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GC-MS GCMS-QP2050

Application News

Simultaneous Analysis of Residual Pesticides Using High-Speed Scan and Smart SIM+ of GCMS-QP2050

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User Benefits

- Fast Automated Scan/SIM Type (FASST) functionality enables quantitative analysis in SIM mode and qualitative analysis in scan mode with a single measurement.
- The industry's fastest scan speed of 30,000 u/sec prevents quantitation accuracy losses even for FASST measurements.

■ Introduction

Residual pesticide regulations have been tightened due to growing global interest in food safety and security. The introduction of a positive list system in Europe, the U.S., and Japan has resulted in growing needs for simultaneous analysis of more than several hundred pesticides. Shimadzu GCMS-QP2050 gas chromatograph mass spectrometers offer the industry's highest level of sensitivity, scan speed, and durability, making them ideal for simultaneous analysis in various industries, such as for residual pesticide analysis.

Fig. 1 GCMS-QP2050 + AOC™-30i/20s U

■ Smart SIM+ and High-Speed Scan Analysis

GCMS-QP2050 systems include a Smart SIM+ function for automatically creating optimized SIM methods. That function sets optimal MS measurement times based on the retention time of each component.

Fig. 2 compares the MS measurement times using Smart SIM+ and conventional Smart SIM. Smart SIM+ can provide enough dwell time (the data acquisition time for each component) for even coeluted peaks, because it only measures data near the expected retention time. Therefore, highly reproducible data are obtained without signal reduction by acquiring data from a sufficient number of ions even if the number of target components are increased.

In contrast, it is difficult to simultaneously analyze all types of pesticides in the SIM or MRM mode, due to insufficient dwell time and the large number of data points from the wide variety of pesticides. FASST is useful in such cases, where high-risk components are quantified by the SIM or MRM mode and other components are comprehensively qualitatively analyzed in the scan mode. With the Smart SIM+ function and the industry's fastest scan speeds (30,000 u/sec), GCMS-QP2050 systems do not compromise quantitative accuracy even in the FASST mode. This Application News describes a new approach for simultaneously analyzing many components using the GCMS-QP2050 FASST mode.

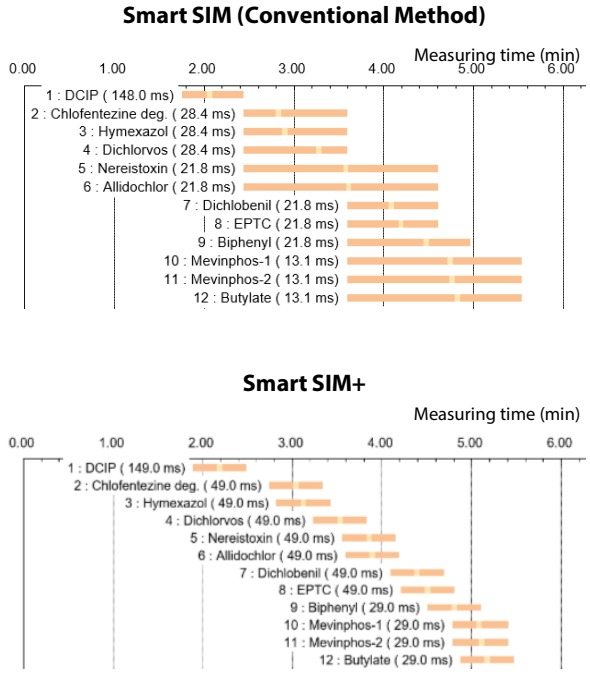


Fig. 2 Comparison of Conventional and New Method for Automatic SIM Method Creation Feature

■ Analysis

This Application News describes using the GCMS-QP2050 FASST mode to simultaneously analyze a large number of residual pesticides and then compares the results with analysis results obtained by the conventional SIM mode using the previous GCMS-QP2020 NX model.

