APPLICATION BRIEF

Gas Chromatography/
Mass Spectrometry

The Application of GC/MS to the Analysis of Pesticides in Foodstuffs

Introduction

Pesticide contamination of foodstuffs has become a worldwide concern, prompting various levels of regulation and monitoring. Traditionally, pesticides are quantified with gas chromatography (GC) combined with selective detectors (ECD, FID, etc.). Selective GC detectors are great tools to quantify one or two pesticide classes at a time. However, screening for a number of different pesticides requires multiple runs utilizing various GC configurations. Chromatographic run times are often long because of the need to achieve sufficient chromatographic resolution for unambiguous quantification. Gas chromatography/mass spectrometry (GC/MS) provides positive confirmation of various pesticides in a single analytical run; its superior selectivity allows interference-free quantification even with peak coelution. As a result, GC/MS has become a preferred technique for pesticide analysis because of its single-run capability.

This paper outlines a GC/MS method, allowing for the quantification of low-level pesticides with SIM, while simultaneously performing quantification of higher concentrations using full-scan acquisition (SIFI™ – single ion and full ion scanning). It also demonstrates the throughput benefits of fast GC oven cool-down.

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Table 1. **Instrument Conditions.**

Gas Chromatograph:	**PerkinElmer Clarus GC**			**Mass Spectrometer:**	**PerkinElmer Clarus MS**
Analytical Column:	Elite-CLPesticides (30 m x 0.25 mm x 0.25 µm)			GC Inlet Line Temperature:	275 ˚C
				Ion Source Temperature:	275 ˚C
Carrier Gas:	He (30 cm/sec)			Function Type:	SIFI
Injector Temperature:	275 ˚C			Scan Range:	*m/z* 40-450
Injection Type:	Splitless			Scan Time:	0.2 sec
Oven Program:	**Temperature**	**Hold Time**	**Rate**	InterScan Delay:	0.1 sec
	80 ˚C	0 min	20 ˚C/min		
	290 ˚C	4.75 min	end		

Experimental

The PerkinElmer® Clarus® GC/MS with programmable split/splitless injector was used for this application. The instrumental conditions used in this study are summarized in Table 1. Sample volumes of 1.0 µL were injected into the programmable split/splitless injector, incorporating a 2-mm i.d. deactivated fused-silica liner. The injection-port temperature was set at 275 ˚C (isothermal). The capillary column used incorporated a proprietary phase specifically suited for pesticides (Elite-CLPesticides) with the dimensions of 30 m x 0.25 mm x 0.25 µm df. The helium carrier gas was programmed with a constant velocity of 30 cm/sec. The oven-temperature program was initially set at 80 ˚C with no hold and ramped to 290 ˚C at 20 ˚C/min with a hold of 4.5 minutes. The total oven program is 15 minutes, with an injection-to-injection time of less than 20 minutes.

The MS method contained multiple SIM functions overlapped by a *m/z* 40 to *m/z* 450 full-scan function.

The timing and selected ions of each individual SIM function are dependent on the elution time and fragmentation of each pesticide of interest. The mass spectrometer transfer line and ion source were heated to 275 ˚C.

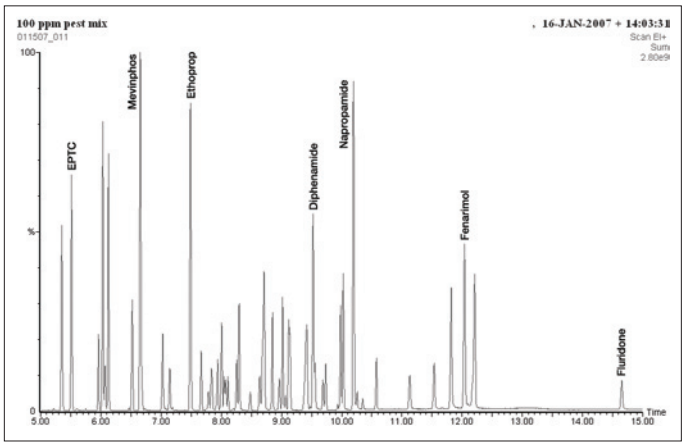

Figure 1. Extracted ion chromatogram of 100-ppm pesticide standard.

