SHIMADZU APPLICATION NEWS

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● GAS CHROMATOGRAPHY MASS SPECTROMETRY

No.M236

Simultaneous Analysis of Residual Pesticides in Food Using Compound Composer Software

■ Introduction

GC/MS enables highly reliable identification of individual substances in complex mixtures. Because of the specificity of the MS, the GC/MS offers superior separation power in comparison with GC equipped with less specific detectors. As a result, GC/MS is utilized for simultaneous analysis of many components, primarily for environmental and food applications. However, conventional measurement requires the confirmation of retention times using standard samples as well as the creation of calibration curves. Problems with the conventional approach are the long times needed for the preparation of standard samples, and the inability to conduct measurements if standard samples are unavailable. Nonetheless, rapid and convenient measurement of many components in a single analysis is important in many situations in order to confirm the degree of contamination by various environmental substances, and to investigate the causes of chemical-related incidents.

To respond to these needs, Shimadzu, in cooperation with Kitakyushu City Institute of Environmental Sciences, has developed a database for simultaneous analysis of numerous analytes, which is especially geared to environmental applications. As shown in Table 1, a number of parameters needed for quantitative analysis are registered in this database, covering a total of 583 toxic substances, including agricultural chemicals. Parameters include compound names, mass spectra, target masses, calibration curves (multi-point internal standards method), and retention times which can be conveniently updated by comparison with those of n-alkanes.

The compounds required for analysis can be selected from the database to create a method file (for GCMS-QP2010). Preparation of standard solutions is not required, and semi-quantitative results can be obtained even for non-ideal target compounds.

Table 1 Compounds Registered in the Database

Category Ⅰ	Number	Category Ⅱ	Number
CH compounds	160	PAHs	49
		PCBs	62
		Other	49
O compounds	81	Phenols	48
		Other	33
N compounds	75	Aromatic amines	36
		Nitro compounds	26
		Other	13
S compounds	8		
P compounds	6		
Agricultural chemicals	253	Insecticides	111
		Herbicides	68
		Fungicides	58
		Other	16
Total number	583		
Internal standards	8		

■ Procedures and Analytical Conditions

The analytical procedures are outlined in Fig.1. Chromatographic column and analysis conditions are fixed, as specified in Table 2, in order to optimize the retention time prediction accuracy and to ensure correct identification. The first step in the setup of the analytical method is analysis of a check sample, consisting of a substance for evaluation of the equipment (DFTPP) and a series of n-alkanes. The retention times for the compounds registered in the database are automatically corrected from the retention time results for the n-alkanes standard. Next, a method file from the database is created, based on the corrected retention times. Data such as the corrected retention time for quantitation, ions for identification and quantitation, the reference mass spectrum, and the calibration curve for each compound are registered in the method file. The next step is the verification of proper equipment performance from the analysis results of the substance for equipment evaluation (DFTPP). Afterwards, the unknown sample is measured with the internal standard substances added. The results obtained for the unknown sample are then analyzed using the method file created from the database.

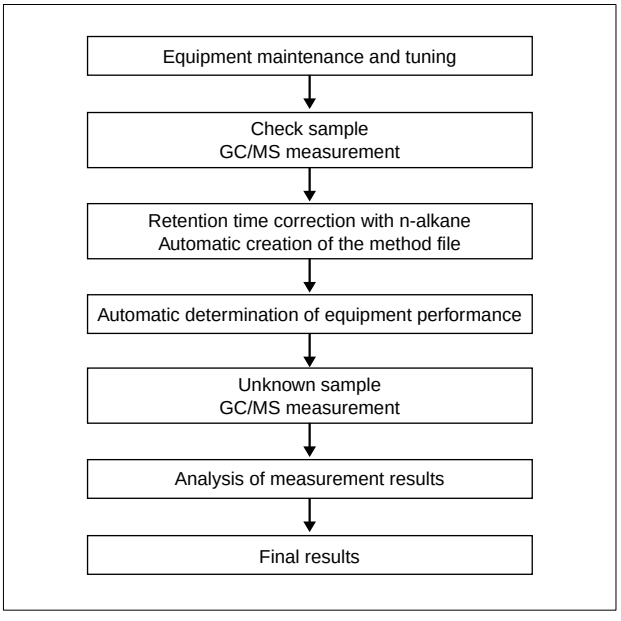

Fig.1 Analysis procedure

■ Results

This method was applied to the analysis of residual pesticides in foods, and the effectiveness of the method for qualitative identification and quantitative determination was evaluated. For evaluation as a sample, agricultural chemicals (Table 3) and internal standard were added to the liquid extract from preprocessing of carrots via solid phase extraction. Predicted values for the retention times utilizing the n-alkanes were accurate to within 0.037 min., as shown in Table 3. In addition, in the comparison with the reference mass spectra, spectral similarity levels were in the range of 85 to 95. As is evident from these results, it was possible to automatically identify all agricultural chemicals in this investigation. Average quantitative results were near the spike level of 0.1 mg/L. There was a single substance showing a value 1.5 times that of the added quantity; this result was attributed to matrix effects.

Table 2 Analytical Conditions

Model	: GCMS-QP2010
Software	: Compound Composer and it's Database
-GC-	
Column	: DB-5 ms,30 m × 0.25 mm I.D. df=0.25 μm
Column Temp.	: 40 ˚C(2 min)→8 ˚C/min→310 ˚C(5 min)
Carrier Gas	: He(Constant linear velocity mode)
Linear Velocity	: 40 cm/s
Injector Temp.	: 250 ˚C
Injection Method	: Splitless (1 min)
Injection Volume	: 1 μL
-GCMS-	
Interface temp.	: 300 ˚C
Ion box Temp.	: 200 ˚C
Ionization Mode	: EI
Scan Mode	: 33 amu-600 amu
Scan Interval	: 0.3 sec

Table 3 Determination Results

Compounds name	m/z	RT (estimated)	RT (measured)	RT deviation	Similarity Index	Additive concentration (mg/L)	Detected concentration (mg/L)
6;1;;a-HCH	219	20.948	20.985	-0.037	85	0.1	0.08
6;1;;Chlorpyrifos	314	24.583	24.588	-0.005	91	0.1	0.08
6;1;;DDVP	185	13.665	13.674	-0.009	92	0.1	0.08
6;1;;EPN	157	30.071	30.086	-0.015	85	0.1	0.15
6;1;;Fenitrothion (MEP)	277	24.181	24.194	-0.013	87	0.1	0.09
6;1;;Fenobucarb	150	19.607	19.615	-0.008	92	0.1	0.08
6;1;;Isophenphos	213	25.623	25.643	-0.02	95	0.1	0.11
6;1;;Isoprocarb	121	18.563	18.568	-0.005	92	0.1	0.09
6;1;;Malathion	173	24.426	24.436	-0.01	84	0.1	0.08
6;1;;Methyl parathion	263	23.543	23.558	-0.015	88	0.1	0.13
6;3;;Flutolanil	173	26.739	26.751	-0.012	92	0.1	0.13
6;3;;Mepronil	119	28.358	28.377	-0.019	97	0.1	0.11

■ Conclusions

Favorable results were obtained for the agricultural chemicals targeted by this investigation, illustrating the effectiveness of the database.

SHIMADZU

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Related Products Some products may be updated to newer models.

› Compound Composer Database Software

GCMS-QP2010 Series Workstation Optional Software

Related Solutions

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