Rapid Analysis of Multiresidual Pesticides in Agricultural Products by Gas Chromatography-mass Spectrometry

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Abstract: A method for the rapid analysis of multiresidual pesticides in agricultural products using gas chromatography-mass spectrometry (GC/MS) was introduced. The matrix solid-phase dispersion (MSPD) technique with slight modification was employed to minimize the matrix interferences. To calculate the recovery, 97 target pesticides were spiked into a range of foods including potato, cabbage, carrot, apple, orange, cucumber, and rice. The combination of the MSPD technique and the GC/MS for the analysis of 97 pesticides can be accomplished within 90 min for each sample. Most pesticides were recovered in the range of 70%–120% with relative standard deviation (RSD) usually less than 10%. The results demonstrated that the method could be successfully applied to a broad range of target pesticides in a diverse range of foods.

Key Words: Gas chromatography-mass spectrometry; Agricultural products; Residual pesticides

1 Introduction

With the increasing focus on food safety in the recent years, there is a dire need to develop analytical methods that can rapidly and exactly determine the residual pesticides in agricultural products. However, the complicated matrix of the agricultural products may affect the accuracy of the analysis. Hence, the pretreatment before the analysis is very important^[1,2]. Although many pretreatment methods have been reported^[3–7], they are time-consuming.

To resolve the problems mentioned above and to obtain the veracity and accuracy, GC/MS was used in many groups to determine the residual pesticides in agricultural products^[8–11]. Ninety-seven pesticides were studied in this work including organophosphorus, organochlorine, organonitrogen, carbamate, and thiocarbamate substances, which were spiked into seven kinds of agricultural products at the 100 ng ml⁻¹ level. The samples were cleaned using the slightly modified MSPD technique and were analyzed using GC or GC/MS^[12,13]. The

combination of the MSPD technique and the GC/MS realized the rapid determination of the 97 pesticides with acceptable recoveries.

2 Experimental

2.1 Instrument and reagents

Acetone and acetonitrile (chromatographic grade) were obtained from Tedia Company, Inc., USA. Water was freshly deionized using a Milli-Q water purification system with a 0.22-μm fiber filter.

The analytical-grade sodium chloride was obtained from the Beijing Chemical Factory, China. Anhydrous magnesium sulfate was obtained from Kanto Chemical Co. Inc., Tokyo, Japan. Primary secondary amine (PSA) sorbent (Bond Elut PSA) was obtained from Varian (Harbor City, CA, USA).

Two groups of stock standard solutions of pesticides were obtained from Kanto Chemical Co. Inc., Tokyo, Japan. One

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group contained 50 pesticides and the other contained 47 pesticides, both being dissolved in acetone. The concentrations of the two groups of stock standard solutions were $10\ \mu\text{g ml}^{-1}$, and the solutions were stored at $-25\ ^\circ\text{C}$. Before each experiment, the solution containing the 97 pesticides for analysis was prepared by mixing the two groups of stock standard solutions and was diluted into the desired concentration using acetone.

The GC/MS-QP2010 used in this study was obtained from Shimadzu Company (Kyoto, Japan). An RTX-5ms column ((5% phenyl) methylpolysiloxane; $0.25\ \text{mm i.d.} \times 30\ \text{m}$ with a film thickness of $0.25\ \mu\text{m}$; Restek Corporation, Bellefonte, PA, USA) was used for the separation of the pesticides. The temperature of the capillary column was set at $80\ ^\circ\text{C}$ for the initial 2 min, which was increased to $180\ ^\circ\text{C}$ at $20\ ^\circ\text{C min}^{-1}$ and then to $280\ ^\circ\text{C}$ at $5\ ^\circ\text{C min}^{-1}$ for 20 min. Helium was used as the carrier gas. The quadrupole mass spectrometer was operated in the electron impact ion (EI) mode with a source temperature of $230\ ^\circ\text{C}$ and an electron energy of 70 eV.

2.2 Sample preparation

All samples were purchased from local markets in Beijing, China. The samples were extensively crushed to achieve good sample homogeneity. After crushing, in the case of rice, an extra step of filtering through a sifter ($0.45\ \text{mm}$ aperture) was required. Once homogenized, the samples were stored at $-25\ ^\circ\text{C}$ until GC/MS analysis was carried out.

