

Poster Reprint

ASMS 2021
Poster number FP461

Innovation Development of Rapid-Sep Screening Technology of the Multi- Residue Pesticides Analysis in Agricultural Products using UHPLC-MS/MS

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Introduction

Pesticides are widely applied for the control of pests, including insects, rodents, weeds, bacteria, mold, and fungus, to increase agricultural productivity, where the safety issue of human consumption and environment persistence is followed with interest. The goal of pesticide residue monitoring has been required to meet regulatory data quality and food safety objectives with the highest throughput and lowest cost possible.

A simple, fast, and inexpensive method of UHPLC-MS/MS was developed for the determination of 200 of pesticides within 5 min. **The innovative Rapid-Sep Screening Technology (RSS Technology)** uses the 1290 UHPLC and Ultivo LC/TQ system to create a mobile laboratory to strengthen the risk management for food-safety and to reach farm-to-fork ideally.

In this study, an Agilent Poroshell SB-C18 column packed with 1.9 μ m particles was used to achieve UHPLC analysis with symmetric peak shapes and better separation. Agricultural extracts were prepared using Custom kits prior to auto-injection for direct quantification using the Ultivo LC/TQ.

Experimental

1290 Infinity II HPLC System Conditions

Column: Agilent Poroshell SB-C18, 2.1X50mm, 1.9 μ m

Mobile phase A: 5mM NH₄CO₂H and 0.1% Formic acid in water

Mobile phase B: 5mM NH₄CO₂H in methanol

Column Oven: 40 °C

Gradient program: (Flow rate: 0.15~0.2 mL min⁻¹ with splitter from 0.7 mL min⁻¹)

Time (min)	0	0.1	1.5	2.5	4
A (%)	99	50	30	1	1
B (%)	1	50	70	99	99

Post-Run Time: 1 min

Experimental

Ultivo LC/TQ - MS/MS System Conditions

Ion source: AJS, Fast switch mode

Nebulizer gas: 55psi

Dry gas: 10 L min⁻¹

Dry gas Temperature: 225°C

Sheath gas: 11 L min⁻¹

Sheath gas Temperature: 300°C

Nozzle voltage: 0V (Positive); 500V (Negative)

Capillary voltage: 4500V (Positive & Negative)



Figure 1. Agilent Ultivo Triple Quadrupole LC/MS

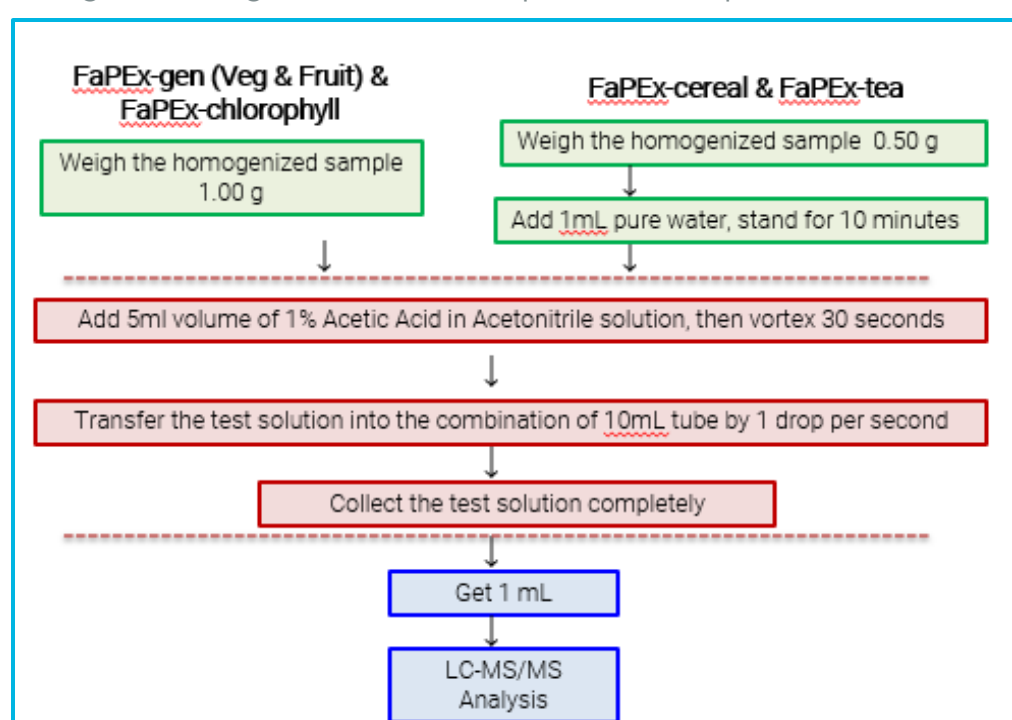


Figure 2. The workflow of sample preparation from difference sample type with FaPEX

