

Stability in quantitative analysis of residual agricultural chemicals in food by GC-MS/MS - Stability verification of pesticides in spinach extract -

Product : Mass Spectrometer (MS)

As "food safety" is recognized as an increasingly important issue on a global scale, many nations have their own regulations on residual agricultural chemicals in food. In Japan, the positive list system, which was enforced at the end of May 2006, stipulates a uniform standard of 10 ppb as a quantity that is considered safe for human health. Under the positive list system, more agricultural chemicals need to be examined, and as a result, techniques capable of accurately and collectively analyzing residual agricultural chemicals in food are in increasing demand. While mass spectrometry (MS) is known for its high detection sensitivity, MS/MS is becoming the mainstream of pesticide analysis for its superior sensitivity and selectivity.

The JMS-TQ4000GC, JEOL's latest GC-MS/MS, has a unique ion storage/ejection mechanism within the MS/MS collision cell and incorporates new firmware to support MS/MS analysis with up to 36,000 transitions. In this work, we report the stability of 8 pesticides that were added to spinach extract.

[Sample and Method]

For the sample, 15 g of spinach was processed by using AOAC 2007.01 extraction method, and the resulting extraction solution was mixed with an 8 component 100 ppb standard solution at 9:1. Table 1 shows the measuring conditions used for the analysis.



GC-MS/MS, JMS-TQ4000GC

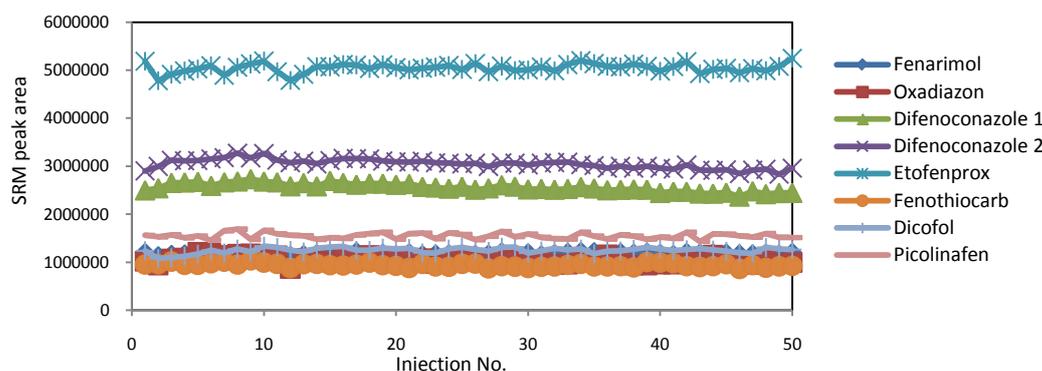
Table 1 Measurement Conditions

[GC-TQMS condition]

System	JMS-TQ4000GC (JEOL)
Ionization mode	El+: 70eV, 50μA
GC column	VF-5ms(Agilent), 30m x 0.25mm, 0.25μm
Oven temp.	50°C (1min)→25°C/min→125°C →10°C/min→300°C
Inlet temp.	250°C
Inlet mode	Splitless, 2μL
He flow	1.0mL/min (Constant Flow)
MS/MS mode	Peak Dependent SRM

[Results and Discussion]

Fig. 1 shows the reproducibility (n = 50) of the SRM chromatographic peak area of the 8 pesticides (10 ppb) added to the spinach extract. And Fig. 2 shows overlaid SRM chromatogram for all 50 injections. The peak area reproducibility for each pesticide was less than CV10%, thus showing strong measurement stability. These results confirmed that the JMS-TQ4000GC shows highly reproducible results for agricultural chemicals.



Compound	SRM peak area ave.	CV (%)
Fenarimol	1168018	1.7
Oxadiazon	1047475	7.1
Difenoconazole 1	2558083	3.4
Difenoconazole 2	3047319	3.2
Etofenprox	5045185	1.9
Fenothiocarb	938218	4.3
Dicofol	1247254	4.6
Picolinafen	1547959	3.8

Fig.1 Reproducibility of SRM chromatographic peak area for the 8 pesticides in spinach extract solution

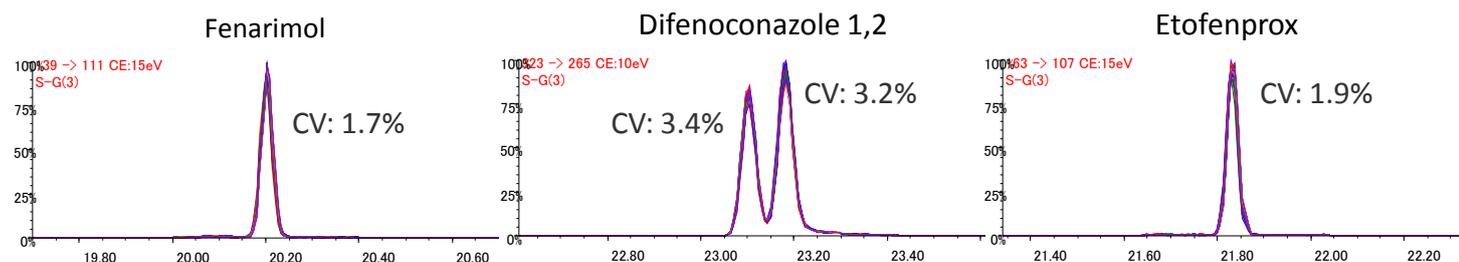


Fig.2 Overlaid of the 50 SRM chromatograms in spinach extract solution

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