SHIMADZU APPLICATION NEWS

LA146-E014

● GASCHROMATOGRAPHY MASS SPECTROMETRY

No.M190

Pesticide Analysis using Fast-GC/MS

It is vital for analytical laboratories to increase productivity of analysis; that is analyzing many samples at low costs. In particular, productivity of chromatographic analysis can be improved by reducing the analysis time. In this regard, the Fast-GC/MS method is attracting attention.

Application Note M189 “Fast-GC/MS Analysis” studied the effect of MS scan speed on the fast-GC/MS method. This Application Note introduces examples of pesticides analysis using the fast-GC/MS method with the SCAN mode to study sensitivity and quantitative performance.

■ Instrumentation, Analytical Conditions and Compounds

[Fast-GC/MS System]
GC/MS : GCMS-QP5050A / GCMS-QP2010
(GC: Highmpower oven model, AFC-17H)
Software : Class-5000 V2.2 (software for fast-GC/MS)
Column : DB-1
10 m × 0.1 mm I.D. df=0.1μm

[Analytical Conditions]
Inj. Temp. : 280˚C
Carrier Gas. : 680kPa
Sampling Time (Splitless) : 1.5min
Column Temp. : 60˚C(1.5min)
-90˚C/min-170˚C-20˚C
/min-250˚C(3min)
Interface Temp. : 280˚C

Target Pesticides

Peak No	Name	Peak No	Name	Peak No	Name	Peak No	Name
1	DDVP	9-1	TPN	16	Chloropyrifos	23	Isoxathion
2	Etridiazole	9-2	Propyzamide	17	Captan	24	Mepronil
3	Chloroneb	10	Diazinon	18	Pendimethalin	25	CNP
4	MCPP methyl ester	11	IBP	19	Isofenphos	26	Pyridaphenthion
5	BPMC	12	Tolclofos-methyl	20-1	Isoprothiolane	27	Iprodion
6	Pencycuron	13	Terbucarb	20-2	Napropamide	28	EPN
7	Bethrodine	14	MEP	21	Butamifos		
8	CAT	15	Benthiocarb	22	Flutolanil		

■ Total Ion Chromatogram (TIC) with Scanning

Fast-GC/MS generally employs split injection method in order to narrow the sample bandwidths. However, in this example, splitless injection was employed in order to improve sensitivity by introducing larger amounts of sample into the column.

Fig. 1 shows the total ion chromatogram (TIC). Compared to the analysis using split injection, peak separation shows slight deterioration. However, it did not cause problems in quantitation because specific ions are used for quantitation with MS

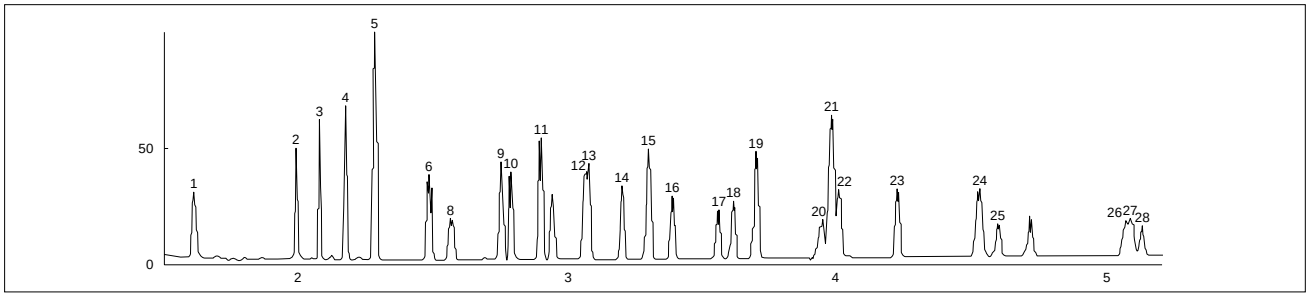

Fig.1 Total Ion Chromatogram

■ Sensitivity of Scan mode

The detection limit is defined by the amount when S/N ratio becomes 3. The results are listed in Table 1.

