Food & Environmental

Multi-Pesticide Quantitation and Identification using Ultra Fast LC with Accelerated MRM Detection

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Introduction

LC-MS/MS is a powerful analytical tool capable of screening samples for numerous compounds. Multiple Reaction Monitoring mode (MRM) is typically used because of its excellent sensitivity, selectivity, and speed. As LC-MS/MS technology continues to be adapted as a preferred method of analysis, demands are made to detect and quantify an increasing number of compounds in a single run. In addition, as the number of samples requiring analysis continues to rise, it is necessary to use shorter analysis times as well, placing a heavier burden on mass spectral detection.

The novel AB SCIEX Triple Quad™ 5500 and QTRAP® 5500 LC/MS/MS Systems incorporate the proven technology of the Turbo V™ source and the Curtain Gas™ interface for ultimate sensitivity and robustness. The unparalleled speed of MRM detection is made possible by advanced eQ™ electronics and the new Qurved LINAC® collision cell to take full advantage of ultra fast LC without compromising data quality. The ability of the new 5500 series systems to detect and quantify hundreds of MRM transitions in a short amount of time is demonstrated by analyzing food and water samples for 75 common pesticides.

Method Details

- Ultra High Pressure Liquid Chromatography using a Shimadzu UFLC$_{XR}$ system with a Phenomenex Synergi Fusion-RP (2.5 µm) column and an ultra fast gradient of water and methanol with 5 mM ammonium formate buffer
- Total run time of less than 5 min
- AB SCIEX Triple Quad™ 5500 System with Turbo V™ Source and ESI probe
- Two MRM transitions , a quantifier and qualifier, for each of the 75 analytes for a total of 150 MRM transitions
- Compound identification using the ratio of the qualifier ion to quantifier ion
- Positive identification if the analyte MRM ratio was 20% of the ratio of the average of the ratio of the standards
- Direct injection of water samples
- Extraction of food samples using a QuEChERS procedure and 10x dilution with mobile phase to minimize possible matrix effects

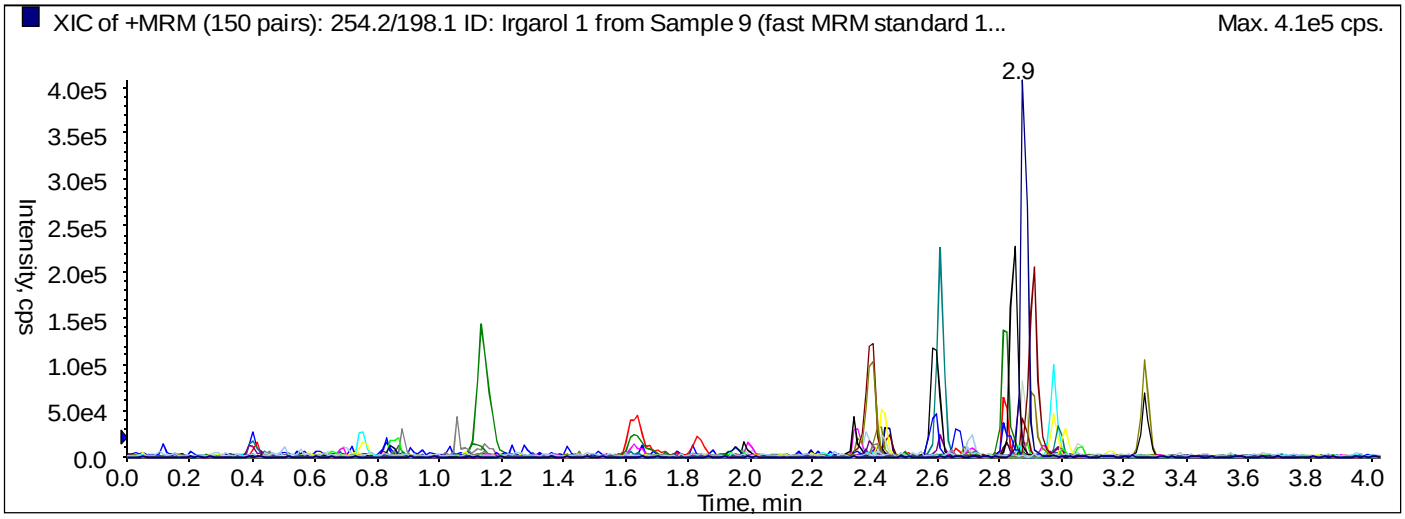

Figure 1. Ultra Fast Screening for 75 Pesticides. This is a representative chromatogram showing a 1 ng/mL standard of 75 pesticides analyzed using the AB SCIEX Triple Quad™ 5500 System. A dwell time of 2 ms per MRM transition and a pause time of 3 ms were used.

