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Automated sample preparation using CTC PAL3 to analyze >570 pesticides in an orange by the combination of GC/MS/MS and LC/MS/MS techniques

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Introduction

Automated sample preparation can not only increase testing efficiency, but also reduces dependency on in-person activity. For example many lab chemists had to stay at home due to COVID-19 pandemic, resulting in a lot of sample testing been delayed due to the shortage of chemist availability. Compared with manual work, an automated workflow, especially coupled online to mass spectrometry, is less manpower reliant and a best choice to tackle this kind of situation.

The manual sample preparation process for QuEChERS includes solvent extraction, salting and cleaning, with multi-time of shaking/vortex and centrifugation. This process avoids dependency on chemist time and increases the precision of the results due to reducing human error.

An automated workflow was developed to analyze multi-class pesticides in orange with the combination of GC/MS/MS and LC/MS/MS techniques. Sample preparation was automatically done with CTC PAL3 on the basis of QuEChERS, which ensured consistent and high quality results. Worth mention, using scripts, the sample preparation was done in overlapped mode, avoiding additional sample prep overhead time.

Validations were done at three different spiking levels (10μg/Kg, 20μg/Kg, 50μg/Kg) with 5 replicates for each level. The validation results for automatic sample preparation and manual sample preparation were summarized and compared.

1290Infinity II LC + 6470

Figure 1. Instruments used in the experiment

Experimental

Automatic sample Prep.

Weigh 5.0 ± 0.01 g homogenized orange sample into 20 mL vial

↓

Add 10 mL of ACN, Vortex at 1800 rpm for 2 min

↓

Centrifuge at 3500 rpm for 3 min

↓

Transfer 3.5 mL supernatant to 10 mL vial (containing 1g Agilent QuEChERS EN salt powder)

↓

Vortex at 1800 rpm for 3 min

↓

Centrifuge at 3500 rpm for 3 min

↓

Load 250 µL supernant to corresponding µSPE cartridge (45 mg) with controlled speed

↓

Elute the cartridge with ACN (1% formic acid) (optional, improve recovery for some herbicides)

↓

Add X µL IS

↓

Add X µL standard (for calibration sample only)

↓

Mix well

Partial parameters of PAL3 auto sample prep.

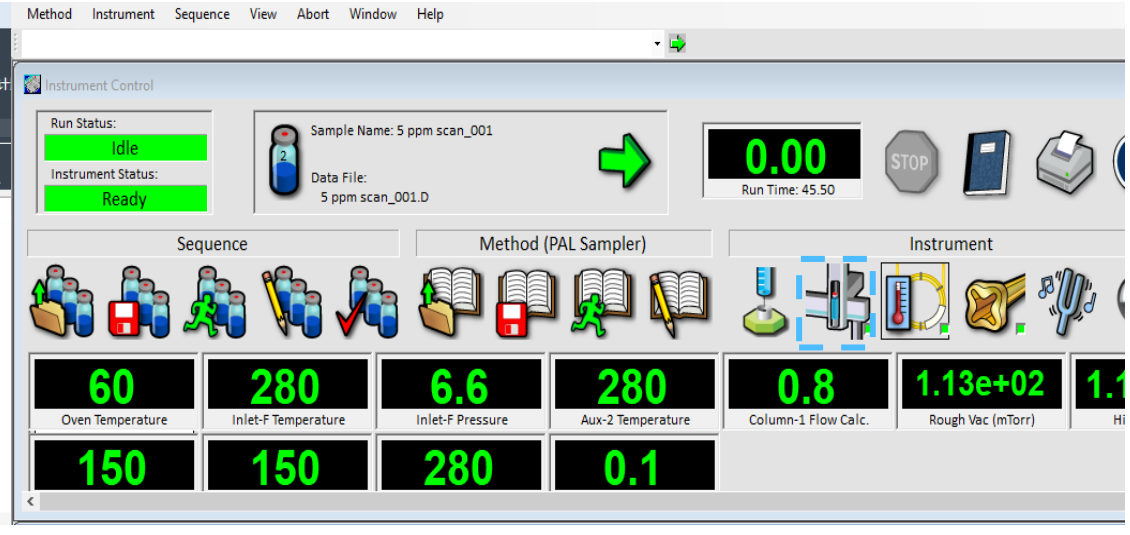
Conditioning		
Conditioning Solvent Source	none	
Conditioning Solvent Index	2	
Conditioning Solvent Volume	100	µL
Conditioning Solvent Fill Speed	10	µL/s
Sample µSPE		
µSPE Sample Load Volume	257	µL
µSPE Sample Fill Speed	5	µL/s
Elution		
Elution Solvent Source	none	
Elution Solvent Index	2	
Elution Volume	100	µL
Elution Solvent Fill Speed	5	µL/s
Protectants		
Protectant Source	none	
Protectant Index	3	
Protectant Volume	0	µL
Protectant Fill Speed	5	µL/s

