

## Quantitative analysis of residual agricultural chemicals in food by GC-MS/MS - Quantitative analysis 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 performed quantitative analysis of residual agricultural chemicals in spinach extract using a JMS-TQ4000GC.

#### [ Sample and Method ]

A pesticide standard solution from FUJIFILM Wako Pure Chemical Corporation (PL series) was used that consisted of equal amounts of PL 1, 2, 3, 4, 5, 6, 11, and 12. Afterwards, the solution was diluted to 1, 5, 10, 50 and 100 ppb. PEG 300 was used to protect the pesticides from thermal decomposition in the GC injection liner.

For the sample, 15 g of spinach was processed by using AOAC 2007.01 extraction method, and the resulting extraction solution was mixed with 100 ppb of the standard solution at 9:1. The sample was quantitatively analyzed for 150 pesticides. Table 1 shows the measurement conditions used for the analysis.

Table 1 Measurement Conditions

[GC-TQMS condition]	
System	JMS-TQ4000GC (JEOL)
Ionization mode	EI+: 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



GC-MS/MS, JMS-TQ4000GC

#### [ Results and discussion ]

Figure 1 top row shows the data acquired from the original spinach extract while the bottom row shows the data from the spinach extraction solution with the pesticides added. MS/MS, with its high mass selectivity, detected agricultural chemicals without being affected by contaminants in the spinach extract. Table 2 shows the quantitative results of 150 pesticides (n=5) and their reproducibility (CV). For most agricultural chemicals, the recovery rate was 70 to 120% and the CV was 10% or less, demonstrating the effectiveness of the JMS-TQ4000GC for pesticide analysis.

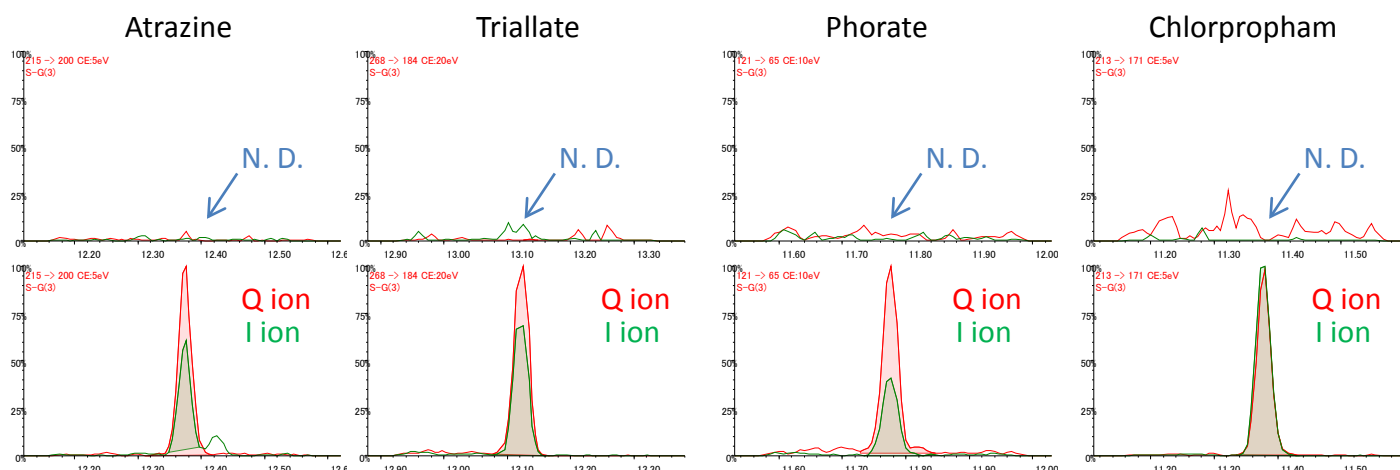


Fig.1 SRM chromatograms,  
upper: Spinach extract solution only, lower: 10ppb pesticides in the spinach extract solution

