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Application Data Sheet

No. 115

GC-MS
Gas Chromatograph Mass Spectrometer

Automated Preparation of Calibration Curve Samples in the Analysis of Residual Pesticides in Foods

The AOC-6000 multifunctional autosampler is equipped with a function to automatically exchange syringes with different capacities, as well as a function to stir the samples. This Application Data Sheet reports on the automated preparation of a series of standard pesticide samples to which an internal standard and pseudo matrix were added using these functions, and the results of their analysis with the triple quad GCMS-TQ8040.

Analysis Conditions

An undiluted pesticide standard solution (10 µg/mL, acetone solution) was diluted with an acetone-hexane mixture (1:1 by volume) to prepare a 1 µg/mL pesticide standard solution. An undiluted pesticide surrogate standard mixture (20 µg/mL, acetone solution) was diluted with an acetone-hexane mixture (1:1 by volume) to prepare a 10 µg/mL internal standard solution. Polyethylene Glycol 300 was diluted with an acetone-hexane mixture (1:1 by volume) to prepare a 100 mg/mL pseudo matrix solution. For automated preparation, the system shown in Fig. 1 was used, and the measurements were performed with the conditions shown in Table 1. With this system, preparation was performed automatically (Fig. 3) after the standard solutions, the pseudo matrix sample, empty vials, and dilution solvent were placed, as shown in Fig. 2.

Table 1: Analysis Conditions

[System Configuration]
Autosampler:	AOC-6000 (High End Model)
GC-MS/MS:	GCMS-TQ8040
Column:	SH-Rxi-5Sil MS (30 m long, 0.25 mm I.D., df = 0.25 µm) (Shimadzu, P/N: 221-75954-30)
Glass Insert:	Sky Liner, Splitless Single Taper Gooseneck w/Wool (Restek, P/N: 567366)

[Automated Preparation Conditions for the Calibration Curve Samples]
Dilution Factors:	1000x, 200x, 100x, 20x, 10x
Concentrations After Dilution:	1, 5, 10, 50, 100 ng/mL
Final Volume of Diluted Sample:	500 µL
Amount of Internal Standard Solution Added:	10 µL
Amount of Pseudo Matrix Added:	1 µL

[GC-MS/MS]
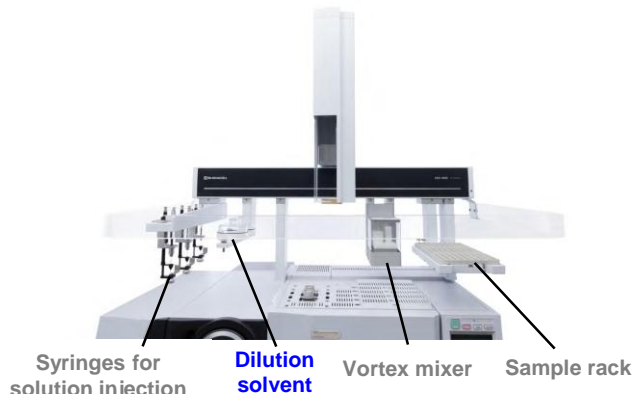The GC-MS/MS analysis conditions registered in the Quick-DB GC/MS Residual Pesticides Database were used.

Syringes for solution injection   **Dilution solvent**   **Vortex mixer**   **Sample rack**
Fig. 1: GC-MS/MS System with the AOC-6000

Empty vials（**corresponding to the number of concentration points in the calibration curve**）

From the left: **Pesticide standard solution; Internal standard solution; Pseudo matrix sample**
Fig. 2: Vials Mounted in the Sample Rack

Solvent   **Pesticide standard solution**   **Internal standard solution**   **Pseudo matrix sample**

(1) Dilution   **(2) Adding the internal standard solution and the pseudo matrix**   **(3) Stirring**   **(4) GC-MS/MS measurement**
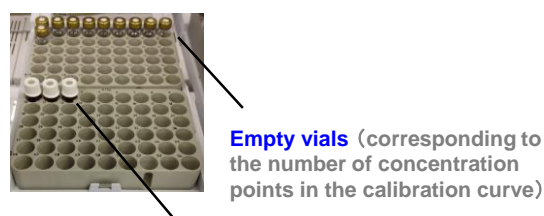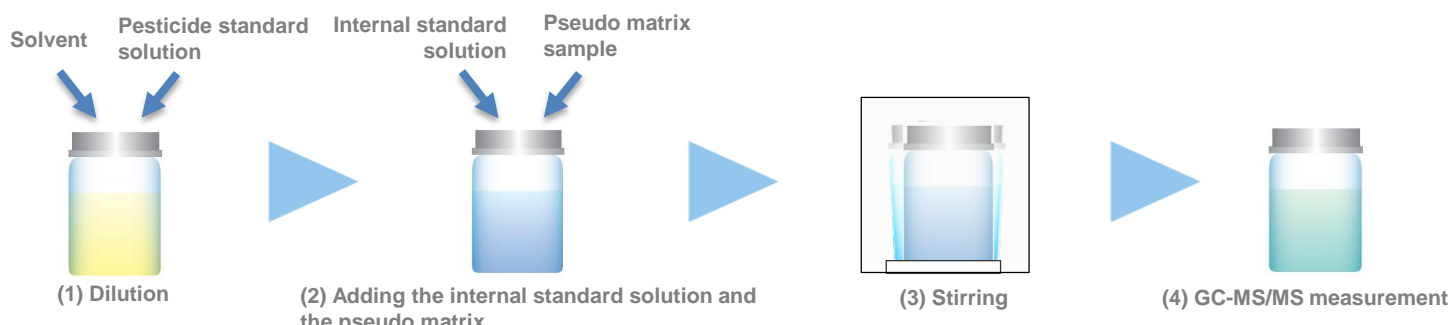
Fig. 3: Standard Sample Preparation Sequence