PL2005 GC/MS pesticide mixtures I to VII diluted to 5 ppb were used as standard samples. The actual measurement samples were prepared by extracting ginger by the QuEChERS method, mixing the extract with the standard pesticide mixtures, and spiking the mixtures with dichlorodiphenyltrichloroethane (DDT) (to achieve final concentrations of 5 ppb and 500 ppb, respectively).

Smart Pesticides Database Ver. 2, the residual pesticides database for GC-MS(/MS) analysis was used as the database. The analysis conditions are shown in Table 1.

Table 1 Analytical Conditions

GC-MS:	GCMS-QP2050 (TMP exhaust: 255 L/sec) or GCMS-QP2020 NX
[GC]	
Column:	SH-I-5Sil MS (30 m × 0.25 mm, 0.25 µm)
Insert:	Topaz liner splitless single taper
Inlet Temp.:	250 °C
Injection Volume:	1 µL
Injection:	Splitless (high pressure 250 kPa)
Carrier Gas:	Helium
Control Mode:	Constant linear velocity
Oven Temp.:	90 °C (1 min) – (30 °C/min) - 130 °C - (10 °C/min) - 320 °C (3 min)
[MS]	
IF Temp.:	290 °C
Ion Source:	230 °C
Ionization Mode:	EI
(GCMS-QP2050)	
Mode:	FASST (Scan/SIM)
Scan Range:	*m/z* 35 – 500
Scan Speed:	30,000 u/sec
(GCMS-QP2020 NX)	
Mode:	SIM

■ Comparison of SIM Data between New and Conventional Methods

The SIM data from GCMS-QP2050 FASST measurements and the SIM data from conventional GCMS-QP2020 NX measurements were compared for over 350 types of target pesticides. The average dwell times (average of dwell time for each component) were 9.2 msec and 7.5 msec, respectively. The GCMS-QP2050 improved average dwell time by over 1.7 msec. The corresponding loop time values and the number of data points (not shown) were equivalent.

■ Supplement

In the FASST measurement mode, data acquisition is performed by alternating between scan and SIM modes, as shown in Fig. 3. Generally, the loop time (the time interval for repeating measurements for one ion) in the FASST measurement mode is longer than the SIM mode and there are fewer data points. That can lead to poor reproducibility in the SIM mode.

The GCMS-QP2050 can acquire scan data even with a short data acquisition time because of its high-speed scanning performance. For this Application News article, loop times and the number of data points were kept equivalent to the SIM mode by limiting the scan mode data acquisition time (event time) in FASST measurements to 0.025 sec.

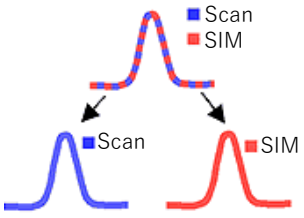


Fig. 3 Summary of Data Acquisitions in FASST Measurement

Next, we compared the reproducibility of peak area values (Table 2). For both GCMS-QP2050 and GCMS-QP2020 NX systems, the %RSD was within 10 % for 337 components, which is 94 % of the 358 pesticide components. The number of components with %RSD values of 10-20 % was 20 with the GCMS-QP2050 and 18 with the GCMS-QP2020 NX, yielding equivalent results. The number of components with a %RSD value over 20 % was 1 with the GCMS-QP2050 and 3 with the GCMS-QP2020 NX. These results confirmed that the same reproducibility of peak area values can be obtained as in the case of SIM measurements only with the GCMS-QP2020 NX, even for FASST measurements with the GCMS-QP2050. Table 2 also shows the reproducibility of peak area values of the pesticides (sample introduction amount: 5 pg) that could be detected in ginger. The GCMS-QP2050 maintained high reproducibility without being affected by complex matrices.

