

Analysis of Organophosphorus Pesticide Residues in Agricultural Products

Concern about food safety is increasing. Particularly, an increasing attention is paid to pesticide residues in agricultural products due to recent reports of the detection of pesticides in imported vegetables and fruits that exceed the regulated levels.

Application News G215 introduced the analysis of organophosphorus pesticides. Pesticides whose concentrations exceed the regulated value should be further verified using GC/MS. This Application News introduces examples of this verification test using GC/MS for samples pretreated in accordance with the official methods. Please refer to G215 for more details regarding the sample pretreatment procedures.

Fig. 1 shows the total ion chromatogram (TIC) for a scan measurement of a 1mg/L standard organophosphorus pesticide mixture. Fig. 2 shows the SIM chromatograms of organophosphorus pesticides at 0.1mg/L.

The upper chromatograms show the quantitation ions and the lower show the reference ions. Fig. 3 shows the calibration curves for diazinon, chlorpyrifos and EPN (0.1 - 1mg/L). Analyses were carried out on spinach and soybeans spiked with a standard organophosphorus pesticide mixture. Fig. 4 shows the SIM chromatogram of a spinach sample where pesticides were added to concentrations of 0.05µg/g. Fig. 5 shows the SIM chromatogram of a soybean sample where pesticides were added to concentrations of 0.1µg/g.

For spinach, chlorpyrifos (5) at $m/z = 197$, marathion (7) at $m/z = 93$, and EPN (10) at $m/z = 169$ were affected by impurities, so their reference ions were eliminated. The quantitation ions were analyzed without being affected by impurities for all substances in both samples.

Reference:
Summary of Food Sanitation Laws and Regulations
2003 edition, Shinnippon-Hoki Publishing (2002)

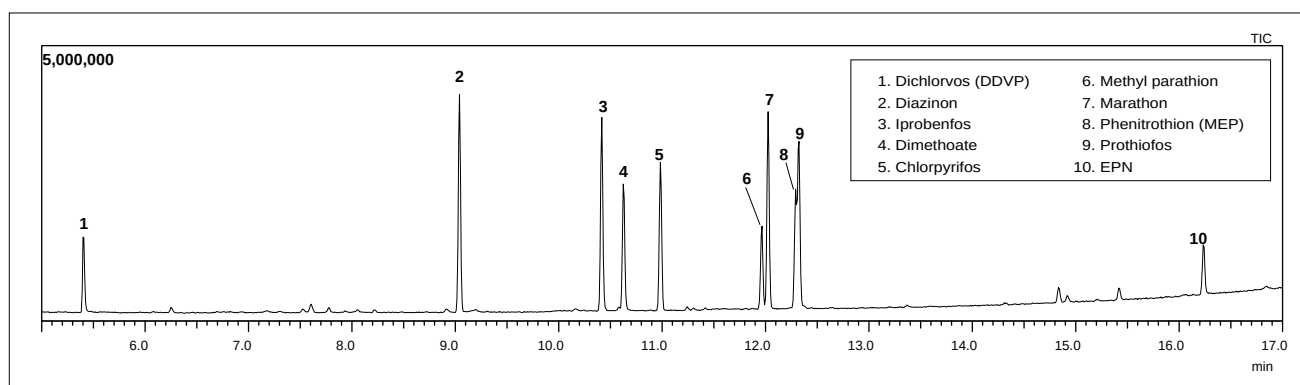


Fig.1 Scan TIC of Organophosphorus Pesticides Standard Solution(1mg/L)

