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Application Data Sheet

No.138

GC-MS
Gas Chromatograph Mass Spectrometer

Efficient Analysis of Residual Pesticides in Foods Using High-Sensitivity GC-MS/MS

In the analysis of residual pesticides in foods, periodic maintenance is required. This includes the replacement of insert liners and column cutting due to the impact of impurities originating in the foods. However, because of issues such as the time for maintenance procedures and the cost of consumables, there is a need to reduce the frequency of maintenance as much as possible.
The GCMS-TQ8050, a high-sensitivity GC-MS/MS, can detect trace ions with high sensitivity, so high quantitative accuracy is obtained even for trace components. Using the high-sensitivity TQ8050, existing lower limits of quantitation can be obtained with small injection volumes. Further, reducing the injection volume lessens the analysis burden on parts such as insert liners, columns, and the ion source, which, in turn, reduces the frequency of maintenance. This Application Data Sheet presents the analysis results of pesticide standard samples and actual tomato samples, under conditions in which the injection volume is reduced to 1/4 the conventional amount, using the GCMS-TQ8050.

Fig. 1: GCMS-TQ8050,
a High-Sensitivity GC-MS/MS

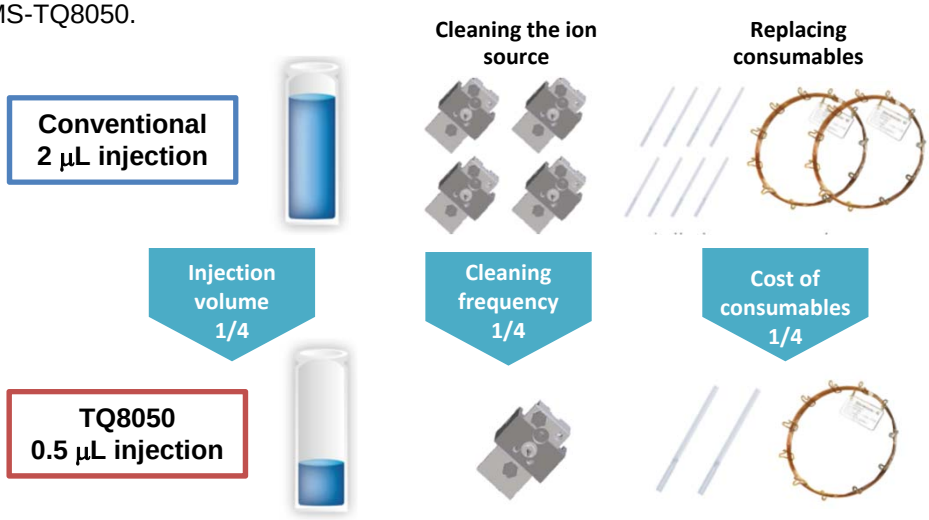


Fig. 2: Reduced Maintenance Frequency Due
to Reduced Injection Volume

Experiment

As the standard samples, pesticide standard mixed solutions (Hayashi Pure Chemical Ind. Ltd. PL2005 Pesticides GC/MS Mix I, II, III, IV, V, VI, and 7) were prepared to achieve final concentrations between 5 ng/mL and 100 ng/mL. At this point, as a virtual matrix, polyethylene glycol 300 was added to ensure a concentration of 200 µg/mL. In addition, as an actual sample for the spike recovery test, the pesticide standard mixed solution was spiked with a tomato extract solution to ensure a concentration of 10 ng/mL.
Smart Pesticides Database Ver. 2 was used to create the analytical method. In this experiment, in order to reduce the injection volume, the analysis was performed with a 0.5 µL injection using a 5 µL syringe.

Table 1 Analytical Conditions

GC-MS:	GCMS-TQ8050
Column:	SH-Rxi®-5Sil MS (30 m long, 0.25 mm I.D., df = 0.25 µm) (Shimadzu GLC, P/N 221-75954-30)
Glass Insert:	Topaz single taper with wool (Shimadzu GLC, P/N 23336)
Syringe for Injection:	AOC-20i 5 µL syringe (Shimadzu GLC, P/N: 221-75173)

GC	
Injection Port Temp.:	250 °C
Column Oven Temp.:	50 °C (1 min) → (25 °C /min) → 125 °C → (10 °C /min) → 300 °C (15 min)
Injection Mode:	Splitless
High-Pressure Injection:	250 kPa (1.5 min)
Injection Volume:	0.5 µL
Carrier Gas Control:	Linear velocity (47.2 cm/sec)

MS	
Interface Temp.:	250 °C
Ion Source Temp.:	230 °C
Ionization Method:	EI
Measurement Mode:	MRM

Analysis Results

The injection volume was reduced to 1/4 the conventional amount, and a 5 ng/mL standard sample was analyzed repeatedly (N = 5). Both sensitivity and repeated analysis accuracy were confirmed. Fig. 3 shows the distribution of area repeatability for a total of 387 components. Favorable results were obtained, with a %RSD of 10 % or less for 95 % of the components, and 20 % or less for 98 % of the components.
In addition, pesticides were spiked to the actual tomato sample, and the quantified values in the actual sample were confirmed. Fig. 4 shows the distribution of the recovery rates calculated from these quantitative results, and Fig. 5 shows typical pesticide chromatograms. For approximately 80 % of the components, a favorable recovery rate between 70 % and 120 % was obtained.
From these results, it is evident that using the high-sensitivity GC-MS/MS, sufficient quantitative accuracy can be obtained even at low concentrations, even when the injection volume is reduced. Reducing the injection volume reduces the frequency of instrument maintenance, enabling more efficient operation.

