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Comparative Study of High-Resolution Q-TOF Fast Polarity Switching versus Single Polarity Data Acquisition on Mass Accuracy, Resolution and Analytical Sensitivity

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Introduction

Fast Polarity Switching Facilitates Increased Sample Throughput for LC/Q-TOF Analysis

Laboratories around the world face the challenge of an ever-increasing sample load and demands for higher productivity and faster turn-around times. Implementing Fast Polarity Switching (FPS) in MS only data acquisition mode is one way to increase sample throughput two-fold, but the impact of FPS on mass accuracy, dynamic range, and analytical sensitivity are also important factors to consider when evaluating the merits of FPS versus Single Polarity (SP) mode.

To evaluate the impact of FPS on mass accuracy and dynamic range, a complex mixture of pesticides in broccoli matrix was analyzed in FPS and SP modes on the new Agilent Revident LC/Q-TOF, which features both high-resolution and wide in-spectrum dynamic range. In addition, the thermally-inert flight tube on the Revident LC/Q-TOF provides long-term mass stability for superior performance over time. Here, we report the average and compound-specific mass accuracy differences observed as a function of concentration and overall analytical sensitivity (1 ppb- 100 ppb).

Figure 1. The Agilent Infinity II 1290 LC and Agilent Revident LC/Q-TOF provide LC/MS data with wide in-spectrum dynamic range and high-resolution, with data collection abilities in Single Polarity or Fast Polarity Switching modes.

Higher throughput analysis with FPS data acquisition on the Revident LC/Q-TOF pairs efficiently with streamlined data analysis available using the LC Screener Tool in MassHunter Quantitative Analysis 12.1 to quickly identify which analytes are present and those which may need further review.

Experimental

Sample Preparation and MS Analysis for Pesticides in Complex Food Matrices

A broccoli matrix was prepared by homogenization and extraction with QuEChERS for Pigmented Fruits and Vegetables. The resulting supernatant was spiked with over 200 pesticide standards from the Agilent LC/MS Pesticide Standard Kit (PN: 5190-0551) at concentrations from 1 ppb to 100 ppb. The spiked samples were analyzed in both FPS and SP MS only data acquisition modes using an Agilent 1290 Infinity II LC interfaced with a high-resolution Agilent Revident LC/Q-TOF with a Dual JetStream (AJS) electrospray ionization source.

Table 1. LC/Q-TOF Conditions for Pesticides Analysis

LC Conditions		
Column	Agilent Zorbax Ecllipse Plus C18, 3.0 x 150 mm, 1.8 µm	
Column temperature	45 °C	
Autosampler temp	4 °C	
Injection volume	3 µL	
Mobile phase	A: Water, 0.2% 4.5 mM ammonium formate, 0.5 mM ammonium fluoride, 0.1% formic acid B: Methanol, 4.5 mM ammonium formate, 0.5 mM ammonium fluoride, 0.1% formic acid	
Flow rate	0.45 mL/min	
Gradient program	**Time**	**%B**
	0.0	2
	0.5	2
	1.0	50
	4.0	65
	16.0	100
	18.0	100
	18.1	2
	20.0	2
Post time	4 min	
Total run time	24 min	
Revident LC/Q-TOF MS Conditions		
Acquisition rate	FPS: 2 spectra/s SP: 2 spectra/s	
Gas temperature, Flow	325 °C, 10 L/min	
Nebulizer pressure	35 psi	
Sheath gas temp, Flow	375 °C, 12 L/min	
Nozzle voltage	200 V	
Capillary voltage	2500 V	

Good Mass Accuracy for both Single Polarity and Fast Polarity Switching Modes on the Revident LC/Q-TOF

Standards for over 200 pesticides in broccoli matrix were analyzed using the Revident LC/Q-TOF in both SP and FPS modes for MS data acquisition. Both data acquisition methods gave strong results with most mass accuracies within ±1 ppm. In addition, the % RSDs of the abundances were under 20% for >98% of measurements made at the 5 ppb and 10 ppb levels for all measured pesticides in both polarities using both FPS and SP modes.

