SHIMADZU
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Application Data Sheet

No.107

GC-MS
Gas Chromatograph Mass Spectrometer

Simultaneous Analysis of 477 Residual Pesticides in Agricultural Crops Using GC-MS/MS - Part 2

Application Data Sheet No. 106 showed that it is possible to simultaneously analyze 477 components with high sensitivity and high accuracy by using a measurement program created using Smart MRM. However, there were still cases where matrix interference was unavoidable even when using highly selective MRM analysis. Therefore, this Application Data Sheet presents results from analysis using two columns with different separation characteristics: a general-purpose 5 % phenyl / 95 % methylpolysiloxane column and a trifluoropropyl methyl polysiloxane column.
By using the Twin Line MS System, both of these columns can be installed in the same GC-MS/MS system at the same time for continuous analysis without having to release the vacuum or replace columns.

Experiment

Matrix solutions were prepared by processing soy bean, orange, brown rice, and spinach samples according to a pretreatment procedure for residual pesticide analysis, and then purifying them using the GPC Cleanup System (from Shimadzu Corporation).[1)] Measurement sample solutions (1 g/mL sample concentration) were then prepared by spiking the prepared matrix solutions with 477 components (including internal standard substances) to a concentration of 5 ppb (or 200 ppb for the internal standard substances). 19 kinds*1 of surrogate pesticides were used as the internal standard substances.
The GCMS-TQ8040 combined with the Twin Line MS System was used to measure samples based on the analytical conditions listed in Table 1. Two transitions were specified for each component, one for quantitation and the other for confirmation, and Smart MRM was used to automatically create a measurement program.

Table 1: Analysis Conditions

GC-MS:	GCMS-TQ8040 (Twin Line MS System)
Column 1:	SH-Rxi-5Sil MS (30 m L., 0.25 mm I.D., df=0.25 µm) (Shimadzu, P/N: 221-75954-30)
Column 2:	SH-Rtx-200 MS (30 m L., 0.25 mm I.D., df=0.25 µm) (Shimadzu, P/N: 221-75811-30)
Glass Insert :	Sky Liner, Splitless Single Taper Gooseneck w/Wool (Restek, P/N: 567366)

[GC]		[MS]	
Injection Temp.:	250 °C	Interface Temp.:	300 °C
Column Oven Temp.:	60 °C (1 min) → (25 °C /min) → 160 °C → (4 °C /min) → 240 °C → (10 °C /min) → 290 °C (11 min)	Ion Source Temp.:	200 °C
		Measurement Mode:	MRM
Injection Mode:	Splitless	Loop Time:	0.4 sec
High Pressure Injection:	250 kPa (1.5 min)	Processing Time Required:	0.3 min
Carrier Gas Control:	Linear Velocity (40.0 cm/sec)		
Injection Volume:	2 µL		

Fig. 1: GCMS-TQ8040 with Twin Line MS System

Analysis Results

Results from analysis using columns 1 and 2 are shown in Figs. 2 and 3. Due to matrix interference, some pesticide peaks cannot be detected properly with column 1, but using column 2 allows separation of the matrix and results in accurate detection. Furthermore, high-precision analytical results can be obtained even when using column 2.
If a peak is detected in data from column 1, then the data from column 2 can be used to confirm that the peak is from a pesticide.

1) E. Ueno, et al., *J. AOAC INT.* **87**, (2004) 1003-1015

*1 Dichlorvos-d_6, acephate-d_6, diazinon-d_{10}, iprobenfos-d_7, carbaryl-d_7, fenitrothion-d_6, linuron-d_6, metolachlor-d_6, chlorpyrifos-d_{10}, diethofencarb-d_7, fosthiazate-d_5, pendimethalin-d_5, thiabendazole-$^{13}C_6$, imazalil-d_5, isoprothiolane-d_4, isoxathion-d_{10}, EPN-d_5, etofenprox-d_5, and esfenvalerate-d_7