To prepare the samples, 10 g of a previously homogenized food material was transferred into a suitable glass vessel (for rice, an extra 10 ml of deionized water was added). Ten milliliters acetonitrile was then added to each sample using an adjustable-volume solvent dispenser. The glass vessels were capped before mixing on Vortex mixer for 1 min at optimum speed. Once the initial sample mixing was completed, 1 g NaCl and 4 g anhydrous MgSO_4 were added and immediately mixed on a Vortex mixer for 1 min.

To separate the phases, the samples were centrifuged for 10 min at 3000 rpm. Using an adjustable repeating pipette, 1.0 ml aliquot of upper acetonitrile layer was transferred into a 1.5-ml flip-top microcentrifuge vial containing 150 mg anhydrous MgSO_4 and 50 mg PSA sorbent. The vial was tightly capped and shaken on a Vortex mixer for 1 min before the extracts (or the batch of extracts) were centrifuged (Wealtec, e-centrifuge) for 5 min to separate the solids from the solution. The solution was then transferred into an autosampler vial for GC/MS analysis. For spiked samples, standard pesticides were spiked into the samples before adding acetonitrile for extraction; the other steps were the same as described above.

3 Results and discussion

3.1 The chromatogram of mixed standard pesticides

The chromatogram of the 97 standard pesticides ($500\ \text{ng/ml}$) is shown in Fig. 1. It is seen that the total analysis time for the 97 pesticides in GC/MS was 50 min, whereas 40 min was sufficient for the pretreatment of each sample. Table 1 shows the retention time and the quantification ions of 10 representative pesticides including organophosphorus, organochlorine, organonitrogen, carbamate, and thiocarbamate substances.

Fig. 1 Chromatogram of 97 standard pesticides ($500\ \text{ng ml}^{-1}$)

3.2 Calibration curve and detection limit

Three standard solutions of all the standard pesticides (50, 100, and $500\ \text{ng ml}^{-1}$) were determined for the calibration curves. Each calibration point was obtained based on three duplicate injections of the standard sample. Because the absorption phenomena may occur at a low concentration of the pesticide compounds, a second order of the calibration curve was employed. The regression equations and the correlation coefficients of the representative pesticides are also shown in Table 1.

It is seen that in this study, the second-order regression equations fitted the calibration data better than the conventional linear equations. The estimated limits of detection (LOD) and quantitation (LOQ) calculated as the concentration that produced a signal equal to 3 times and 10 times the background noise level, for the three analyzed matrices: potato, apple, and rice were obtained, which are listed in Table 2.

3.3 Recovery test

Recovery of the 97 pesticides ($100\ \text{ng ml}^{-1}$) spiked into potato, cabbage, carrot, apple, orange, cucumber, and rice was investigated using GC/MS. The chromatogram of the pesticides spiked into rice, apple, and cabbage is shown in Fig. 2. To calculate the recovery, the spiked sample from each agricultural product was prepared thrice, and the unspiked samples were also investigated. Some of the average recovery results and the relative standard deviations (RSD) are shown in Table 3. Most pesticides were recovered within an acceptable recovery range of 70%–120%. The reasons for some higher or lower recoveries may be due to some

interferences that remained in the matrix, or some unstable compounds that decomposed in the extraction step or in the analysis process. In addition, more pesticides in vegetables and fruits showed acceptable recoveries than those in rice because of the more complicated matrix of rice. On the whole,

the MSPD technique reduced the interferences of the matrix to a large degree and could be applied to a more complicated matrix such as rice. Furthermore, the low RSD values (usually <10%) indicated the high reproducibility of the system.

Table 1 Regression data, retention times, and quantification ions of the representative pesticides

Pesticides	Retention time (min)	Quantification ion (<i>m/z</i>)	$y = ax^2 + bx + c$			<i>r</i>
			a	b	c	
Eptam	6.600	128	−0.3215222	5173.438	−37879.61	0.9998
Butylate	7.118	156	0.383311	3158.303	−6202.944	0.9998
α -Benzene hexachloride	9.535	219	0.470097	1181.535	−58.5139	0.9998
δ -Benzene hexachloride	10.741	219	0.456664	705.060	1728.819	0.9997
Carbaryl	11.823	144	3.60645	1112.737	−46132.75	0.9988
Diethofencarb	12.682	267	1.004072	858.599	−14990.14	0.9995
Fenthion	12.850	278	2.969206	3138.779	−7438.472	0.9996
Chlorpyrifos	12.896	197	0.944697	886.365	2237.986	0.9996
Tebufenpyrad	20.243	318	1.659283	1403.177	−683.083	0.9995
Pyridaben	23.307	147	13.40633	5326.545	59432.15	0.9990

y and *x* indicate the peak area and the concentration (ng/ml) of the pesticides, respectively. The correlation coefficients are expressed as *r*

