Plus C18 3.0 x 150 mm, 1.8 μ m	
Column temp	45°C	
Injection volume	2 μ L	
Mobile phase	A: Water, 0.5mM NH ₄ F +4.5mM NH ₄ formate+ 0.1% Formic Acid B: MeOH, 0.5mM NH ₄ F +4.5mM NH ₄ formate+ 0.1% Formic Acid	
Flow rate	0.45 mL/min	
Gradient	Time	B%
	0	2
	0.5	2
	1	50
	4	65
	18.1	2
Stop Time	20min, Post Time 1.5min	

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 of Ultivo was outstanding due to the technological innovations as illustrated in Figure 1 and 2.

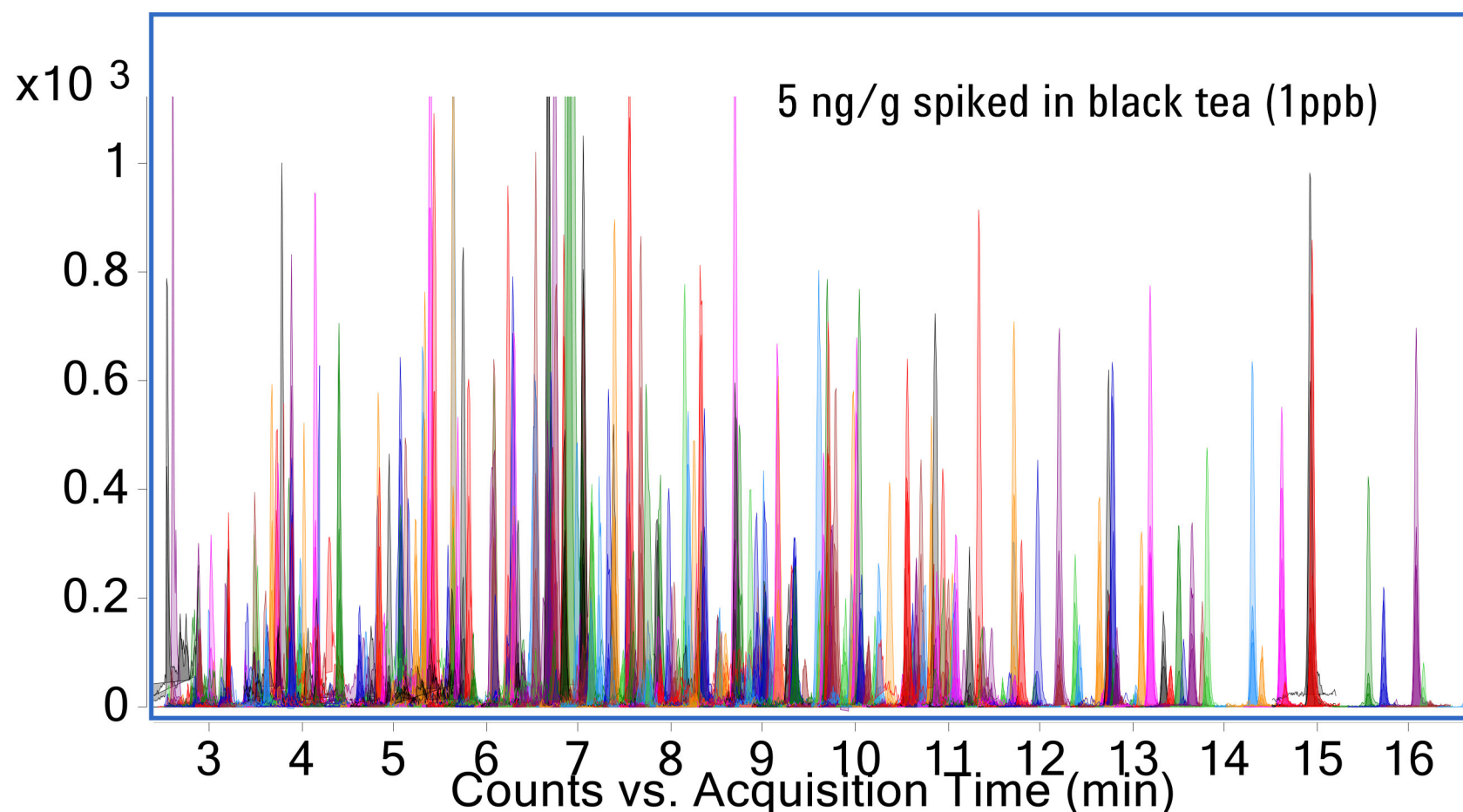