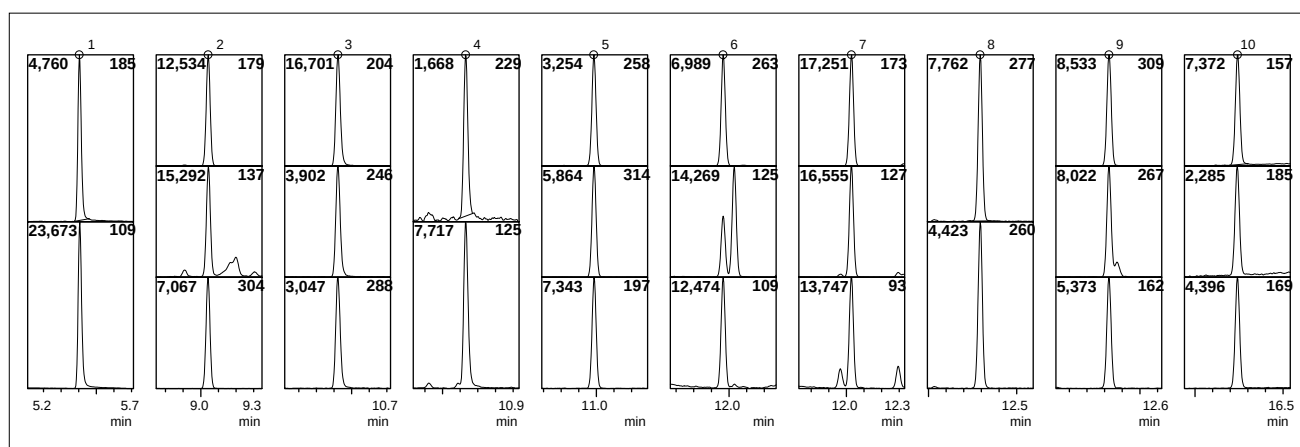


Fig.2 SIM Chromatograms of Organophosphorus Pesticides in Standard Solution (0.1mg/L)

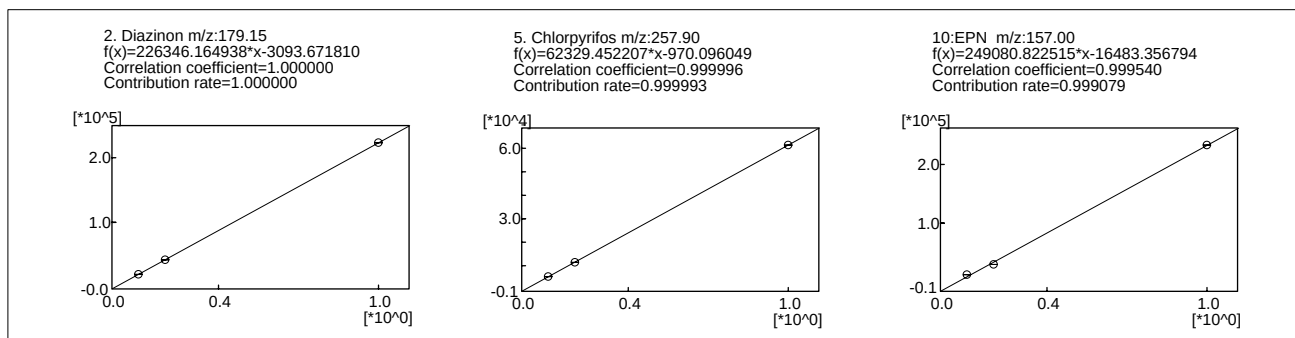


Fig.3 Calibration Curves of Diazinon,Chlorpyrifos and EPN(0.1~1mg/L)