Table 2 Estimated limits of detection (LOD) and quantitation (LOQ) calculated as the concentration that produced a signal equal to 3 times and 10 times the background noise level

Pesticides	LOD (ng/g)			LOQ (ng/g)		
	Potato	Apple	Rice	Potato	Apple	Rice
Eptam	15	56	90	50	186	297
Butylate	65	47	52	216	156	173
α -Benzene hexachloride	58	14	23	193	47	77
δ -Benzene hexachloride	52	30	71	173	100	236
Carbaryl	27	54	47	90	180	156
Diethofencarb	26	26	30	86	86	100
Fenthion	8	6	7	27	20	23
Chlorpyrifos	25	32	33	83	107	110
Tebufenpyrad	12	9	22	40	30	73
Pyridaben	12	11	10	40	37	33

Table 3 Results of the recovery test

Pesticides	Potato		Cabbage		Carrot		Cucumber		Apple		Orange		Rice	
	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
1 (Z)-Dimethylvinphos	94	2.3	99	1.7	97	6.7	112	7.6	116	4.0	164	4.4	116	4.4
2 Acephate	194	5.1	205	1.6	220	6.4	75	27.8	116	4.3	108	1.4	108	4.7
3 Acetamiprid	89	2.9	100	3.0	108	3.3	106	6.4	88	1.7	101	5.0	198	1.0
4 Acrinathrin	85	4.6	113	3.1	88	8.8	104	6.5	116	1.7	120	5.3	114	4.2
5 Bendiocarb	71	4.3	94	4.3	75	7.6	97	2.9	107	6.4	165	5.4	111	4.7
6 Benfuresate	75	2.2	77	1.9	77	3.7	71	3.5	78	4.9	114	6.6	87	6.7
7 Bitertanol	95	3.1	91	5.8	114	1.1	189	9.3	115	4.0	130	1.3	199	3.4
8 Fenobucarb	79	1.4	86	1.5	74	5.3	93	3.7	79	1.0	119	1.0	83	8.4
9 Butylate	68	1.9	69	2.4	69	0.6	64	2.5	57	5.8	93	2.8	60	7.2
10 Cadusafos	74	0.9	77	1.2	80	1.2	92	3.3	81	4.0	97	3.1	84	4.3