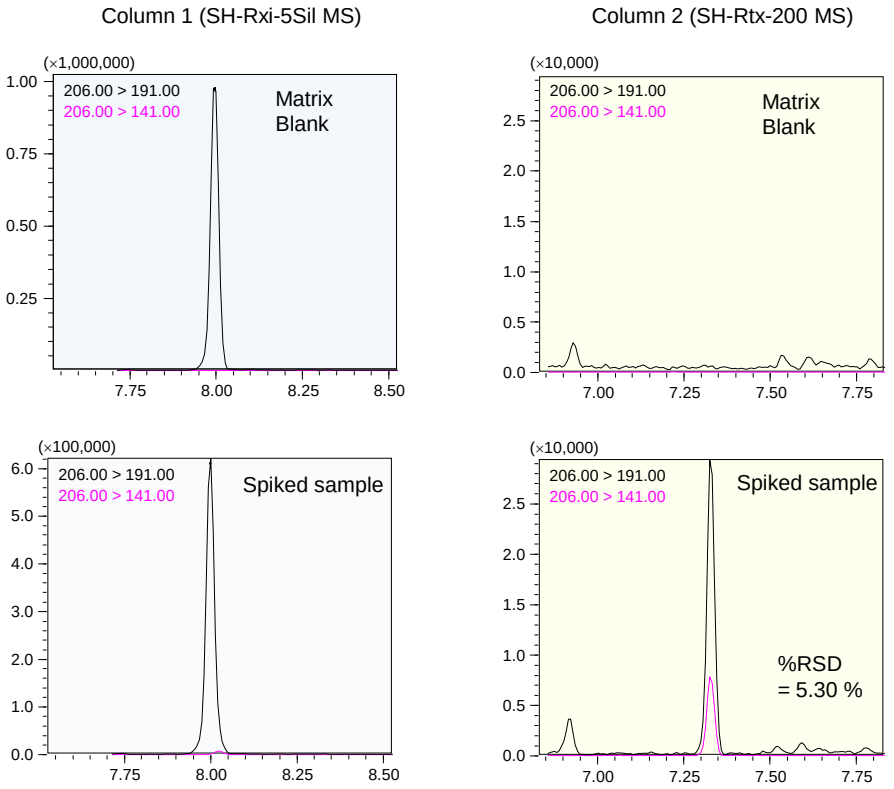

Fig. 2: MRM Chromatograms of Chloroneb in a Soy Bean Sample Using Column 1 (left) and Column 2 (right)

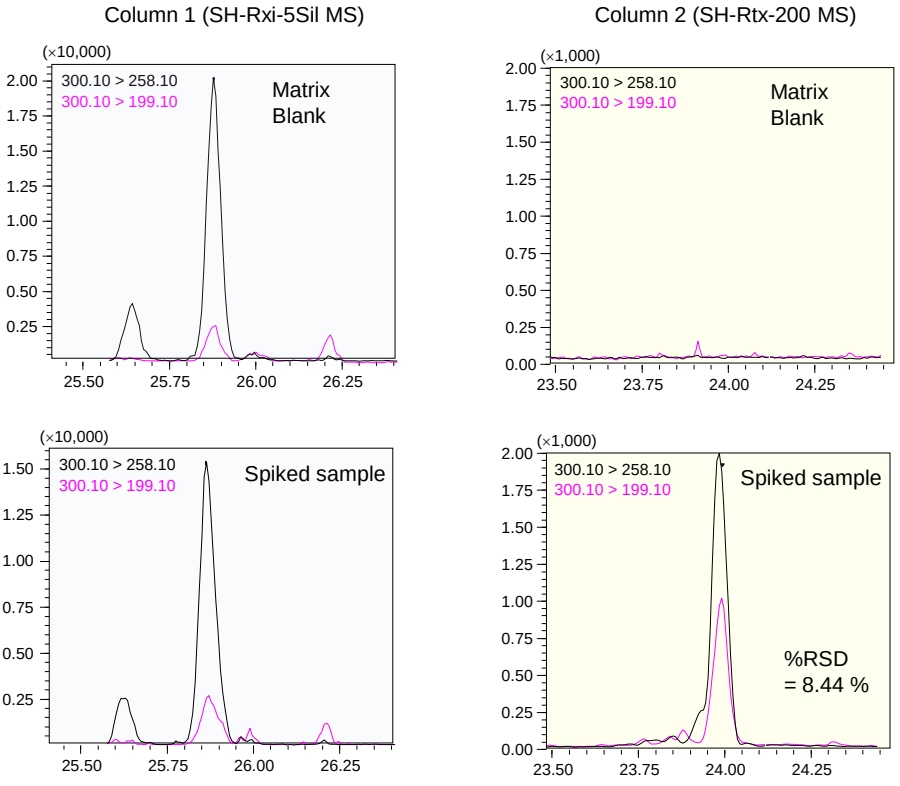

Fig. 3: MRM Chromatograms of Bifenazate in a Brown Rice Sample Using Column 1 (left) and Column 2 (right)

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