Simultaneous determination of 75 pesticide residues in fruits and vegetables by LC-MS/MS

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Introduction

The laboratory would like to propose, an exhaustive list to customers, of pesticide residues found in fruits and vegetables.
Poor volatility, high polarity, and/or thermal instability of some pesticides, prevents their analyzation by gas chromatography.
Therefore, the laboratory wanted to develop a new method utilizing liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS). The method had to be specific and sensitive, to detect pesticide residue traces in elaborate matrices.

Experimental

First, the mass parameters were optimized; a solution of each pesticide was introduced into the spectrometer one by one, with the aid of a syringe pump. The appropriate ions were selected for each pesticide to determine MRM transitions.
Second, optimization was performed for 6 compound specific parameters for each compound. There were in total 450 parameters for 75 pesticides. The 2 most important parameters were: the DP for declustering potential and the CE for collision energy.
Then, chromatographic development was performed. The analytical column was a Zorbax C18 (150mm*4.6mm id, 5µm). The mobile phase was chosen according to the best sensitivity for the most pesticides. After that, its composition had to elute all molecules in the shortest possible time.
Finally, the optimization of the dwell time, another parameter, was done for all pesticides to increase sensitivity.

Figure 1: simultaneous detection of 75 pesticides at the level of 20 ppb, using the new developed method.

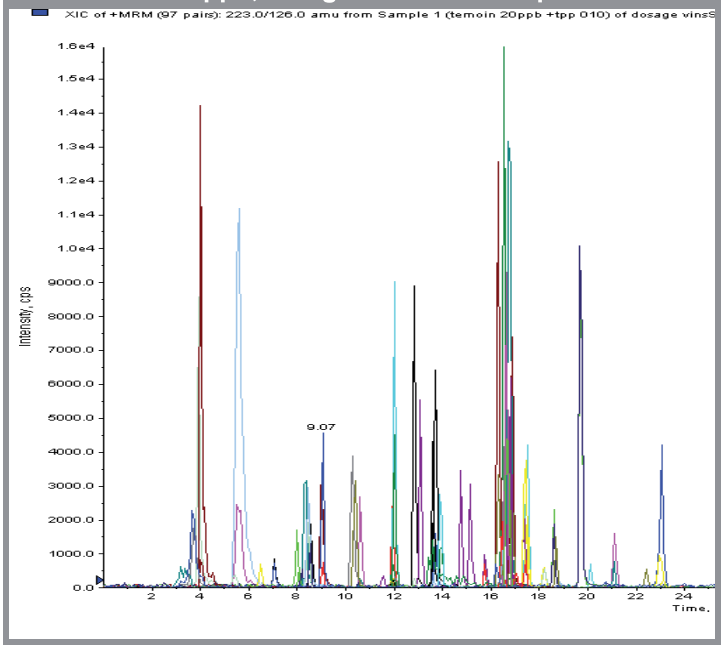

Table1: example of some pesticides researched.

Chemical family	Pesticides
Benzimidazoles	benzimidazole
	carbendazim
	thiophan00alaite methy
Strobilurins	Azoxystrobin
	trifloxistrobin
Ureas	isoproturon
	metoxuron
	linuron
Triazole	epoxyconazole
	fenbucozanole
	tetraconazole
Carbamates	iprovalicarb
	thiodicarb
Pyridilmethyamines	Acetamiprid
	imidachloprid
Various	phenmedipham
	rotenone
	tebufenozide
	lenacil
	thiamethoxam

This new method allowed for the simultaneous detection and quantification of 75 pesticide residues found on fruits and vegetables.
The limit of quantification reached was about 10µg/kg.
With a Zorbax C18 column (150mm*4.6mm id, 5µm) and an elution gradient, the chromatographic run time was approximately 40 min.
The mobile phase was acetonitrile + 0.1% acetic acid/ water + 0.1% acetic acid. The initial composition was 80% water to 20% water from 0 to 15min, followed by 20% water held from 15 to 40min.
The flow rate was fixed at 0.5ml/min.

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