Results

The new AB SCIEX Triple Quad™ 5500 and QTRAP® 5500 LC/MS/MS Systems allow the use of very short dwell and pause times to monitor MRM transitions. This feature is especially important when UHPLC is combined with MS/MS to quantify and identify a larger panel of analytes.

The developed method was applied to detect 150 transitions in a chromatographic run of less than 5 minutes. Sensitivity, reproducibility, linearity were investigated using 2ms pause time 3ms and compared to traditional settings of 5ms pause and dwell time. In addition, the cross talk behavior was studied when ultra fast MRM detection is performed.

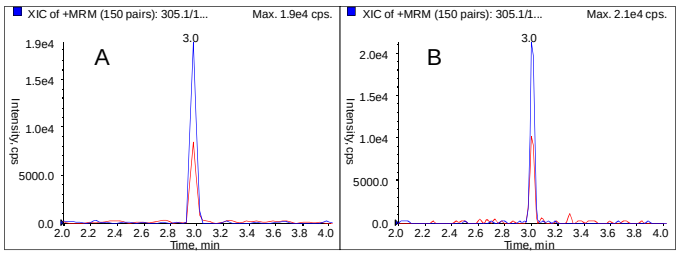

Figure 2. Effect of cycle time on data quality. Both chromatograms show the injection of a 1 ng/mL standard of Diazinon using UHPLC conditions and detection in MRM mode. Figure 2A shows data for acquisition using a 5 ms dwell time and 5 ms pause time. The %CV measured over three replicates was 9.1%. Figure 2B shows data for acquisition using a 2 ms dwell time and 3 ms pause time. More data points were collected across the LC peak, which improves peak shape versus Figure 2A. The %CV for three replicates improved to 5.5%.

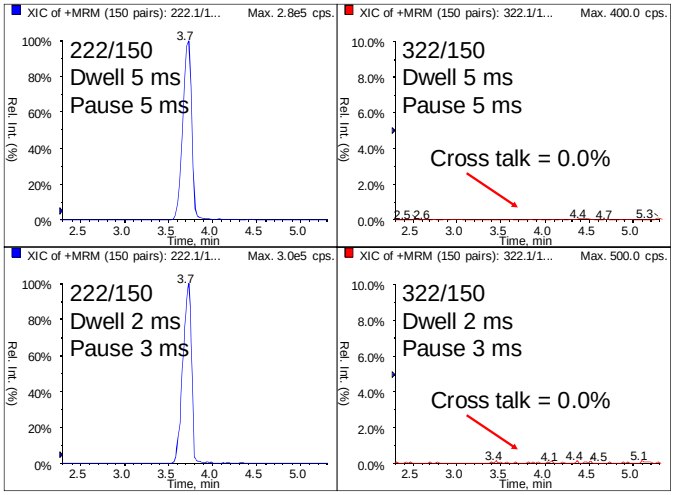

Figure 3. Study of cross talk. The affect of dwell time and pause time on cross talk was studied to determine minimum times that can be utilized. Two MRM transitions were monitored; one corresponding to an analyte (222/150) and the other an artificial transition that had the same Q3 mass as the analyte to monitor possible cross talk. No crosstalk was observed with dwell times and pause times set as low as 2 ms and 3 ms, respectively.

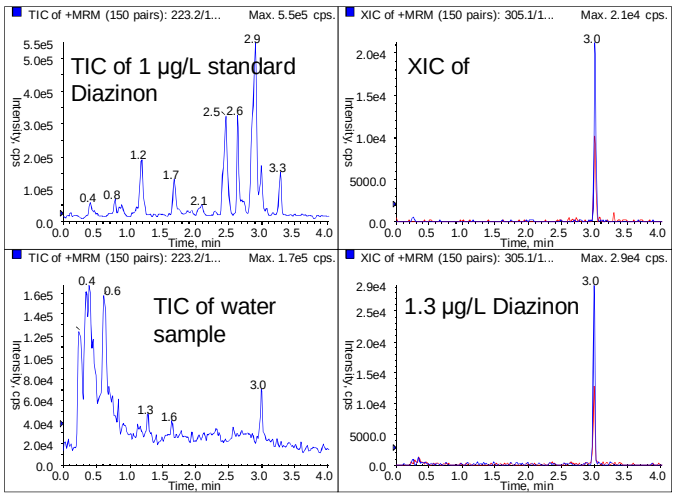

Figure 4. Detection of Diazinon in a water sample collected in an urban area. The extracted ion chromatograms for the quantifier and qualifier ions of Diazinon (RT 3.0 min) at a concentration of 1 µg/L are shown on top. Standards had an average MRM ratio of 0.46 resulting in an acceptable identification range of 0.37-0.55. Diazinon was detected at 1.3 µg/L in the water sample (bottom). The water sample had a Diazinon MRM ratio of 0.42, clearly identifying the presence of this pesticide.