Results and discussion

The maximum allowable level of pesticide residues in foodstuffs varies between countries. Japan, for example, has set a low level of 0.01 ppm. In this study, analytical standards comprised of organochlorine, organonitrogen and organophosphorous pesticides were analyzed between 0.01 ppm and 100 ppm.

The standards analyzed here contain over 50 pesticides, of which 25 are pictured in Figure 1. The chromatogram shown in Figure 1 is a composite of extracted ions from the full-scan acquisition of a 100-ppm standard.

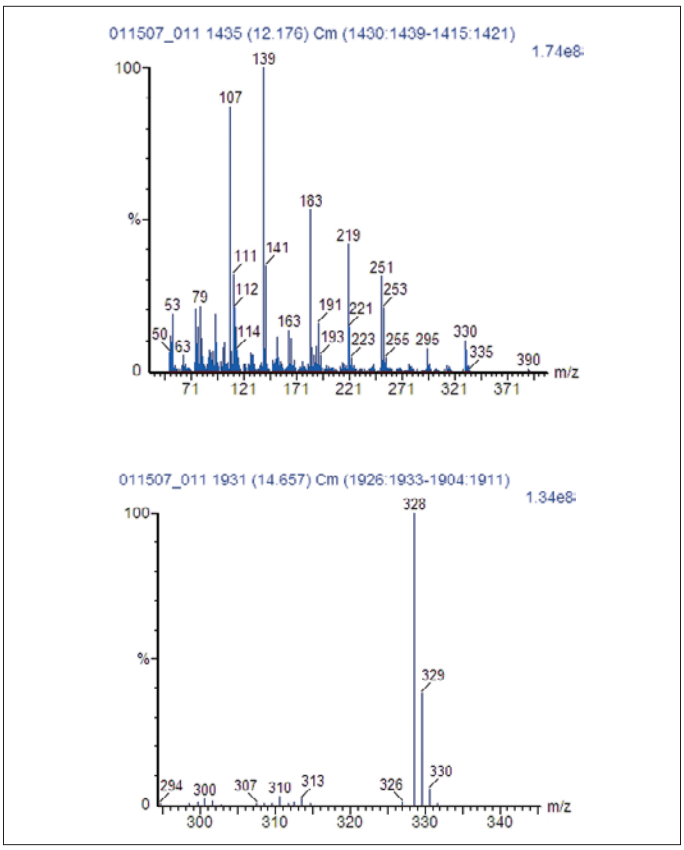

Figure 2. Background subtracted spectra of Fenarimol (top) and Fluridone (bottom).

The spectral data provided by the mass spectrometer allows for the use of chromatographic conditions that resolve only peaks with similar spectra, allowing for faster oven programs and short analysis times. Pictured in Figure 2 are the background-subtracted spectra of Fenarimol and Fluridone. As you can see, the spectra of Fenarimol are quite complex and fragment into many different ions, while Fluridone is quite simple with only three major ions. When analyzing for pesticides with high levels of fragmentation, such as Fenarimol, achieving detection limits may become a challenge. The SIFI capabilities of the Clarus MS will aid in overcoming these challenges. Additionally, SIFI will allow the acquisition of both full scan and SIM data simultaneously; the full-scan data will give library-searchable spectra, while SIM will allow for low-level quantitation.

Conclusion

The adaptation of pesticide screening of foodstuffs from multiple GC analyses to a single GC/MS analysis will allow faster analysis of a wide range of pesticide classes. The Clarus GC/MS provides a robust and efficient platform to perform this analysis. Its novel oven design and best-in-class cooling reduce the injection-to-injection time to less than 20 minutes. The injection-to-injection time was compressed with the use of injection pre-rinse, which allows the autosampler to begin its solvent and sample-rinse steps prior to the GC Ready signal, and together with the best-in-class cooling of the Clarus GC.

The SIFI mass spectral data allow positive confirmation and quantification of various classes of pesticides in a single analytical run. Furthermore, GC/MS data will not only allow for the positive identification of known peaks, but will also provide the analyst the ability to execute a library search of unknown peaks.

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