Table 2 Peak Area Reproducibility of Pesticides in Standard Mixtures (samples spiked with 5 pg)

	GCMS-QP2050 (w/o Matrix)		GCMS-QP2020 NX (w/o Matrix)		GCMS-QP2050 (w/ Ginger Matrix)	
RSD	Number of components	Ratio (%)	Number of components	Ratio (%)	Number of components*1	Ratio (%)
≤ 10 %	337	94	337	94	145	99
≤ 20 %	20	6	18	5	1	0.7
20 % <	1	0.3	3	0.8	0	0

*1 Easily degradable and unstable components in samples, such as dioxathion degradation products, were excluded.

■ Peak Identification Utilizing High-Speed Scan Data

Users can display the mass spectrum acquired by scan measurement (Fig. 4(1)) and the chromatograms acquired by SIM measurement (Fig. 4(3)), while referring to the standard spectrum registered in the Smart Pesticides Database (Fig. 4(2)) in the data processing window of LabSolutions GCMS, as shown in Fig. 5. Misidentification risk can be reduced by identifying peaks while referring not only to the peak elution time and ion ratio of SIM data but also to the spectral pattern of scan measurements. Fig. 5 shows the results from FASST measurement of 100 ppb methyl demeton and the standard spectrum. Two peaks are detected around the expected retention time in the SIM data shown in Fig. 5(1). It is difficult to identify methyl demeton from only SIM data because both peaks have similar ion ratios. Therefore, the scan data from each peak was compared to the standard spectrum (Fig. 5(2)). As a result, peak A was more similar to the standard spectrum than peak B and it was determined that peak A was from methyl demeton (Fig. 5(3), (4)).

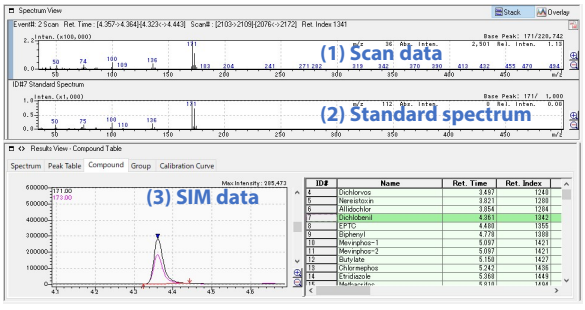
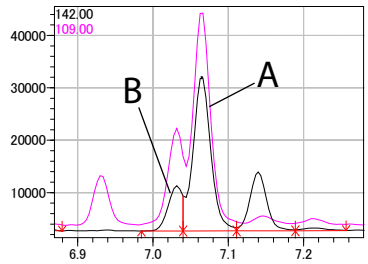
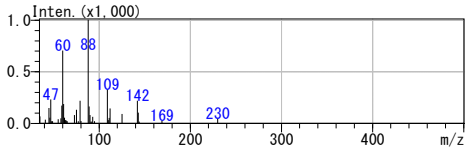


Fig. 4 Data Processing Window of LabSolutions GCMS

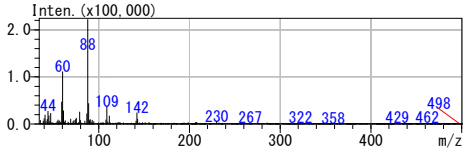
(1) SIM chromatogram



(2) Standard spectrum of methyl demeton



(3) Scan spectrum of peak A



(4) Scan spectrum of peak B

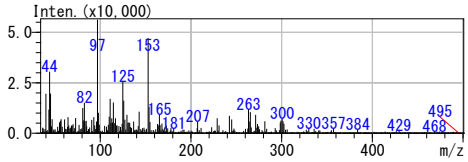


Fig. 5 Mass Chromatograms and Standard Spectra of Methyl Demeton

■ Residual Pesticide Inspection Using High-Speed Scan Data

Using the Smart Pesticides Database, the optimal measurement conditions and analysis parameters are automatically configured in the analysis method according to the selected measurement mode. In this Application News article, the database was used to confirm whether DDT added to ginger samples could be detected with scan mode FASST measurements. As shown in Fig. 6, the measurement mode for DDT was set to the scan mode in advance before FASST measurements. As a result, peaks of the ions being quantified and qualified could be detected at the expected retention time of DDT configured in advance (Fig. 7). Also, as shown in Fig. 9, more accurate qualitative analysis was enabled by searching for the mass spectrum of this peak (Fig. 8 (1)) in the library. In this way, components with low detection risk or low priority can be easily added to inspection targets just by specifying the scan mode in the Smart Database™.

