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Determination of 198 pesticide residues in eggplant using Gas Chromatography tandem Mass Spectrometry/Mass Spectrometry

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Introduction

The number of relevant pesticides grows yearly, and has reached hundreds of compounds. Multi-residue simultaneous analysis of hundreds of pesticides in agricultural products is always a challenge. And as required detection limits for many pesticides fall to 10μg/kg (10ppb), more sophisticated analytical tools are demanded.

Due to its excellent sensitivity and selectivity, GC-MS/MS with MRM acquisition mode is utilized for the analysis of residual pesticides in foods. The aim of this work was the development and validation for the fast method of the simultaneous analysis of 198 pesticides in eggplant using gas chromatography tandem mass spectrometry (GC-MS/MS) in multiple reaction monitoring (MRM) acquisition mode. The samples were pretreated with the QuEChERS method. The treated samples were then subjected to MRM analysis for 198 pesticides using GC-MS/MS, every compound has 2 MRM transitions (primary for quantification, secondary for qualification), totally 396 transitions in 38 minutes. The established method was sensitive, repeatable and reliable for simultaneous analysis of the 198 pesticides in eggplant samples.

Experimental

Sample pretreament

The samples were pretreated with the QuEChERS method. Internal standard substance (heptachlor-endo-epoxide, 50 ng/mL) was used to overcome matrix effect and achieve quantitative determination.

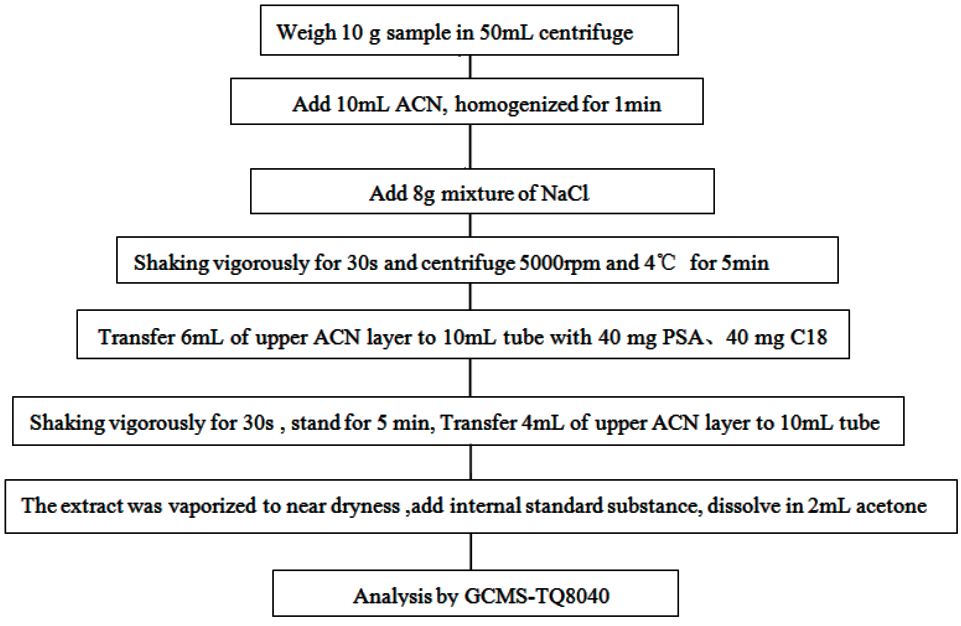

Figure 1 Schematic flow diagram of the sample preparation

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GC/MS/MS Analysis

Treated samples were analyzed in MRM mode using a gas chromatograph coupled with a triple quadrupole mass spectrometer (GCMS-TQ8040, Shimadzu Corporation, Japan). The MRM transitions and collision energies for every compound were acquired from the pesticide MRM database provided by Shimadzu Corporation which contains 588 pesticides.

Analytical Conditions	
GC-MS	: GCMS-TQ8040
Column	: Rxi-5 Sil ms, 30 m x 0.25 mm, 0.25 µm
GC	
Injection port temperature	: 250 °C
Temperature program	: 50 °C (1 min)-25 °C/min-125 °C-10 °C/min-300 °C (15 min)
Injection mode	: splitless (1 min)
Injection Volume	: 1µL
Linear velocity	: 47.2 cm/sec
MS	
Ion source temperature	: 200 °C
Interface temperature	: 250 °C
Measurement Mode	: MRM
Loop Time	: 0.3sec

