



Fully automated Sample clean-up and Pesticide Screening

With Agilent 6410 LC/MS QQQ online SPE System

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Agilent LC-MS QQQ Online SPE System for Pesticide Residue Screening in Fruit and Vegetables

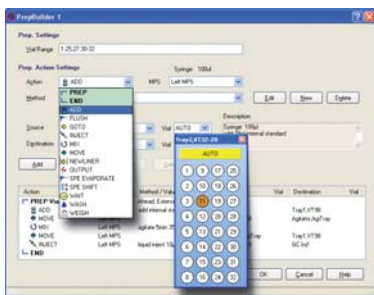
The document describes an effective multi-residue method for the determination of Pesticides in fruit and vegetables needed to ensure food safety. Sample preparation is an important part of the method development. The online SPE/LC/MS/MS multi-residue method enables automated clean-up by SPE immediately followed by the analysis of the pesticide extract. The methods shows good reproducibility, excellent recovery and limits of determination for a wide range of pesticides with different polarities and for different matrices. The procedure can be fully used for routine analysis.

All steps in the sample preparation procedure, including sample introduction, are automated using a GERSTEL MultiPurpose Sampler with an automated SPE accessory. The SPE unit is connected to an Agilent 1200 Rapid Resolution system. MS/MS detection is performed using Agilent 6410 Triple Quadrupole Mass Spectrometer.

Calibration Range: Excellent linearity range from 5 to 200 ng/mL (in matrix) for polar pesticides as well as for more apolar pesticides

Sample types: For a wide range of pesticides with different polarities and different matrices

Sample Throughput: The overall analysis time is 30 min. with parallel sample preparation by online SPE



The SPE system is controlled by the GERSTEL MAESTRO software. All parameters for the various SPE steps are entered by mouse-click in the GERSTEL PrepBuilder. A link to the Agilent MassHunter Software, which controls the LC/MS system and the Data Acquisition, is implemented in MAESTRO Configuration Editor. The Injection volume of the SPE eluate, the MassHunter LC/MS method and the MassHunter Data Folder can be specified in MAESTRO without additional entries in the MassHunter software



Methods and Operation

Automated SPE

15 mL of an acetonitrile/water mixture (80:20) is added to a 5 g sample of fruit or vegetable for extraction. The SPE cartridge (M&N C-18ec; 6 mL, 1 g) is conditioned using 10 mL methanol and 10 mL water. A 5 mL sample is added to the cartridge, which has to be subsequently rinsed with 5 mL water.

Analytes then were eluted using an acetonitrile/water mixture, added at a flow-rate of 600 $\mu\text{L}/\text{min}$, the liquid is not aspirated through the cartridge by applying a vacuum at the outlet, rather it is added under positive pressure using a syringe, so flow and elution speed can be accurately controlled in this way.

Finally the eluate is concentrated under a flow of inert gas for six minutes at 50°C and the concentrate is taken up in 5 mL of an acetonitrile /formic acid mixture (30:70). This automated clean-up step takes around 20 min to complete, so it does not exceed the overall analysis time of the LC/MS/MS run.

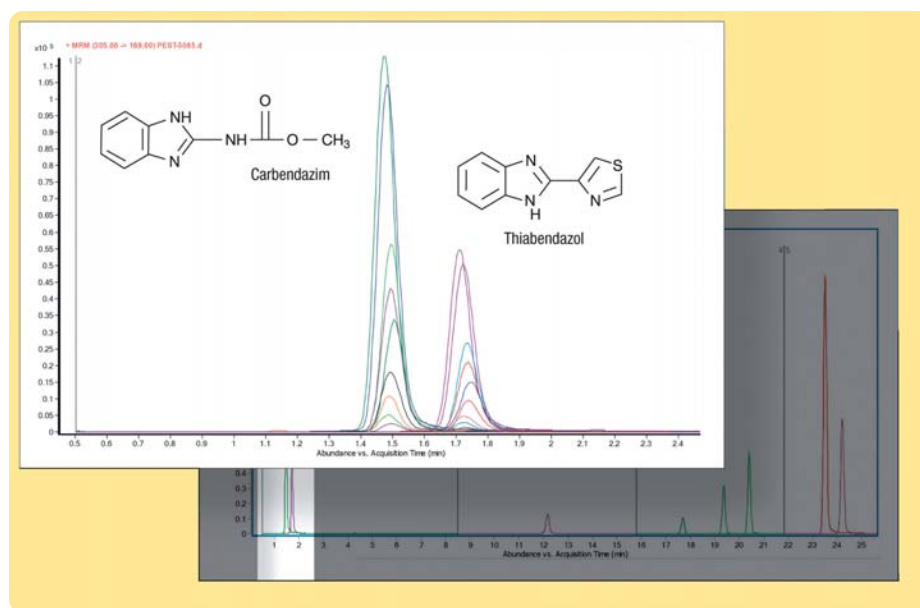


Agilent is supplying a complete package including SPE cartridges and HPLC columns for the application.