RSS technology is combined of three parts: the FaPEX extraction, Study Manager control, and intelligent Target Analysis System (iTAS). The first sample analysis is carried out within the first 30min, then a series of samples were analyzed sequentially within 5 min per sample. The iTAS software is an intelligence system to report qualified results automatically. The FaPEX and iTAS were derived from products developed by Taiwan Agricultural Chemicals and Toxin Substances Research Institute (The patent numbers are US 9,581,579B2 and US 10001,462 B2), respectively. The validation was performed according to criteria found in the SANTE/12682/2019 guidelines

RSS technology equipped with the Agilent Ultivo LC/TQ can be integrated and developed into a “ Mobile laboratory”.

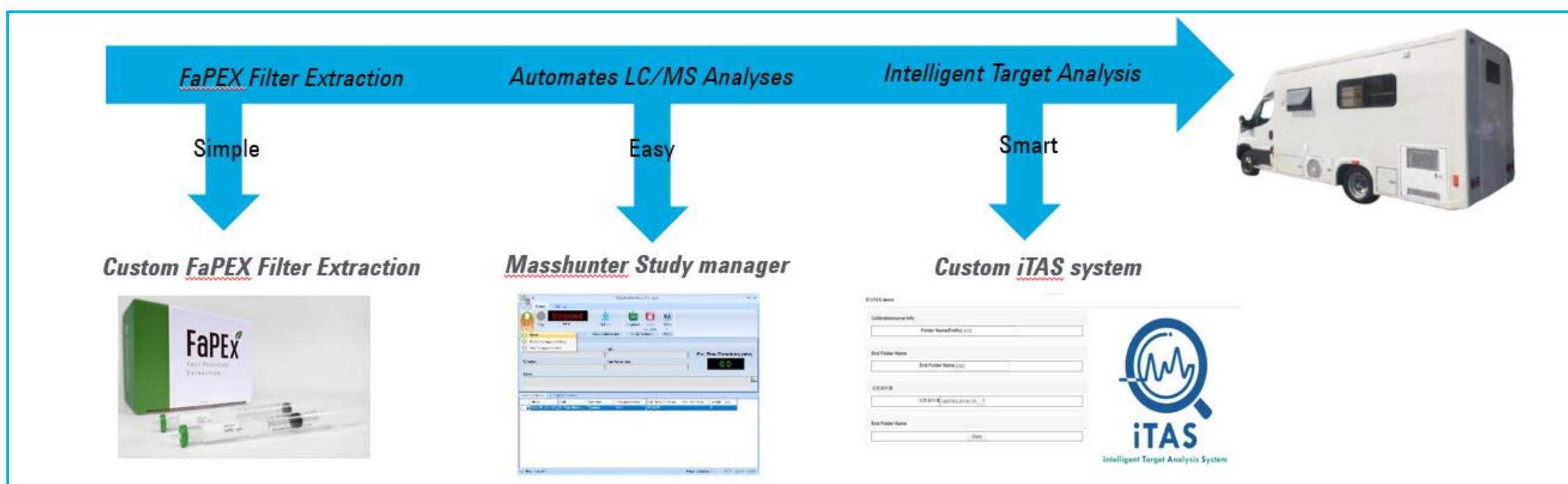


Figure 3. The Workflow of RSS Technology

The 200 pesticides are detected using dynamic MRM mode. The chromatographic overlap of 489 transitions can be seen in the Figure 4. We can observe that maximum concurrent MRMs of 176 transitions were overlapped. The minimum dwell time was just 1.26ms with about 600ms of total cycle time.