Results and Discussion

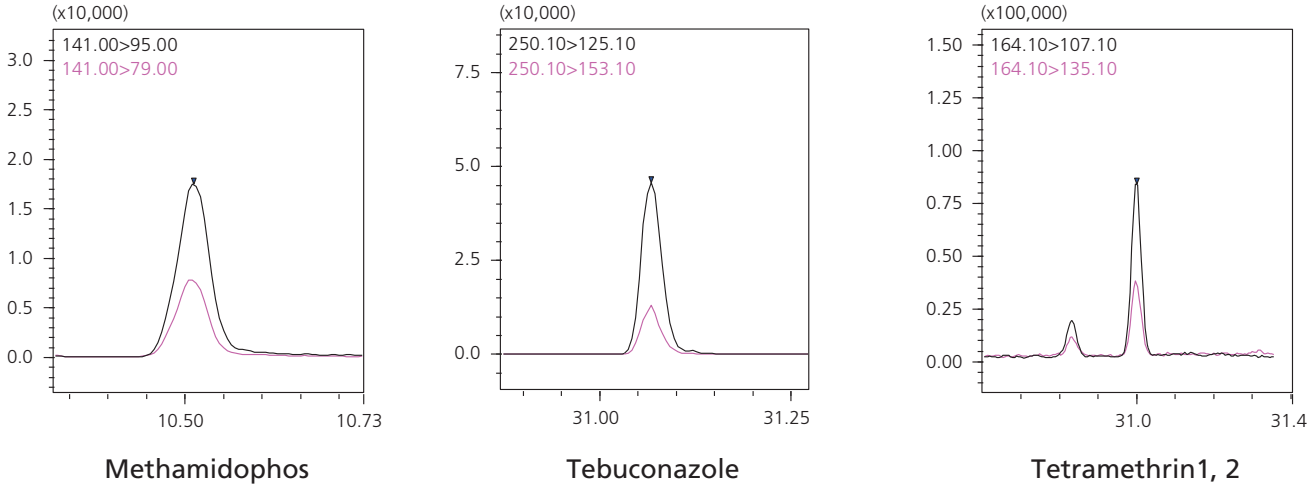

Figure 2 MRM chromatograms of Methamidophos, Tebuconazole and Tetramethrin spiked in grape samples (10 ng/mL)

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The eggplant samples were prepared using the aboved method, 198 pesticides were spiked in the sample extract (10 ng/mL). Spiked samples were analyzed in MRM mode using GC-MS/MS. Fig.2 shows mass chromatograms for Methamidophos, Tebuconazole and Tetramethrin spiked in eggplant samples (10 ng/mL).
In order to assess the method linearity, calibration curves were constructed for the198 pesticides spiked in the sample matrix, using heptachlor-endo-epoxide as the internal standard. The limits of detection (LODs) of all the pesticides studied were 0.1µg/kg. The average recoveries were 70%-120% of target compounds and the relative standard deviations (%RSD, n=6) were less than 12% in spiked levels at 5µg/kg.

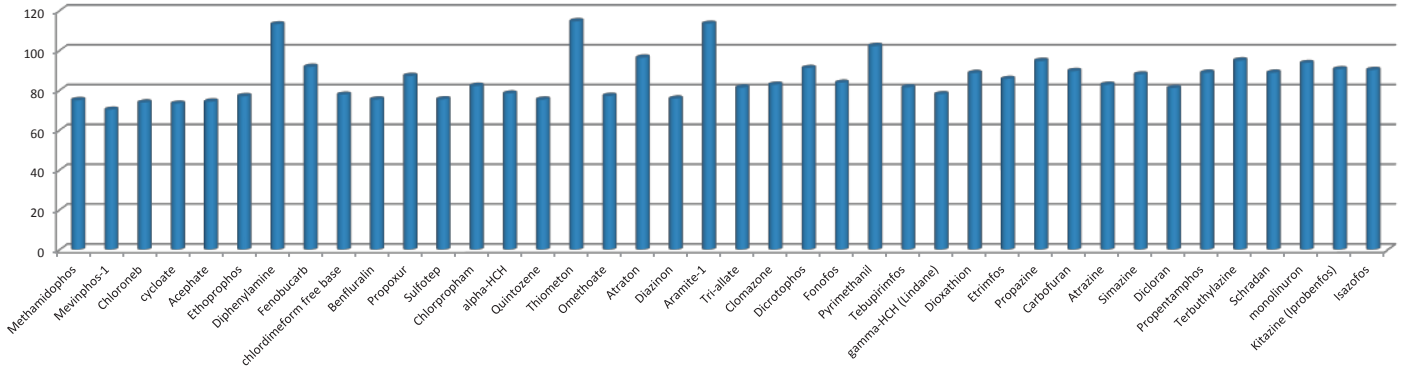

Figure 3 Recovery of part of 198 pesticides at 5µg/kg spiked in eggplant sample

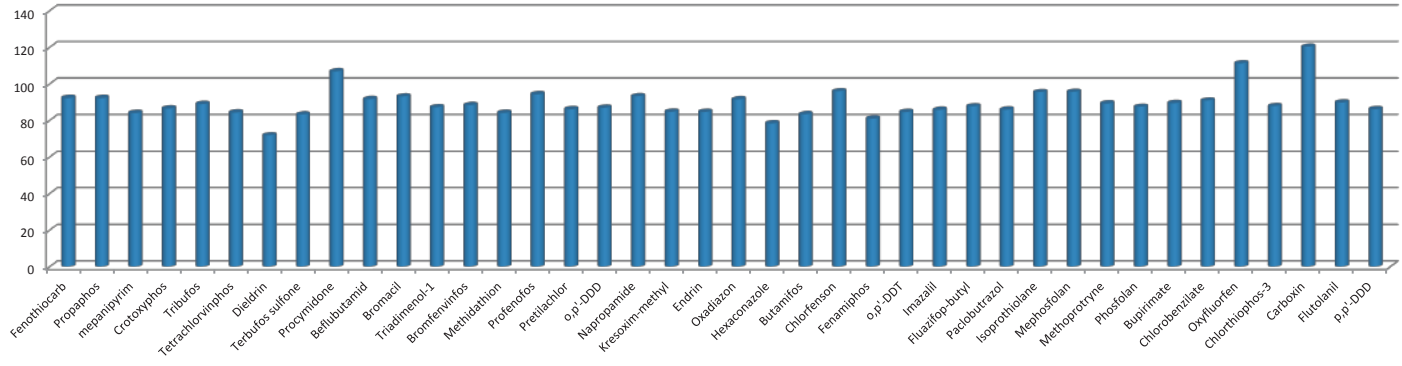

Figure 4 Recovery of part of 198 pesticides at 5µg/kg spiked in eggplant sample

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Conclusions

- A fast method was developed for the simultaneous analysis of 198 pesticides in eggplant using gas chromatography tandem mass spectrometry (GC-MS/MS) in multiple reaction monitoring (MRM) acquisition mode.
- The established method was sensitive, repeatable and reliable for simultaneous analysis of the 198 pesticides in eggplant samples.

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