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Application Data Sheet

No.92

GC-MS
Gas Chromatograph Mass Spectrometer

Screening of residual pesticides in food with two different columns

In recent years, with increases in the number of pesticides and the diversification of substances under investigation, there have been calls for quick and high-accuracy screening for residual pesticides in foods using GC-MS.

The Quick-DB database includes mass spectra, retention times, and calibration curves for 478 pesticide components. It can be used to quickly calculate semi-quantitative values without requiring analytical standards.
If a pesticide peak is detected, it is essential to check for potential interference from co-eluting contaminants to insure highly accurate screening. One of the ways for checking is to analyze the samples with two columns with different types of stationary phase. For this purpose, the Twin Line MS system is very useful because it enables the installation of two types of columns to one MS.
The Quick-DB contains information compatible with two different columns, so that it can also be applied to the Twin Line MS system. In this Data Sheet, residual pesticides in foods were analyzed by applying Quick-DB and the Twin Line MS system.

Experiment

Commercially-available oranges and soya beans were processed with the QuEChERS method using Restek Q-sep™. A mixture of 138 pesticides was added to the sample solutions, with the concentrations adjusted to 10 ng/mL. The pesticide-spiked samples were subjected to Scan/SIM analysis using the analysis conditions stored in Quick-DB. Frequently detected components were analyzed with high sensitivity in SIM mode. For components with low detection frequency, a comprehensive analysis was performed in Scan mode. Table 1 shows the analysis conditions.

Table 1: Analysis Conditions

GC-MS:	GCMS-QP2010 Ultra with Twin Line MS System		
Column 1:	Rxi-5Sil MS (30 m L., 0.25 mm I.D., df=0.25 µm) (Restek Corporation, P/N: 13623)		
Column 2:	Rtx-200 MS (30 m L., 0.25 mm I.D., df=0.25 µm) (Restek Corporation, P/N: 15623)		
Glass Insert:	Sky Liner, Splitless Single Taper Gooseneck w/Wool (Restek Corporation, P/N: 567366)		
[GC]		[MS]	
Injection unit 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
		Solvent elution time:	1.5 min
Injection mode:	Splitless	Measurement mode:	FAAST (Scan/SIM simultaneous measurement)
High-pressure injection:	250 kPa (1.5 min)		
Carrier gas control:	Linear velocity (40.0 cm/sec)	Scan mass range:	*m/z* 50 to 600
Injection volume:	2 µL	Scan event time:	0.15 sec
		Scan speed:	5,000 u/sec
		SIM event time:	0.3 sec

<Twin Line MS System>
The inlets of the two different columns are connect to two different injection ports and the outlets are introduced directly to the mass spectrometer interface at the same time. One column is chosen for analysis while the other, non-used column, has a reduced carrier gas flow rate. This enables application data from the different columns to be acquired without venting the MS vacuum to change columns. Moreover the retention times and retention indices are the same as a single column system. A high-capacity differential vacuum system provides the same sensitivity as that obtained by a single column system.

Analysis Results

The pesticide-spiked samples (10 ng/mL) were analyzed using the Twin Line MS and the data were processed utilizing the Quick-DB. Figs. 1, 2, and 3 show the obtained mass chromatograms for 3 selected compounds. The left shows those run on the Rxi-5Sil MS and the right shows those on the Rtx-200 MS. Carbaryl and Aldrin peaks were impacted by close- or co-eluting contaminants on the Rxi-5Sil MS (Fig. 1 and 2 (left side)). For these two compounds, the calculated semi-quantitative values obtained from the calibration curves stored in the Quick-DB were higher than the spike amount (10.0 ng/mL) because of the interference. However, on the Rtx-200MS (on the right), the co-eluting contaminants were chromatographically separated from Carbaryl and Aldrin peaks (Fig. 1 and 2 (right side)), and the semi-quantitative values were much more closer to 10 ng/mL spike levels. Pyrimethanil were not impacted by co-eluting contaminants with either of the columns. The results obtained from two different columns enhances the reliability of screening results.
These results demonstrated that the Twin Line MS allows the reliable determination of pesticides in complex samples with the potential for matrix interference, such as processed foods. This works for a semi-quantitative values using the Quick-DB.