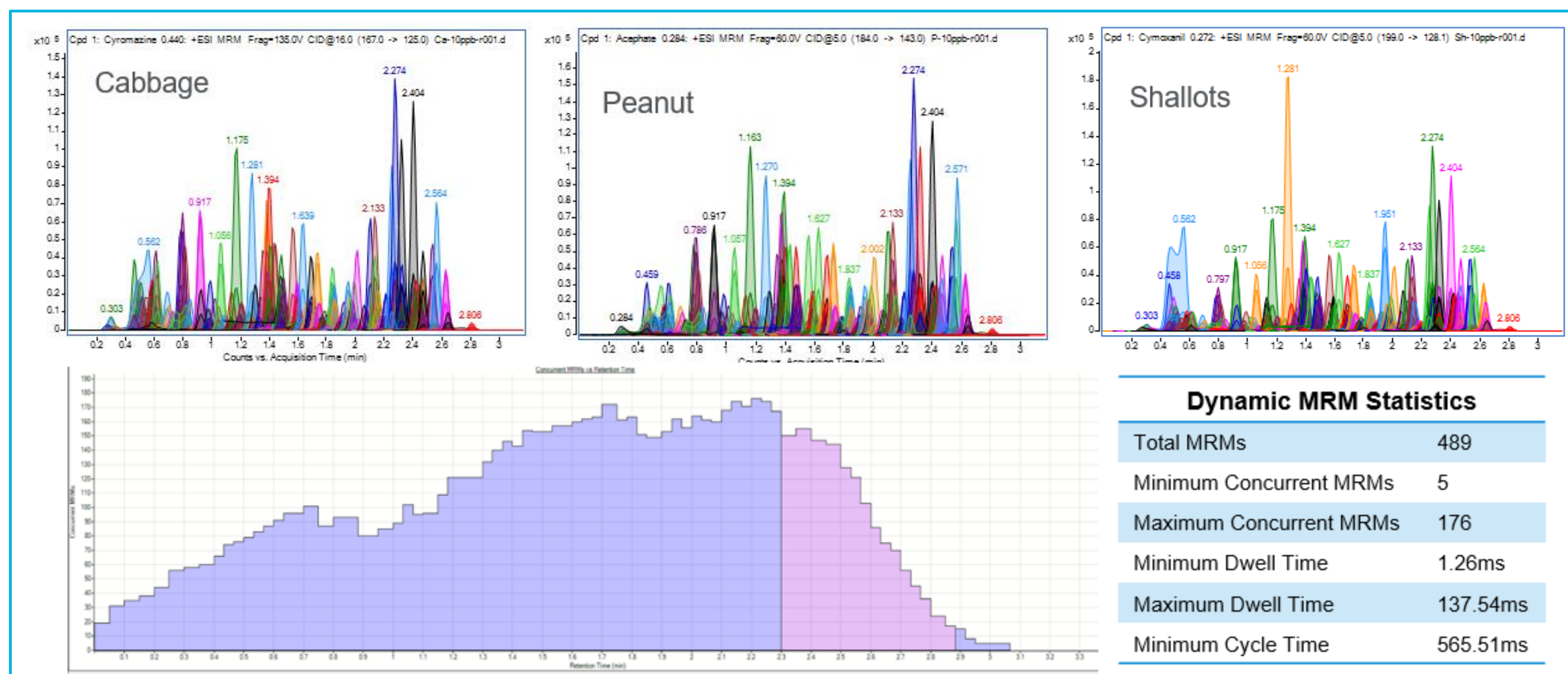


Figure 4. The MRM chromatography of three extracts, cabbage, peanut and shallots were spiked 10 ng g⁻¹ and the results of Dynamic MRM statistic.

Results and Discussion

Good recovery of over 98% pesticides were found to be in the range of 50-110% and the screening detection limit (SDL) was 10.0 ng mL⁻¹ or 50.0 ng mL⁻¹ for fast qualitative analysis with single calibration point. All multi-targets analytes can work with matrix-match calibration (MMC) and reach 1.0-2.0 ng mL⁻¹ for quantitative analysis. Moreover, good precision and linearity were obtained for quantitative determination in agriculture products (Data not shown).