Figure 2. Outstanding signal response of 5 ng/g spike in Black Tea matrix (1ppb)

Sensitivity and Precision

Most of the compounds could be detected far below MRL with accuracy of 80-120% for at least 4 out of 6 replicates. The precision was excellent, with %RSD less than 10% for most of the compounds at the LOQ as shown in Figure 3, 4.

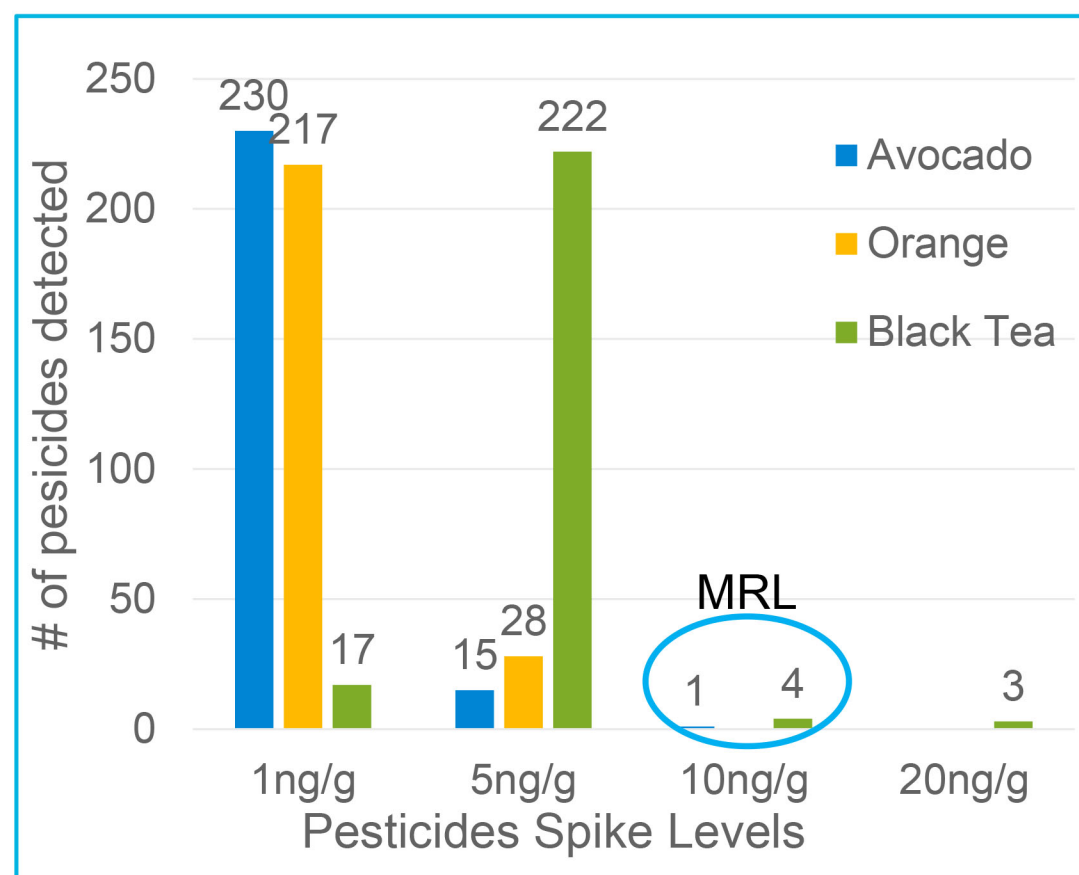


Figure 3. Outstanding sensitivity: Most of compounds could be accurately detected far below MRL (one compound was not detected in orange due to matrix interference).

