Poster Note 72216

Complete Pesticides Workflow - Combination of LC-MS/MS and LC-HRAM Analysis

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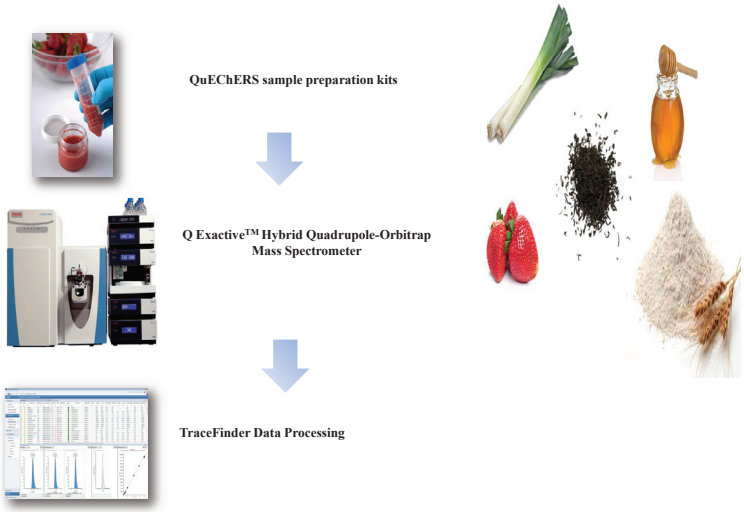
Aim of the study

The aim of our presentation is to report a complete solution package for pesticides analysis workflow comparing two MS based techniques: liquid chromatography-triple quadrupole mass spectrometry (MS/MS) and liquid chromatography-high resolution accurate mass spectrometry (HRAM) Both methods were validated according to the European SANCO guidelines 12495/201.

Methods

Total Pesticide Solution Workflow



Instrumental analysis

Instrumentation

Systems: Thermo Scientific™ TSQ Endura™ Triple Stage Quadrupole MS & Q-Exactive Focus™ High Resolution Mass Spectometer both coupled to UltiMate® 3000 RSLC system.

LC conditions (on both systems):

Column: Accucore column aQ 100 mm x 2.1 mm x 2.6 µm
Mobile phase:
A: Water:MeOH (98:2) + 5mM Ammonium formate & 0.1% FA
B: MeOH:Water (98:2) + 5mM Ammonium formate & 0.1% FA
Injection volume: 1µl; *Flow rate:* 300 µl/min
Column temperature: 25°C*; Run time:* 15 min

TSQ MS conditions:

Source: HESI
Detection mode: t-SRM (1045 SRM transitions)

HRAM MS conditions:

Source: HESI
Detection mode: FullScan-vDIA (variable Data Independent Analysis)*
*vDIA is not available in USA

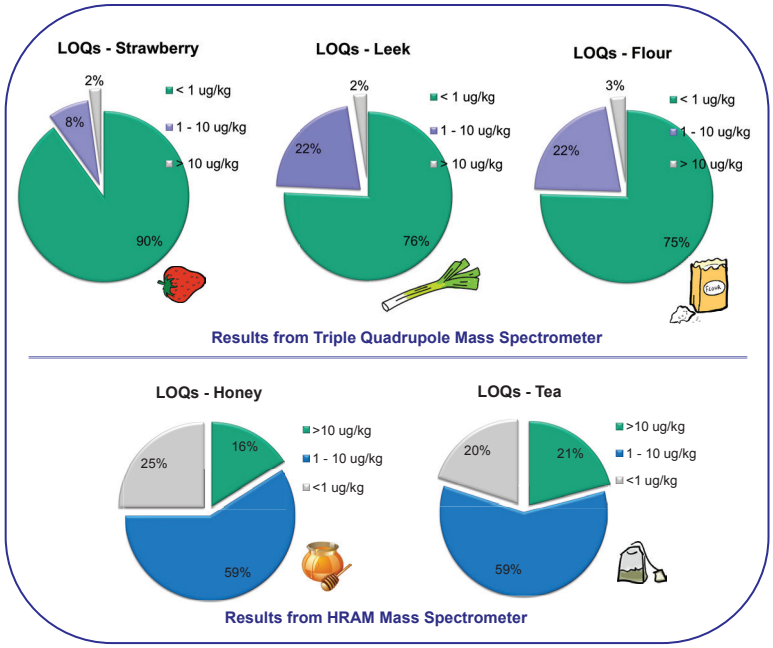
Table 1. Comparison of LOQ values from Triple Quadrupole and HRAM mass spectrometer with MRL values for 12 representative compounds for five different food matrices (in µg/kg).