Internal Standard		
Internal Standard Source	none	
Internal Standard Index	54	
Internal Standard Volume	7.7	µL
Internal Standard Fill Speed	1	µL/s
Solvent Addition		
Solvent Source	none	
Solvent Index	1	
Solvent Volume	16.45	µL
Solvent Fill Speed	5	µL/s
Target Standard		
Target Standard Source	none	
Target Standard Index	52	
Target Standard Volume	3.85	µL
Target Standard Fill Speed	1	µL/s
Mixing		
Mix Cycles	5	
Mix Volume	500	µL
Injection		

Experimental

Instrument control panel

LC/MS/MS

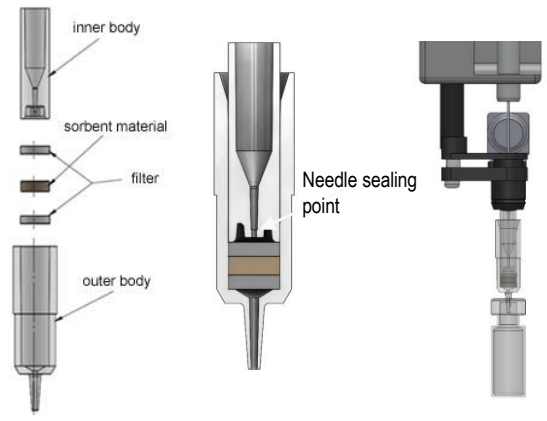


GC/MS/MS

Picture: liquid tools

Picture: centrifuge

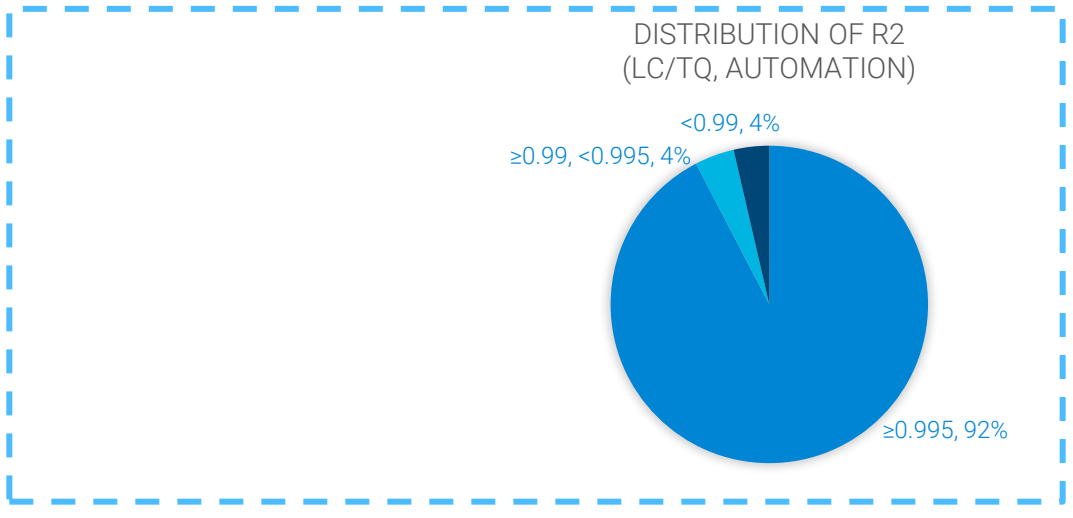
Picture: vortex

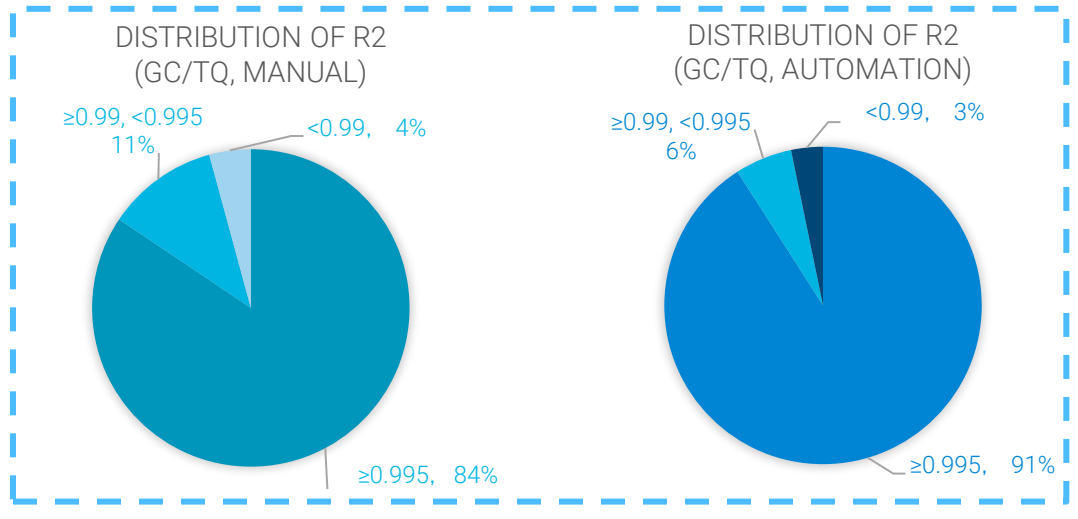


Picture: Design of µSPE

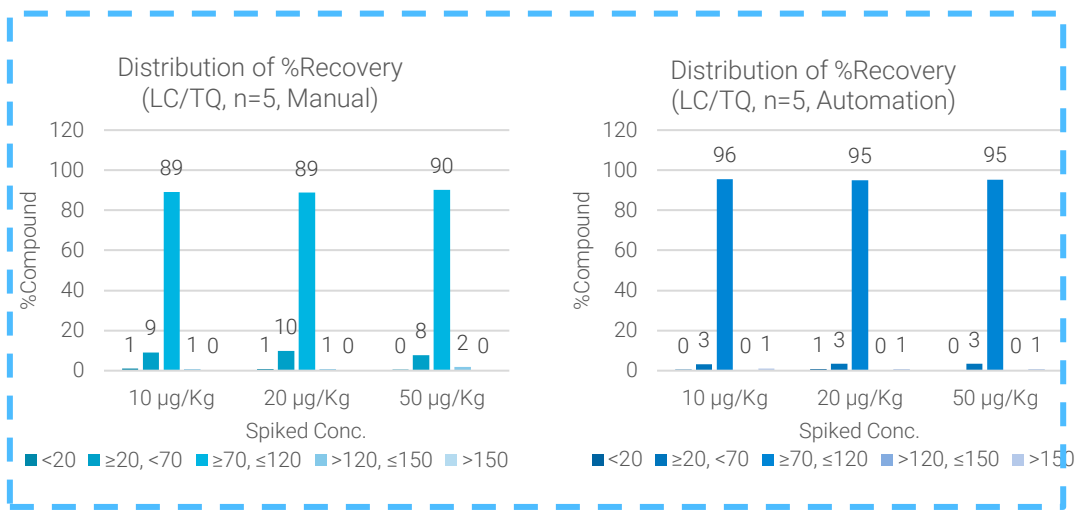
Results and Discussion

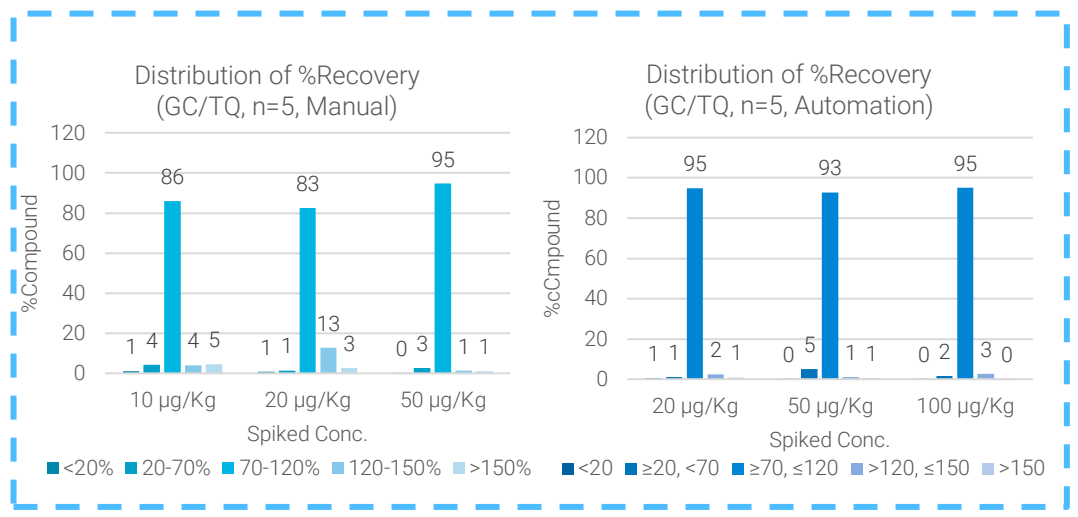
Linearity (*manual vs. automation*)