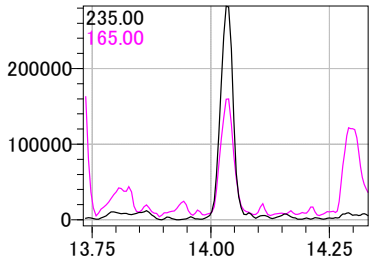
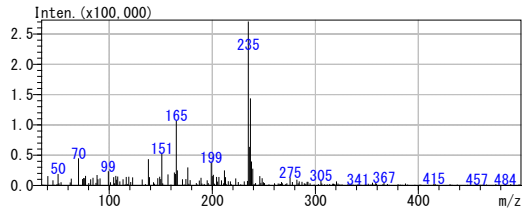


Fig. 7 Scan Mode Mass Chromatogram of DDT

(1) Mass spectrum obtained by FASST mode



(2) Mass spectrum registered in NIST

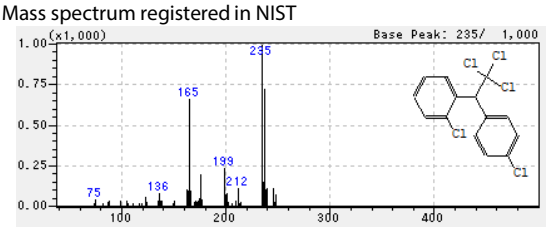


Fig. 8 Mass Spectra of DDT

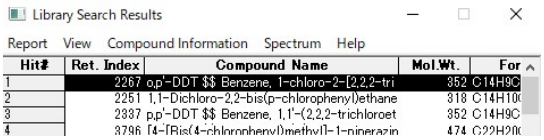


Fig. 9 Result of Library Search of NIST

■ Conclusion

Simultaneous analysis of residual pesticides using the GCMS-QP2050 system confirmed sufficient area reproducibility and quantitation accuracy. In addition, components that were difficult to identify with SIM data alone could be correctly identified by comparing the mass spectra of scan data with standard spectra. Furthermore, low priority components could be qualitatively analyzed using the scan mode. In this way, the GCMS-QP2050, with its Smart SIM+ function and the industry's fastest scan performance, ensures sufficient dwell time in SIM mode, even for simultaneous multi-component analysis by FASST measurements, and maintains high quantitative performance. In addition, by utilizing scan data, it is possible to reliably identify components in SIM data and target a wider range of components in simultaneous inspections. Consequently, the GCMS-QP2050 is ideal for simultaneous multi-component analysis in a variety of industries.

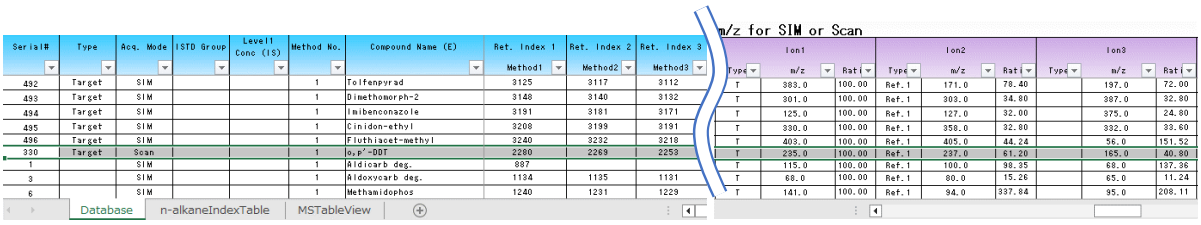


Fig. 6 Compounds Table Created with Smart Database™

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