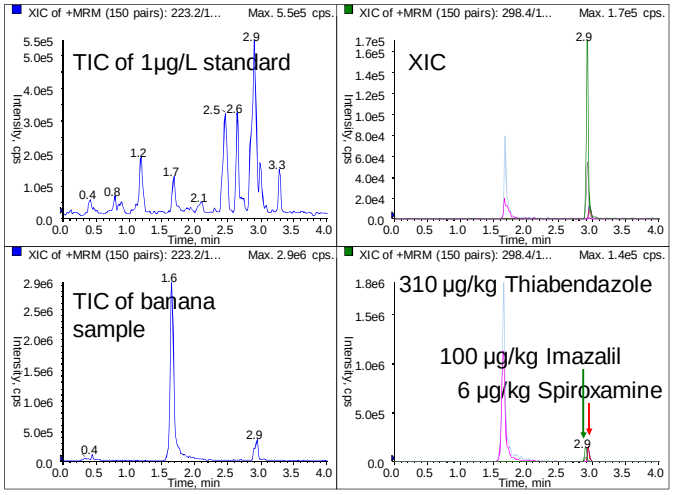

Figure 5. Analysis of a banana sample. Thiabendazole, Spiroxamine, and Imazalil were detected in a banana sample at concentrations of 310 µg/kg, 6 µg/kg, and 100 µg/kg, respectively (bottom). Extracted ion chromatograms of a calibration standard for these three pesticides are shown on top. The ratio of the qualifier ion to the quantifier ion was used to clearly identify the quantified pesticides.

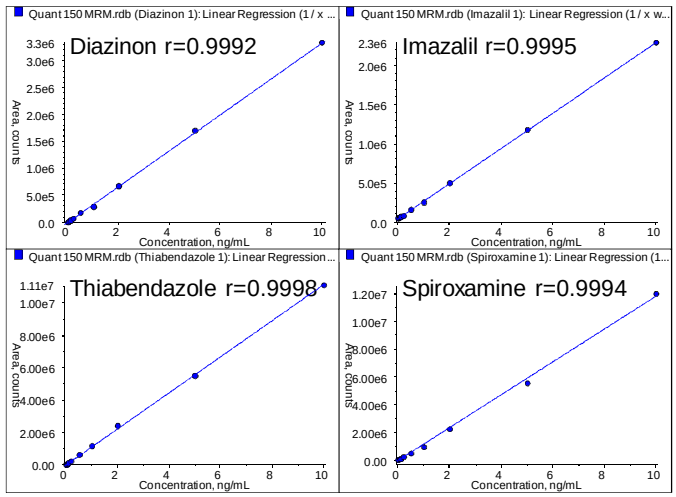

Figure 6. Examples of calibration curves. The wide linear dynamic range of the developed multi-pesticide quantitation method is illustrated by the calibration curves of Diazinon, Imazalil, Thiabendazole, and Spiroxamine.

Summary

The new AB SCIEX Triple Quad™ 5500 and QTRAP® 5500 LC/MS/MS Systems are powerful tools for ultra fast multi-analyte quantitation and identification in matrices such as water and food samples. In the example presented, water and fruit samples were screened for the presence of 75 pesticides and identified using MRM ratio with a run time of less than 5 minutes. Because of the excellent sensitivity of this mass spectrometer, water samples can be injected directly and food extracts can easily be diluted at least 10 times prior analysis and still meet required detection limits. This dilution step helps to minimize any potential matrix effects and interferences, as well as helps ruggedness of the overall technique. The high speed scanning capabilities of the new 5500 series systems permit the use of very short dwell times and pause times, resulting in the ideal detector for the ultra-fast LC methods being employed in today's modern laboratories. Analysis times can be reduced, therefore sample throughput increased, while maintaining data quality (sensitivity, reproducibility, linear dynamic range, no crosstalk).

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