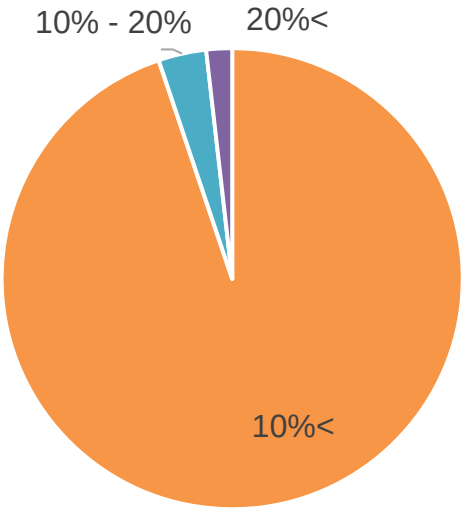


Fig. 3: Distribution of Area Repeatability (%RSD) for a 5 ppb Pesticide Standard Sample

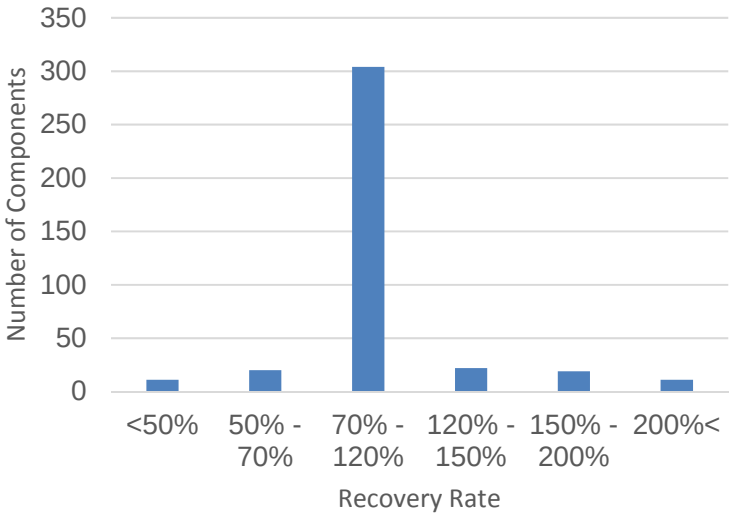
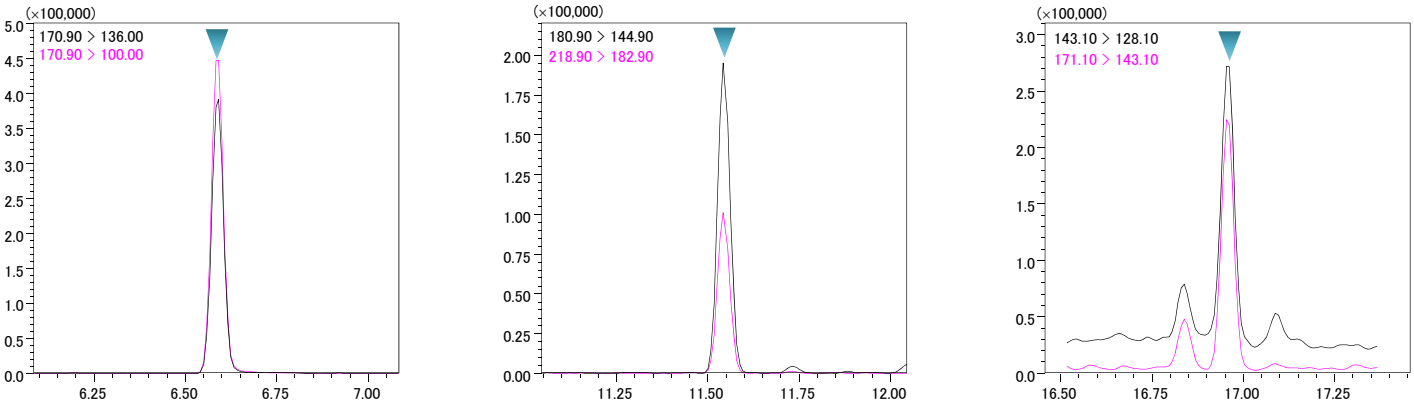


Fig. 4: Distribution of Spike Recovery Rates for an Actual Tomato Sample



Dichlobenil　　delta-BHC　　Resmethrin-1, 2

Fig. 5: Typical Chromatograms for Pesticides in an Actual Tomato Sample

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