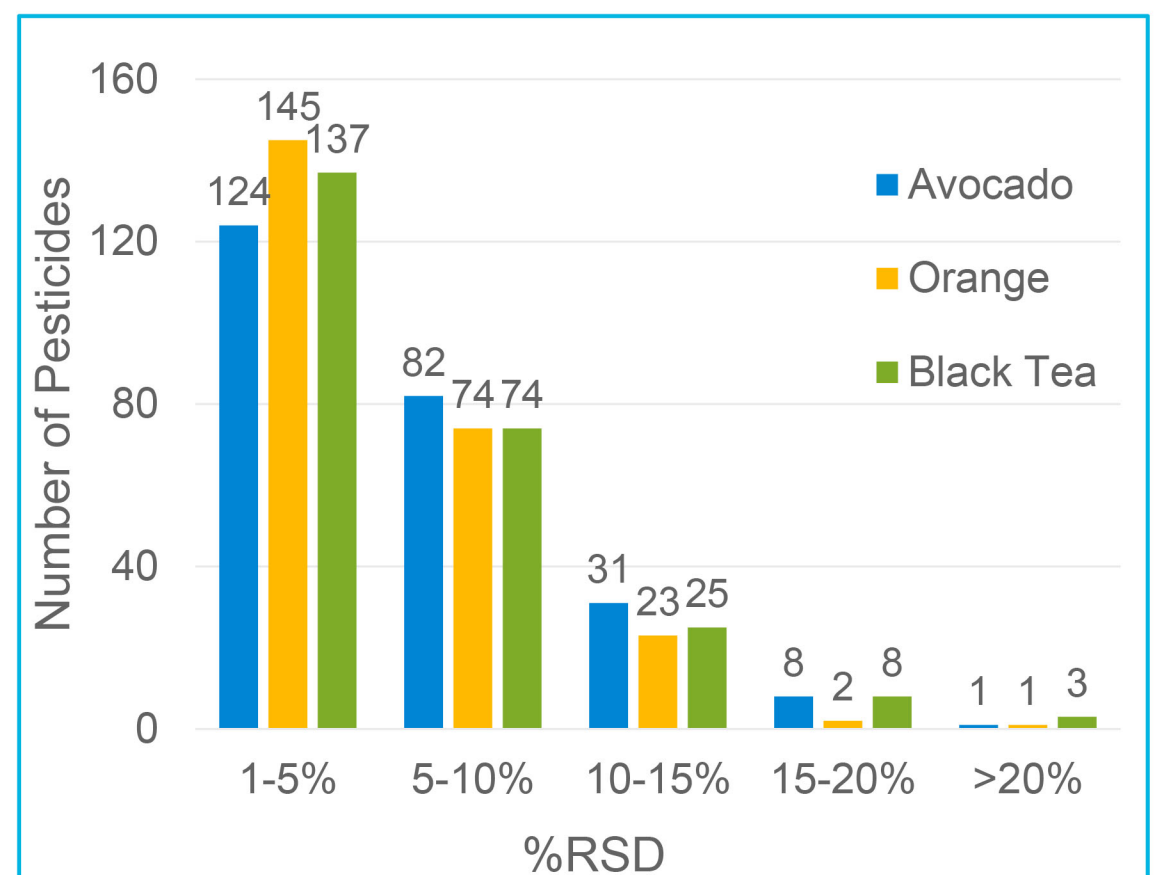


Figure 4. Excellent precision: most of compounds had %RSD less than 10% at the lowest quantitation level (n=6) without any outlier rejection.

Results and Discussion

Real World Samples Analysis: Non-Organic Orange and Avocado

Non-organic 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 non-organic avocado, while 3 pesticides were detected in non-organic orange above MRL (Figure 5).