Compound	Triple Quadrupole							HRAM				
	Solvent	Strawberry		Leek		Flour (wheat)		Solvent	Tea		Honey	
	LOQ	LOQ	MRL	LOQ	MRL	LOQ	MRL	LOQ	LOQ	MRL	LOQ	MRL
Acephate	1.0	1.0	50	1.0	10	1.0	10	2.5	10	50	10.0	20
Azoxystrobin	0.005	0.01	50000	0.01	10000	0.3	300	0.25	1.25	100	0.75	50
Carbaryl	1.0	1.0	50	1.0	10	1.0	500	0.25	3.0	50	2.50	50
Cymoxanil	1.0	1.0	50	10	50	5	50	0.25	5.0	50	1.25	50
Dimetomorph*	1.0	1.0	50	0.1	1500	1.0	10	12.5	25	50	12.5	50
Diniconazole*	1.0	1.0	10	1.0	10	0.6	10	2.0	10	50	2.50	50
Etrimfos	1.0	1.0	10	0.3	10	3.0	10	1.25	2.5	10	1.25	10
Oxamyl	0.05	0.01	10	0.1	10	0.05	10	1.25	5.0	50	2.50	50
Pencycurone	0.30	0.3	50	0.3	50	1.0	50	0.25	1.75	50	0.25	10
Pyraclostrobin	0.05	0.1	1500	0.3	700	0.1	200	0.25	1.25	100	0.25	50
Spinosad A	1.00	1.0	300	1.0	500	1.0	1000	12.5	25.0	50	12.5	50
Zoxamide	0.30	0.3	50	1.0	20	1.0	20	1.25	1.75	50	1.25	10

* (sum of isomers)

Method validation: LOD, LOQ values

Comparison of LOQ values for different matrices measured with triple quadrupole mass spectrometer and HRAM instrument are shown below and in Table 1. All LOQ values were under or at level of required MRLs.



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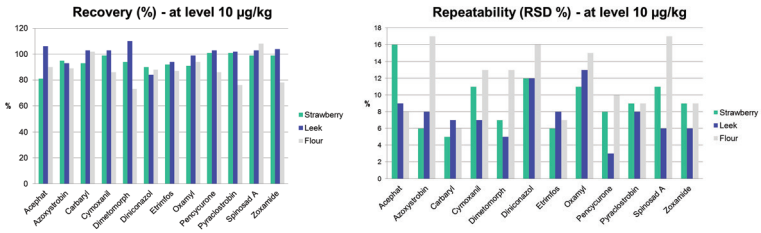
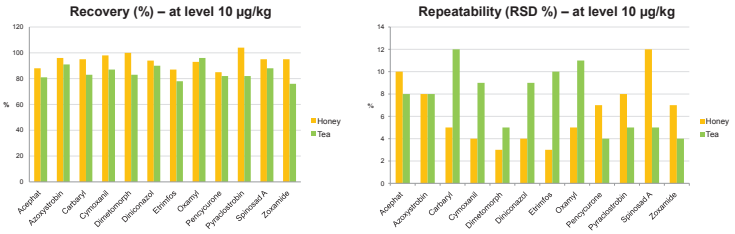
Method validation: Accuracy

Method accuracy was evaluated by measurement of fortified blank samples at low level (10 µg/kg) with six replicates. The results of recovery and repeatability for 12 representative compounds are shown below.



Results from Triple Quadrupole Mass Spectrometer



Results from HRAM Mass Spectrometer

Conclusion

- Both methods were thoroughly tested and capable to be used for routine testing of pesticides residues in various food matrices
- Analytical parameters as linearity, specificity, LOD, LOQ, precision and accuracy were evaluated. For validation fortified homogenized samples were used. The validation results showed satisfactory results for both methods and all of these data are discussed in details in this poster presentation.
- The performance for the majority of target compounds complies with current regulatory requirements. All LOQ values fall under the relevant MRL values.
- Methods are available for easy method transfer.

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