Table 1 Sensitivity of Pesticides

Peak No	Name	S/N=3(pg)	Peak No	Name	S/N=3(pg)	Peak No	Name	S/N=3(pg)
1	DDVP	4.4	10	Diazinon	13.8	20-1	Isoprothiolane	9.2
2	Etridiazole	6.3	11	IBP	6.0	20-2	Napropamide	9.8
3	Chloroneb	3.5	12	Tolclofos-methyl	1.5	21	Butamifos	36.0
4	MCPP methyl ester	3.3	13	Terbucarb	1.2	22	Flutolanil	1.4
5	BPMC	2.9	14	MEP	21.9	23	Isoxathion	47.9
6	Pencycuron	2.1	15	Benthiocarb	1.9	24	Mepronil	4.9
7	Bethrodine	3.8	16	Chlorpyrifos	6.1	25	CNP	39.3
8	CAT	4.7	17	Captan	39.7	26	Pyridaphenthion	33.6
9-1	TPN	5.1	18	Pendimethalin	32.6	27	Iprodion	20.6
9-2	Propyzamide	12.0	19	Isofenphos	9.8	28	EPN	41.3

Calculated quantities to achieve S/N=3

■ Calibration Curve

Fig. 2 shows the calibration curves for Etridiazole, Simazine (CAT), Benthiocarb and Iprodione The concentrations of standards are 10, 50, 100, 200 and 500μg/L. Good linearity was obtained for these substances, as well as the other substances not shown here.

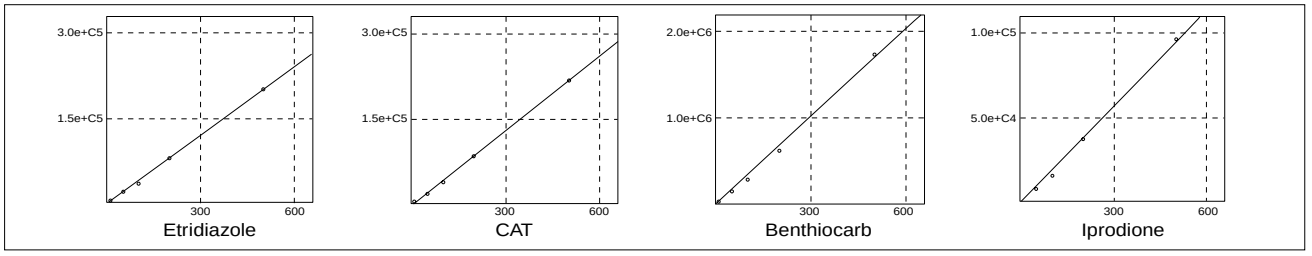

Fig.2 Calibration Curve (10~500μg/ℓ)

■ Repeatability

Table 2 shows the results of repeatability obtained by analyzing 200μg/L standard samples five times. The CV values are 8% or less for all pesticides.

Table 2 Repeatability (200pg, n=5)

Peak No	Name	CV (%)	Peak No	Name	CV (%)	Peak No	Name	CV (%)
1	DDVP	1.04	10	Diazinon	3.90	20-1	Isoprothiolane	2.83
2	Etridiazole	5.22	11	IBP	4.64	20-2	Napropamide	4.31
3	Chloroneb	3.85	12	Tolclofos-methyl	1.91	21	Butamifos	5.24
4	MCPP methyl ester	7.44	13	Terbucarb	3.77	22	Flutolanil	0.99
5	BPMC	3.28	14	MEP	5.50	23	Isoxathion	5.54
6	Pencycuron	5.28	15	Benthiocarb	0.50	24	Mepronil	3.03
7	Bethrodine	5.47	16	Chlorpyrifos	5.46	25	CNP	4.68
8	CAT	3.47	17	Captan	4.63	26	Pyridaphenthion	4.60
9-1	TPN	7.20	18	Pendimethalin	7.42	27	Iprodion	5.29
9-2	Propyzamide	2.12	19	Isofenphos	4.70	28	EPN	5.16

■ Summary

Pesticides were analyzed using the fast-GC/MS methode. Splitless injection was employed to improve sensitivity. As a result, there was a slight deterioration in peak separation compared to split injection. However, it did not cause problems in quantitation because specific ions were used for quantitation with MS. Calibration curves showing good linearity were obtained. The excellent repeatability with less than 8% CV (n=5) was obtained.

Though the scan mode was used in this example, even higher sensitivity is anticipated with using SIM(Selected Ion Monitoring) mode.

SHIMADZU

SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

Printed in Japan 3100-02419-10A-IK