Analysis Results

Five automatically prepared 100 ng/mL pesticide standard samples were measured with GC-MS/MS. The repeatability data for seven typical pesticides are shown in Table 2. The %RSD was 4.2% (ethofenprox) or less, which is a favorable result.

Table 2: Repeatability of the Seven-Pesticide Standard Sample (100 ng/mL, Area)

Pesticide	Data 1	Data 2	Data 3	Data 4	Data 5	Average	%RSD
Diazinon	259821	254480	252259	269370	259173	259021	2.6
Iprobenfos	834017	838790	839281	914304	864153	858109	3.9
Chlorpyrifos-methyl	272681	268938	275955	288632	287979	278837	3.2
Metolachlor	2591179	2508642	2558336	2648199	2629109	2587093	2.2
Thiabendazole	1248274	1266538	1311997	1363488	1342165	1306492	3.7
Isoprothiolane	165477	165806	162462	174687	164912	166669	2.8
Ethofenprox	1773129	1626398	1608742	1690189	1610465	1661785	4.2

Calibration curves were created using the results from the measurements of automatically prepared pesticide standard samples (1 ng/mL, 5 ng/mL, 10 ng/mL, 50 ng/mL, and 100 ng/mL). Fig. 4 shows those calibration curves. The correlation coefficient (R) was at least 0.999379 (Chlorpyrifos-methyl), indicating that favorable linearity was obtained.
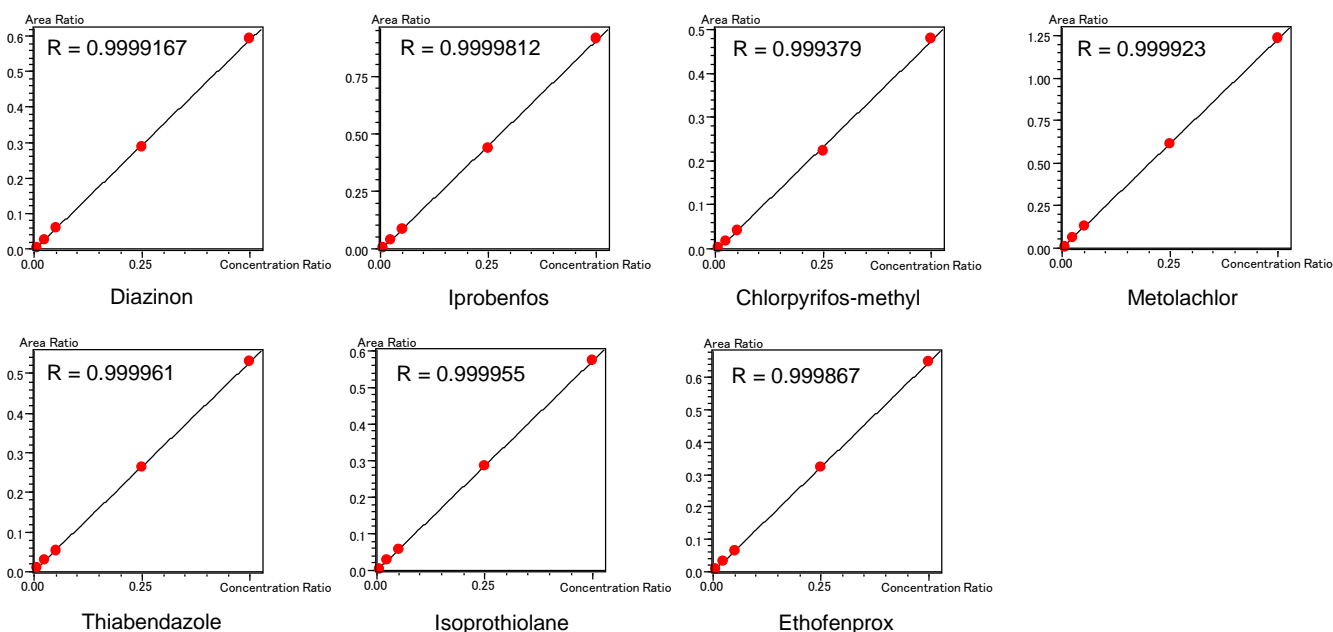
Fig. 4: Calibration Curves and Correlation Coefficients (R) for Seven Pesticides

Conclusion

Favorable repeatability was obtained from the results of GCMS-TQ8040 measurements of pesticide standard samples prepared automatically using the AOC-6000. In addition, the calibration curves obtained from the analysis of a series of automatically prepared standard samples showed favorable linearity.
These results show that automated preparation of calibration curve standard samples using the AOC-6000 can shorten processing times and eliminate human error, which can help analysis laboratories to improve their productivity and reliability.

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