Carbaryl

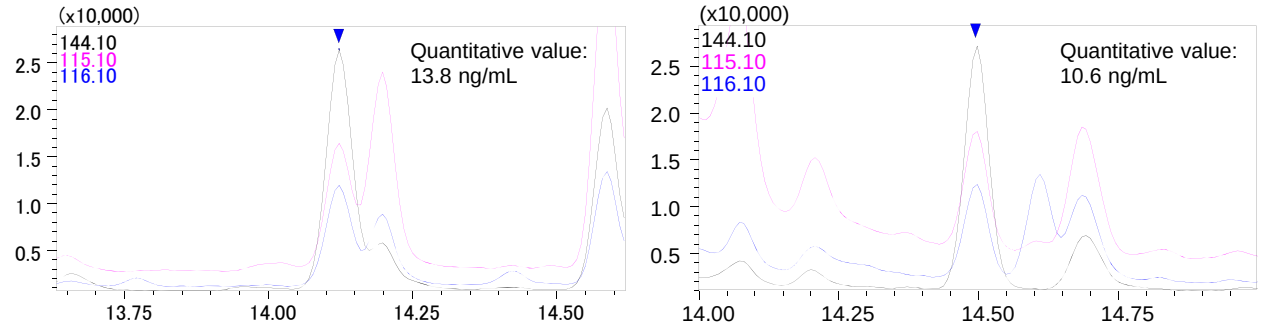

Fig. 1 Mass Chromatogram of Carbaryl (10 ng/mL) Added to Liquid soya beans Extract (Left: Rxi-5Sil MS; Right: Rtx-200 MS)

Aldrin

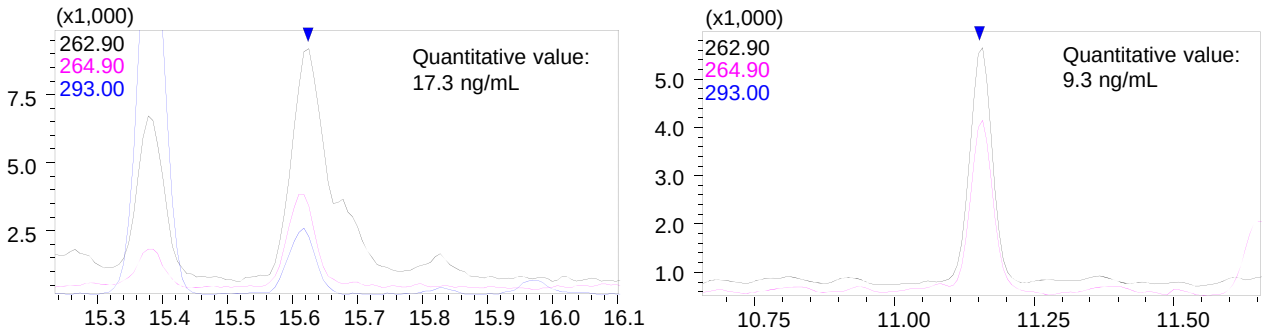

Fig. 2 Mass Chromatogram of Aldrin (10 ng/mL) Added to Liquid Orange Extract (Left: Rxi-5Sil MS; Right: Rtx-200 MS)

Pyrimethanil

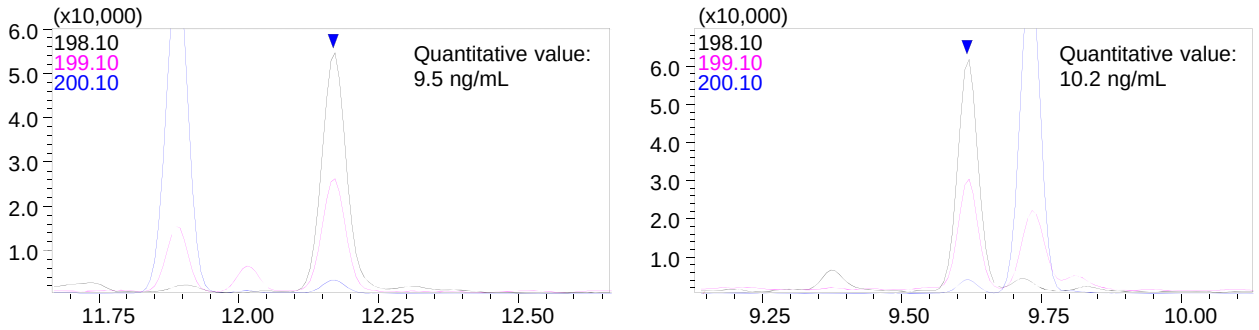

Fig. 3 Mass Chromatogram of Pyrimethanil (10 ng/mL) Added to Liquid soya beans Extract (Left: Rxi-5Sil MS; Right: Rtx-200MS)

First Edition: November, 2013

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