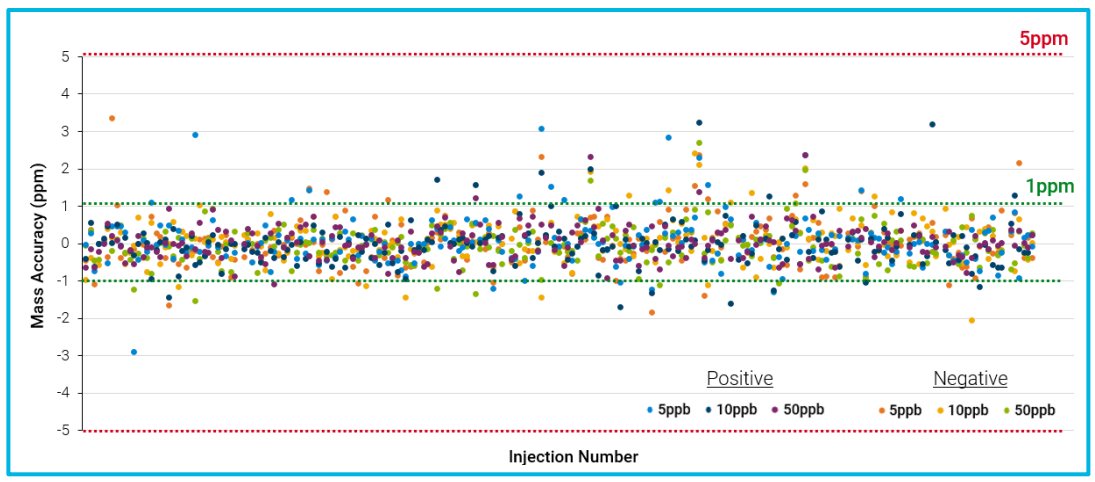

Figure 2. Mass accuracy stability for 200+ pesticides across across 8 hours in Fast Polarity Switching (FPS) mode. >93 % of measurements fell within ±2 ppm (green lines) and 100 % within ±5 ppm (red lines).

Fast Polarity Switching Yields Comparable Compound Identification to Single Polarity Data Acquisition

The number of pesticides identified with FPS data acquisition was nearly identical to the number of pesticides identified through a combination of both positive and negative single polarity data acquisition across multiple concentrations levels, as shown in Table 2. These results demonstrate that the higher throughput achieved with FPS data acquisition does not limit compound identification, even in complex matrix with a combination of analytes that favor positive ionization and analytes that favor negative ionization.

Table 2. Number of Pesticides Identified at 5ppb, 10ppb, and 50ppb with .Fast Polarity Switching and Single Polarity Data Acquisition Modes.

Number of Pesticides Identified			
Level	**FPS**	**Positive Only**	**Negative Only**
5ppb	196	191	6
10ppb	203	196	6
50ppb	216	208	6

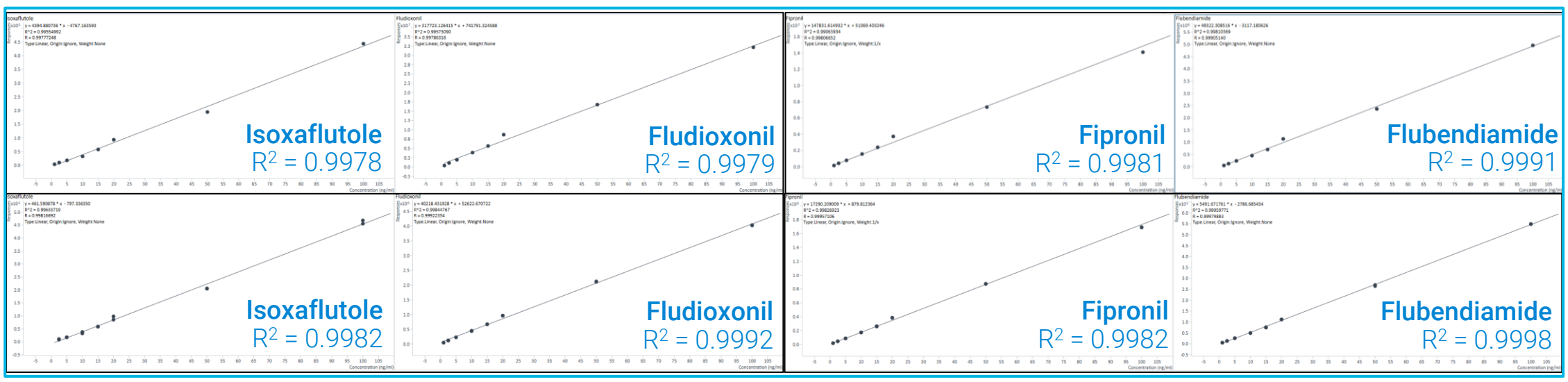

Figure 3. Calibration curves for negative ion compounds show good linearity from 1ppb– 100ppb in both fast polarity switching (top) and single polarity (bottom) data acquisition modes with R^2> 0.99 for all identified pesticides in matrix.