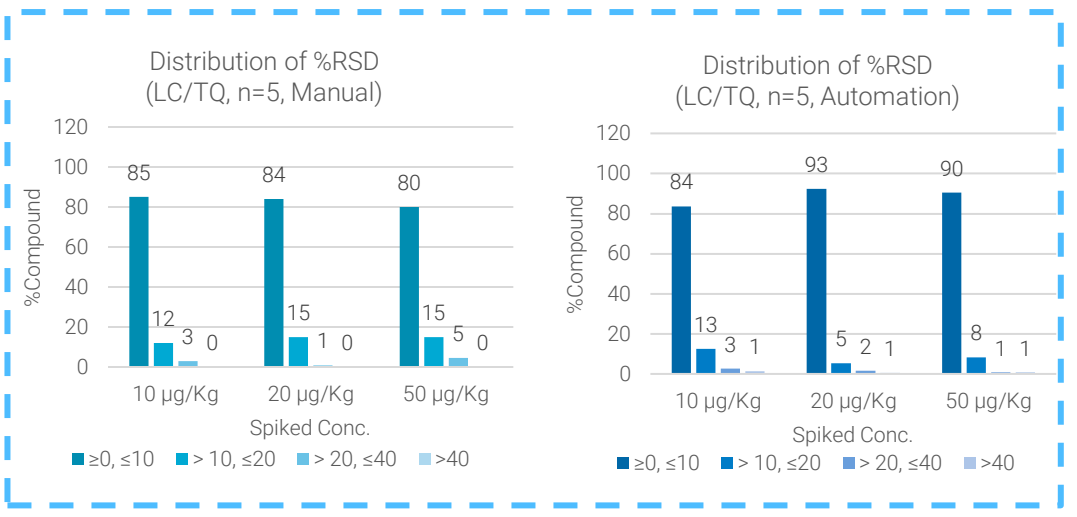


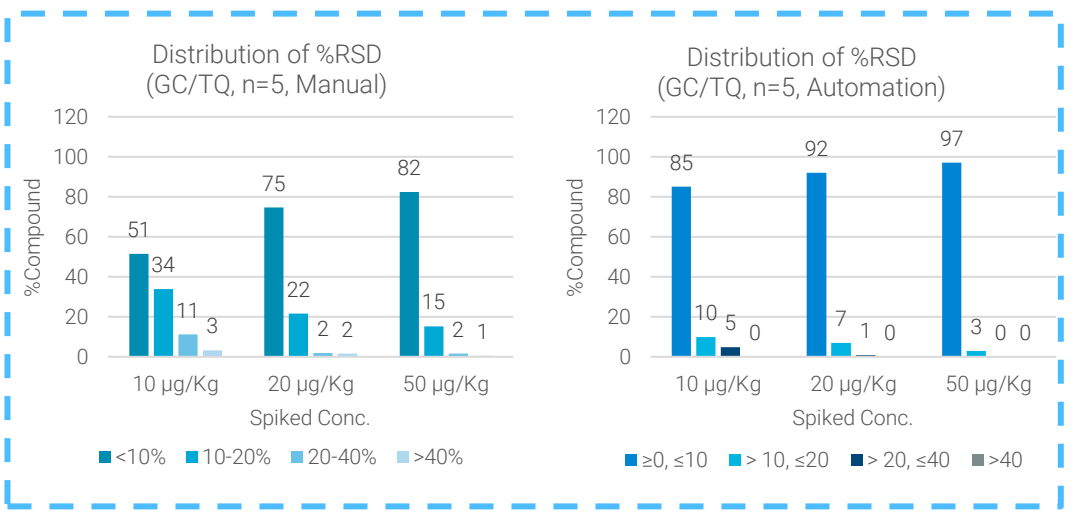
Accuracy (*manual vs. automation*)





Accuracy (*manual vs. automation*)





Conclusions

Full automation, high efficiency, low cost

This is the first time to develop a whole automatic sample preparation workflow for analyzing > 570 pesticides in orange by the combination of GC/MS/MS and LC/MS/MS techniques.

In general, automatic workflow has better performance than manual workflow by comparison of linearity, accuracy and repeatability of validation results done by manual and automated sample preparation.

References

GB2763-2021: National food safety standard- Maximum residue limits for pesticides in food.

GB23200.121-2021: National food safety standard- Determination of 331 pesticides and metabolites residues in foods of plant origin-LC/MS/MS

GB23200.121-2018: National food safety standard- Determination of 208 pesticides and metabolites residues in foods of plant origin-GC/MS/MS

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