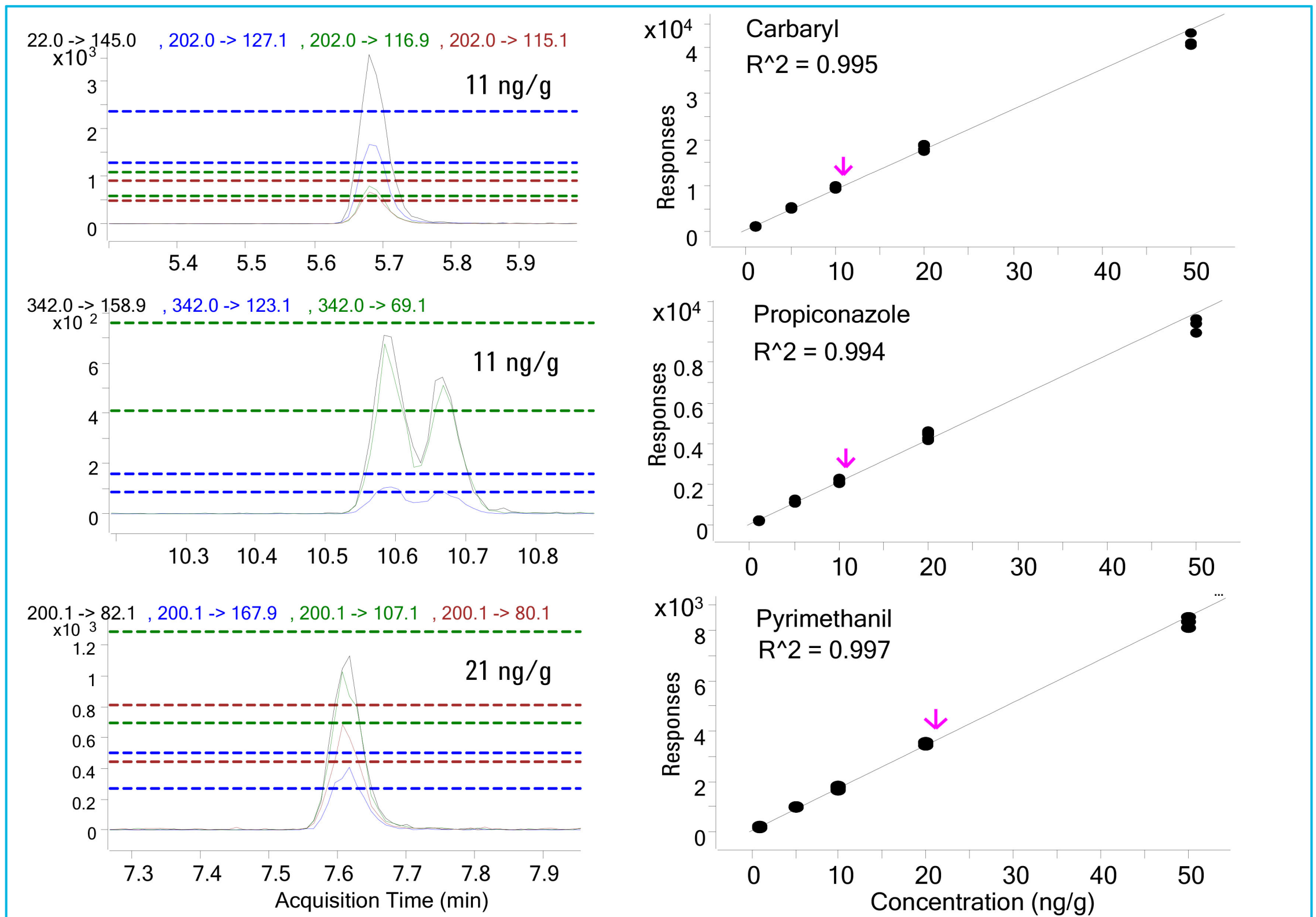


Figure 5. Pesticides detected above MRL in non-organic orange

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.