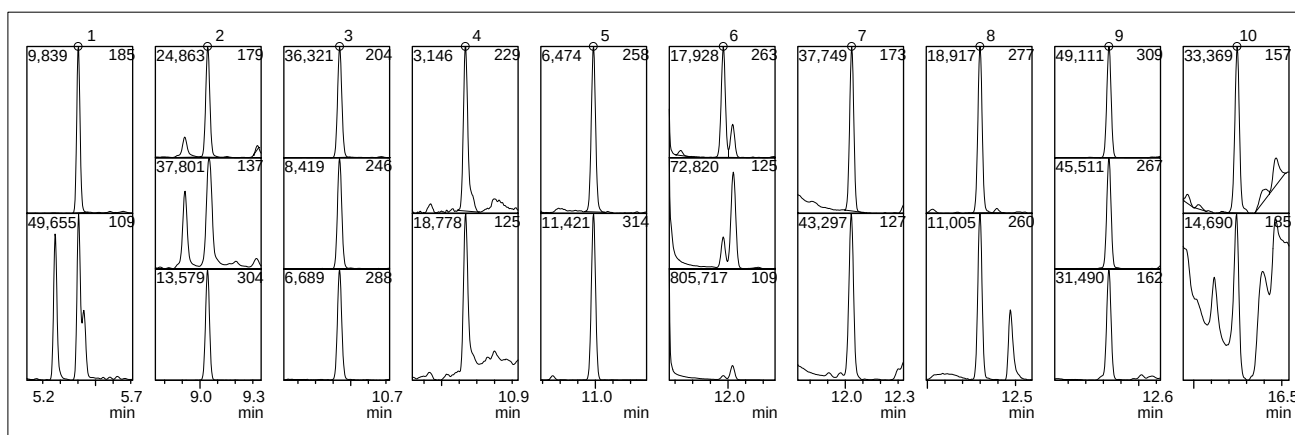


Fig.4 SIM Chromatograms of Pesticide Standards added to Spinach Extract (0.05µg/g equiv.)

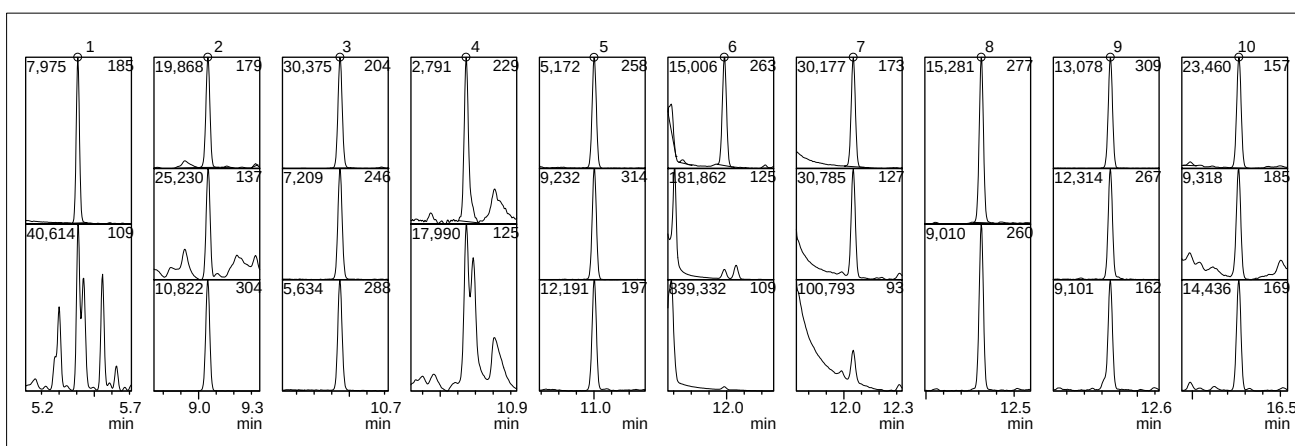


Fig.5 SIM Chromatograms of Soy-beans Extracts which added Pesticides Standard(equivalent to 0.1µg/g)

Table 1 Analytical Conditions

Model	: GCMS-QP2010		
-GC-			
Column	: Rtx-200 30m×0.32mm I.D. df=1.5µm	High Press.Injection	: 120kPa(1min)
Col.Temp.	: 70°C(1min)-25°C/min-125°C-10°C/min -280°C(30min)	Inj.Temp.	: 260°C
Carrier Gas	: He,55kPa(57.8cm/sec;Constant Liner Velocity Mode)	Injection Method	: Splitless(1min)
		Injection Volume	: 1µL
-MS-			
I.F. Temp.	: 280°C	Scan Range	: 40~500
I.S. Temp.	: 200°C	Scan Interval	: 0.5sec
Ionization	: EI	SIM Interval	: 0.2sec



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