LC/MS/MS Method

The total run time required to determine around 130 compounds is in the order of 35 minutes. 20 μL of the cleaned-up extract is introduced directly to the LC/MS/MS-system by the GERSTEL SPE equipped with an injection valve. For the LC method a solvent mixture of 5 mMol formic acid (A) and acetonitrile (B) is used as mobile phase based on the following gradient program: 0 min: 20%B, 5 min: 20% B, 30 min: 90% B. The column (ZORBAX XDB-C18, 100 x 2.1 mm, 1.8 μm) has a temperature of 50°C with a flow-rate of 0.5 mL/min.

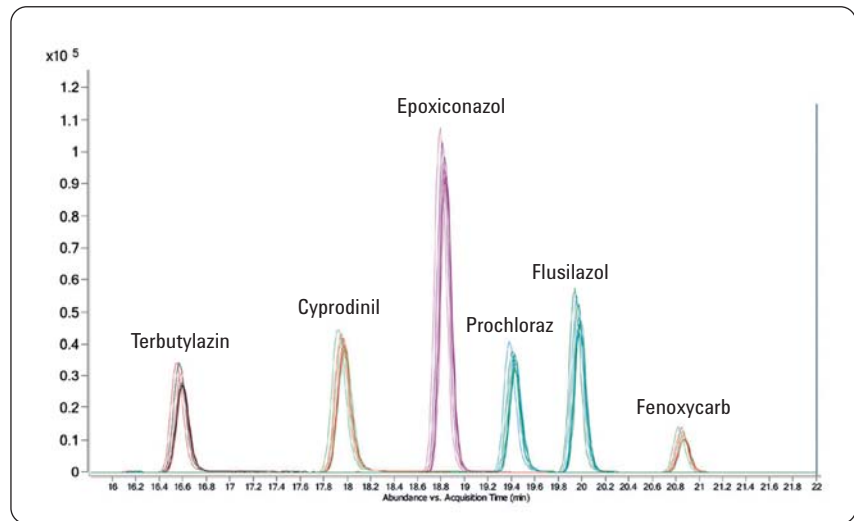
The analytes are detected with an Agilent 6410 Triple Quadrupole Mass Spectrometer using positive electrospray ionization by an ESI source. The source settings are optimized for the defined LC eluent system, and the triple quadrupole instrument is operated in multiple reaction mode (MRM), dividing the run-time in five different time segments, in each segment 40-50 analytes are monitored with two transitions for each pesticide.



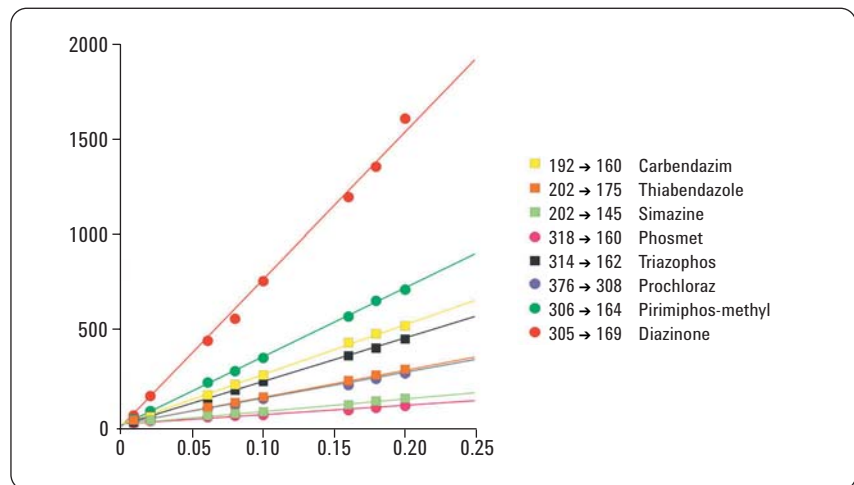
Determination of polar and apolar pesticides respectively in orange oil, overlay chromatograms covering 9 different concentrations.



Herbicide suites covered



Overlay of TIC (medium polarity section) of 8 separate sample preparations and injections of a bell pepper sample spiked with a standard mixture of pesticides (100 ng/mL) each.



Calibration curves for eight pesticides from 5 to 200 ng/mL (in matrix) using the SPE-LC/MS/MS method.

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