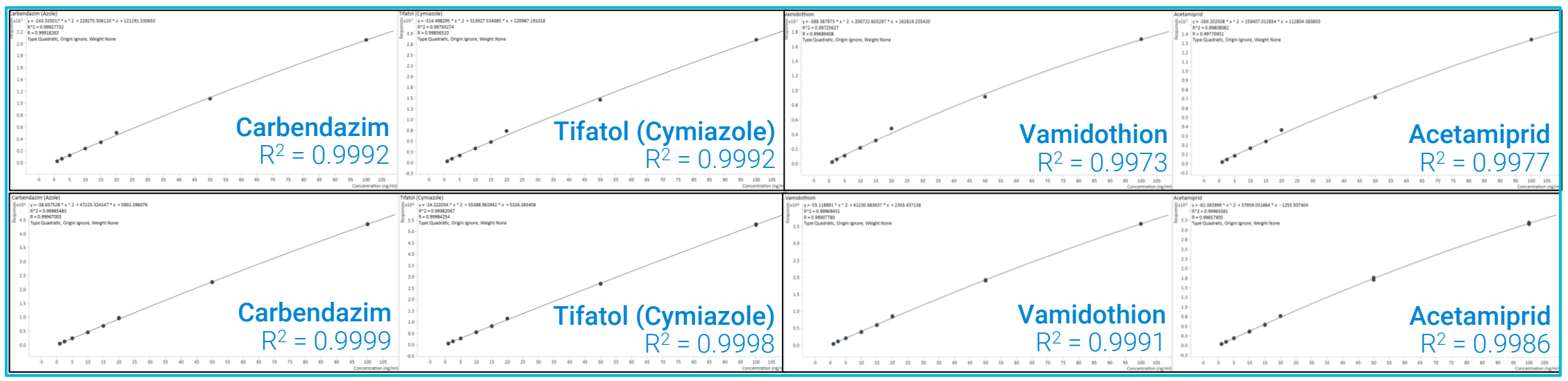

Figure 4. Calibration curves for positive ion compounds show good linearity from 1 ppb – 100 ppb in both fast polarity switching (top) and single polarity (bottom) data acquisition modes with R^2 > 0.99 for all identified pesticides in matrix.

Results and Discussion

Fast Polarity Switching Provides Strong Performance for Positive and Negative Ions across Calibration Curves

Both FPS and SP data acquisition yielded calibration curves with $R^2 > 0.99$ for all identified compounds, with similar performance for post acquisition modes across both positive and negative polarity data across concentrations ranging from 1 ppb to 100 ppb (Figures 3 and 4). Strong performance across this concentration range is particularly relevant for pesticide analysis following SANTE guidelines.

LC Screener Tool in MassHunter Quant Streamlines Data Analysis

The LC Screener Tool in MassHunter Quantitative Analysis 12.1 was used to summarize data based on the presence (green), absence (red), or possible detection (orange) as decided by modifiable screening settings based on attributes of data including mass accuracy, signal-to-noise ratio, mass match score, and retention time. Screening parameters for pesticide data analysis were set to SANTE guidelines.[1] Prior to analysis with MassHunter Quant, acquired FPS data was recalibrated using the Agilent TOF Reprocess UI to include reference mass calibration for both positive and negative data.

Figure 5. LC Screener tool provides an easy to navigate table with a clear, color-coordinated display that connects seamlessly with the corresponding spectral displays.

https://www.agilent.com/en/promotions/asms

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The combination of a robust library of pesticides complete with accurate mass and retention time information and the LC Screener Tool settings of mass accuracy ≤ 5 ppm, coelution score above 80%, and signal-to-noise ≥ 3 facilitated the streamlined identification of more than 200 pesticides with both FPS and SP data across concentration levels. These results in combination with the demonstrated preservation of mass accuracy and sensitivity demonstrate the timesaving ability of FPS data acquisition without loss of data quality with the Revident LC/Q-TOF.

Faster Scan Speeds with Agilent's Revident LC/Q-TOF Facilitate more Rapid Polarity Switching

During mass spectrometric analysis, many compounds respond better to a particular ionization mode to produce mostly positive or mostly negative ions, creating an analytical challenge when a sample mix contains both analytes that favor positive ionization and analytes that favor negative ionization. To meet this challenge for high-resolution analysis, FPS data acquisition can be used to detect analytes in a mixture regardless of their preferred polarity. The faster a mass spectrometer can switch polarities, the more scans it can acquire from a particular chromatographic peak. The 1 second total cycle time for both positive and negative acquisition with fast polarity switching on the Revident LC/Q-TOF is faster than legacy instruments, allowing for superior performance

Conclusions

Fast Polarity Switching with the Revident LC/Q-TOF Delivers Strong Mass Accuracy, Isotope Fidelity and Mass Resolution

- More than 200 pesticides identified in complex broccoli matrix below, at, and above SANTE guideline levels with fast polarity switching data acquisition.
- Good mass accuracy was achieved with both fast polarity switching and single polarity MS acquisition.
- MassHunter Quantitative Analysis facilitates easy, routine data analysis.
- 1 second polarity cycle switching on the Revident LC/Q-TOF makes it ideal for high-level suspect screening using fast polarity switching mode.

References

[1]Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed, SANTE/113212, 2021, v2. https://food.ec.Europa.eu/system/files/2023-11/pesticides_mrl_guidelines_wrkdoc_2021-11312.pdf.

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