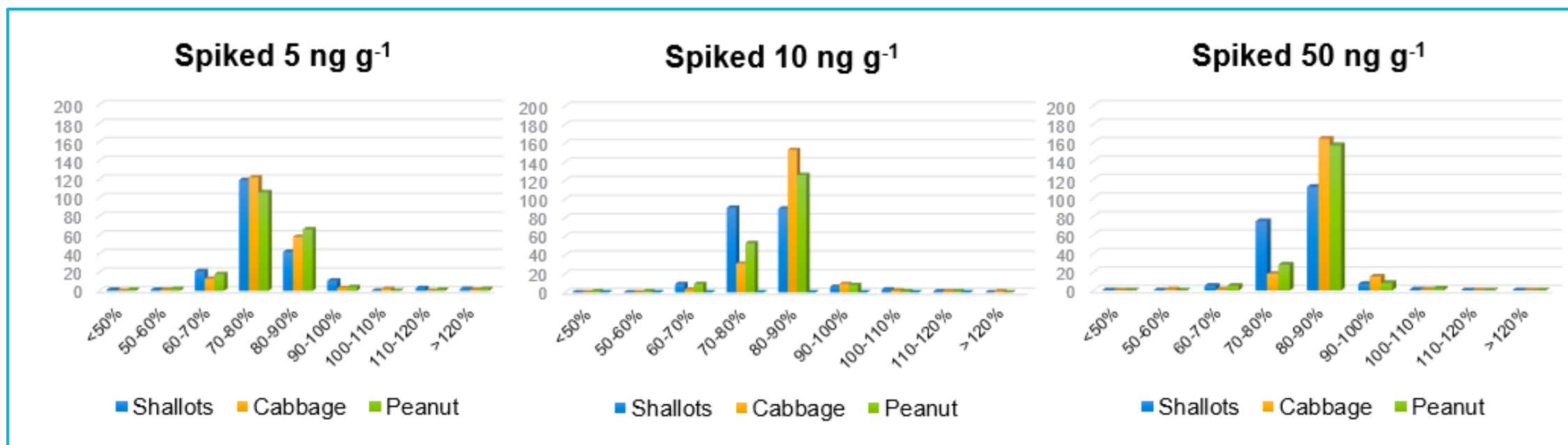


Figure 5. Recovery % of 200 pesticides spiked at difference concentrations. (5 ng g⁻¹, 10 ng g⁻¹, and 50 ng g⁻¹)

Results and Discussion

In this study, the high flow, 0.7 mL min⁻¹ was split to 0.15~0.2 mL min⁻¹ to improve the electrospray efficiency. The result for eleven targeted pesticides was enhanced for sensitivity and better signal as a result. The range of sensitivity enhancement was from 144% to 473% to reach the lowest detection limit for the seven pesticides. The others showed the better stable signal from “no signal” or “unstable signal”.

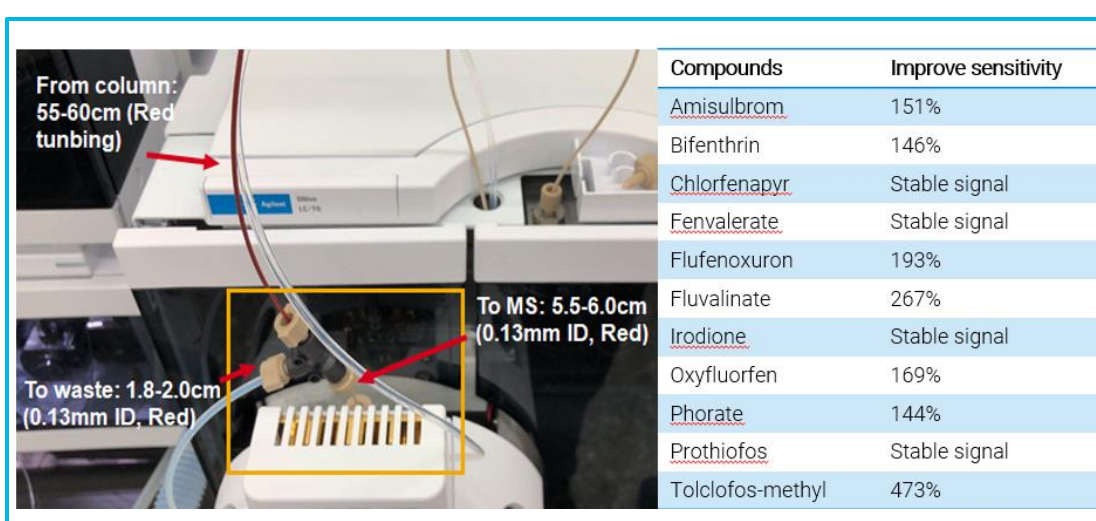


Figure 6. The splitter device was designed to improve the sensitivity and signal stability.

Conclusions

Gradient elution and dynamic MRM in RSS Technology was employed for simultaneous identification of the two hundred pesticides. The precision was below 20% and the R² value was better than 0.99 within linear range optimized with Ultivo system (Data not shown). **The innovative RSS technology also can be applied to all Agilent mass spectrometers** and offered an ultrafast, highly selective, and sensitive detection for food-safety applications by successfully employing the method to qualitatively and quantitatively determine multi-pesticides in complex agriculture products.

References

- [1] FaPEX Multipesticide Residue Extraction Kit for Minimizing Sample Preparation Time in Agricultural Produce., *Journal of AOAC INTERNATIONAL*, Volume, Issue 6, 2019, Page 1864-1876.
- [2] Possibilities and Limitation of Isocratic Fast Liquid Chromatography-Tandem Mass Spectrometry Analysis of Pesticide Residue in Fruits and Vegetables., *Chromographia* (2019) 82: 235-250.

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DE44475.5716550926

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Published in USA, October 20, 2021

