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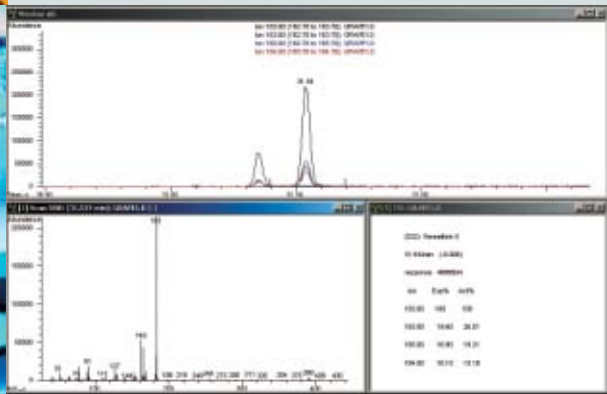


Figure 2 RTL screener for permethrin II

Twister-RTL-Capillary GC-MS.

A versatile method to monitor 440 Pesticides in different matrices like Water, Beverages, Fruits or Vegetables

Pesticide residue analysis is usually costly and time-consuming. Analysis of individual pesticides or classes of pesticides e.g. organochloro pesticides (OCPs), organophosphorus pesticides (OPPs), triazines, etc., is more and more replaced by multiresidue methods. The first aim of a multiresidue method (MRM) is to detect as many pesticides as possible with one analytical technique including sample preparation and chromatographic analysis. Only after elucidation of the pesticides in a specific matrix, accurate and precise quantification can be performed.

In recent years important improvements have been made in multiresidue analysis by capillary gas chromatography – mass spectrometry. Through the features of Electronic Pneumatic Control (EPC), Retention time Locked Libraries (RTL) for GC amenable pesticides and endocrine disruptors could be constructed and by linking the locked retention times to the mass spectral data hardly any pesticide that is in the library can escape detection and elucidation [1,2]. The Agilent Technologies RTL-MS library presently comprises 567 substances.

An MRM also requires sample preparation and numerous methods have been developed for this purpose. We recently evaluated the GERSTEL Twister as sample preparation technique for the enrichment of pesticides from different matrices and came to the

conclusion that 440 pesticides out of the RTL-MS library can be enriched from water, beverages, fruits and vegetables with recoveries complying with the required Limits Of Quantification (LOQ) set by regulatory organizations. The complete list of pesticides amenable to Twister enrichment and RTL-Capillary-GC-MS analysis will be published in one of the next issues of LC.GC Europe [3]. The recent introduction by GERSTEL of the new Twister Desorption Unit TDU that can be installed on the MPS 2 enables fully automated analysis of 98 or 196 Twister stir bars. This makes the application of this new MRM very cost-effective.

The principle and potential of Twister-RTL-Capillary-GC-MS is illustrated with the determination of pesticides in grapes. The sample preparation, valid for all fruits and vegetables, is as follows:

15 gram grapes purchased from a local supermarket were mixed with 30 mL methanol and 1 mL of the extract was diluted with 10 mL water. A Twister was added and stirred for 60 min. The Twister was then desorbed at 280 °C (5 min) in the splitless mode at 50 mL/min and the solutes were cryofocused at -100°C. The GERSTEL CIS operated in the splitless mode was then programmed at 600 °C/min to 280 °C (2 min). Analysis was done on an Agilent Technologies 6890 GC with 5973 MSD combination equipped with a 30 m x 0.25 mm I.D., 0.25

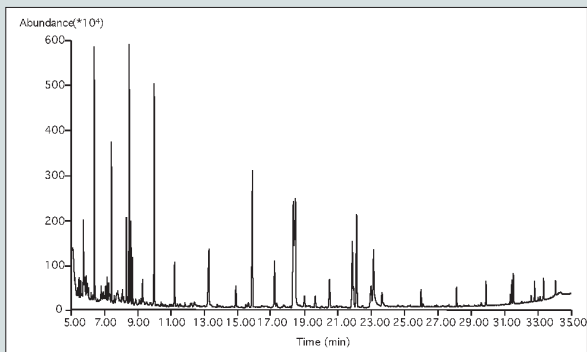


Figure 1 Total ion chromatogram of Twister-RTL-Capillary GC-MS of grapes

μm HP-5MS column. The oven temperature was programmed from 70 °C - 2 min - 25 °C/min - 150 °C - 3 °C/min - 200 °C - 8 °C/min - 300 °C. This is the RTL program and locking was performed on p,p'-DDT (26.999 min). The MS was operated in the scan mode (m/z 40-500).

Figure 1 shows the total ion chromatogram and on this chromatogram the RTL screener was applied.

Figure 2 shows a typical screening result. Permethrin II is identified through its retention time and four specific ions. The total screening then gives all pesticides positively identified in the grapes (Figure 3) and by standard addition the quantities could be measured.

Note that chlorpyrifos is present at the 0.1 ppb level which means that whatever the matrix is e.g. drinking water, beverages, fruits, vegetables, etc., limits of determination can easily be reached in the MS scan mode.

Operation of the MS in the ion monitoring mode allows ppt level measurements which can be of use to check on the pesticide zero-tolerance in baby food.

Acknowledgement

My coworkers and co-authors of the article in LC.GC Europe, F. David (RIC) and B. Tienpont (Ghent University) are thanked for their contribution.

References

1. Ph. Wylie and B. Quimby, Agilent Technologies Application note 5967-5860 E (2000) www.chem.agilent.com
2. C. Kai Meng, Agilent Technologies application note 5988-4392EN (2001) www.chem.agilent.com
3. P. Sandra, B. Tienpont and F. David, submitted LC.GC Europe.

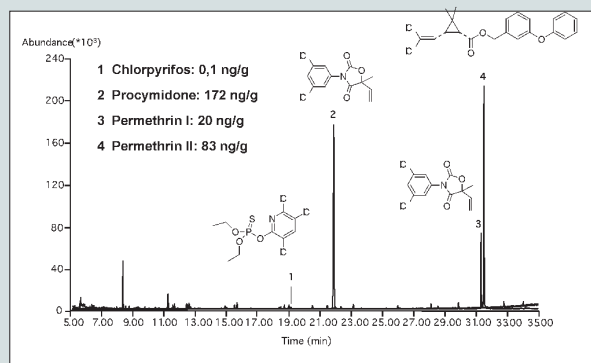


Figure 3 Ion extracted chromatogram of the pesticides positively identified by the RTL screener and quantified in the grapes.

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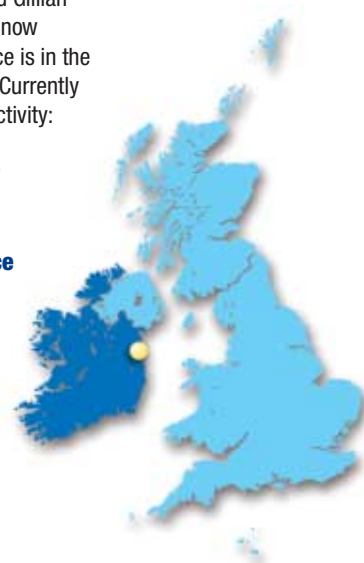
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