Pesticides		Potato		Cabbage		Carrot		Cucumber		Apple		Orange		Rice	
		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
11	Captan	69	5.6	ND	/	51	4.0	37	9.7	61	2.5	86	3.4	25	6.0
12	Chinomethionat	49	0.2	49	2.5	48	4.0	43	1.1	44	3.1	53	4.0	41	1.2
13	Chlorobenzilate	86	1.2	84	2.2	92	1.1	129	4.5	116	2.1	148	2.7	143	6.8
14	Chlorpropham	72	1.5	77	1.6	76	2.3	77	9.4	72	3.2	110	0.3	77	5.5
15	Chlorpyrifos	70	1.2	71	3.7	73	4.9	88	5.1	82	1.6	120	1.7	98	3.0
16	Cyfluthrin	67	9.5	79	6.3	75	4.4	105	3.4	110	5.9	95	3.3	116	5.4
17	Cyhalothrin	86	3.4	112	4.4	110	9.2	113	7.5	107	3.0	118	1.7	132	4.1
18	Cypermethrin	55	2.6	85	12.7	79	7.8	91	8.2	79	4.3	91	9.1	98	9.7
19	Cyproconazole	83	3.6	84	3.4	91	3.2	125	7.0	109	3.4	139	0.1	134	3.3
20	Dichlorobenzophenone	78	0.9	82	2.9	80	0.7	88	4.0	81	1.6	114	4.5	95	6.6
21	Dichlorvos	72	1.5	78	1.8	73	4.4	74	6.6	67	4.3	99	1.8	64	9.3
22	Deltamethrin	78	3.9	81	6.8	108	10.6	62	7.2	92	0.4	82	7.1	31	11.1
23	Diazinon	76	1.4	78	0.4	79	3.1	86	4.4	83	1.7	116	2.7	86	9.5
24	Dichlofluanid	63	2.2	58	1.5	61	2.3	47	5.0	58	4.4	81	6.4	44	4.6
25	Diethofencarb	86	3.5	84	2.9	89	5.3	102	5.7	102	2.2	152	4.5	128	9.7
26	Difenoconazole	95	1.8	87	2.1	117	2.6	143	8.4	111	8.6	142	9.8	144	1.9
27	Difolatan	ND	/	ND	/	ND	/	45	2.8	96	4.9	53	3.7	46	8.7
28	Dimethipin	84	1.6	89	8.0	73	5.6	85	4.3	85	2.3	112	4.9	73	3.6
29	Edifenphos	132	2.0	143	11.8	128	10.5	143	6.7	136	2.7	180	4.5	169	5.1
30	O-Ethyl-O-(4-nitrophenyl) Phenyl phosphonothioate	95	4.5	97	10.2	113	2.1	158	2.9	103	7.1	113	0.4	183	5.5
31	Eptam	69	0.4	69	1.6	70	2.7	60	4.9	50	3.4	84	0.8	60	15.2
32	Esprocarb	77	1.4	79	4.1	82	1.7	91	2.6	85	4.4	116	0.9	94	7.4
33	Ethiofencarb	105	5.9	121	5.4	98	8.1	113	9.7	115	3.8	177	1.5	112	5.6
34	Ethoprophos	78	2.1	78	4.0	82	4.8	84	3.2	85	1.6	118	1.0	79	9.4
35	Etrimfos	74	1.8	75	2.5	78	3.4	103	4.5	93	1.4	132	3.4	95	5.8
36	Fenarimol	85	1.1	85	6.0	95	1.0	132	3.0	110	6.9	141	5.5	143	3.3
37	Fensulfothion	89	1.6	97	3.7	122	3.0	136	9.2	110	2.5	110	8.5	140	4.3
38	Fenvalerate	74	2.9	105	4.0	89	4.9	81	0.7	113	2.4	111	5.3	115	4.2
39	Flucythrinate	93	6.3	111	8.5	110	4.4	116	4.3	119	8.1	129	2.8	116	6.9
40	Flusilazole	80	1.1	85	3.6	87	2.5	115	2.9	107	5.0	120	2.4	117	3.9
41	Flutolanil	85	1.4	88	2.2	104	1.6	120	2.8	115	2.6	139	5.9	130	6.4
42	Fluvalinate	82	11.5	133	7.3	75	2.7	112	6.3	127	5.1	113	4.4	129	2.2
43	Fosthiazate	127	3.0	127	2.6	129	5.3	110	2.9	147	6.9	193	1.6	92	5.6
44	Halfenprox	92	1.9	99	5.3	115	5.0	120	2.1	120	0.4	130	2.2	139	5.6
45	Imibenconazole	88	7.0	99	3.7	113	4.0	110	4.0	75	3.2	107	7.7	104	1.9
46	Iprodione	138	0.7	156	5.7	91	2.7	111	5.2	117	0.3	97	6.0	136	2.1
47	Isofenphos Oxon	118	5.9	112	4.5	110	18.1	191	3.4	167	5.7	178	7.0	205	3.2
48	Isophenphos	80	3.8	83	4.1	88	2.4	118	0.6	112	1.5	135	4.7	118	4.3
49	Isoproc carb	80	3.4	87	4.9	69	4.2	88	6.9	81	5.5	114	1.5	83	7.1
50	Lenacil	91	2.0	92	4.5	104	4.6	124	0.8	118	0.4	134	1.7	143	9.2
51	Malathion	83	1.3	91	4.3	90	3.1	113	6.3	106	5.6	139	4.3	112	6.3
52	Mefenacet	93	1.1	95	5.5	107	3.1	125	6.0	115	3.1	131	0.8	144	6.6
53	Fenitrothion	81	1.8	74	2.7	94	4.9	120	3.1	119	5.2	156	8.4	125	5.1
54	Mepronil	85	1.0	85	2.6	93	2.0	120	2.0	116	2.4	129	1.2	134	4.7
55	Methamidophos	71	2.0	84	4.1	68	6.8	109	3.3	110	1.4	111	2.7	103	3.0
56	Methiocarb	90	4.1	126	8.1	80	10.8	134	9.4	113	0.8	185	5.6	109	2.9
57	Methyl-Parathion	85	6.7	82	5.2	92	9.0	108	0.5	115	4.6	151	3.9	120	1.1
58	Metolachlor	80	0.4	80	4.5	86	2.9	118	1.3	100	5.5	139	8.4	117	4.4

Pesticides	Potato		Cabbage		Carrot		Cucumber		Apple		Orange		Rice	
	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
59 Fenthion	76	1.4	81	1.6	83	1.8	95	6.1	89	4.8	112	0.8	88	7.9
60 Myclobutanil	84	1.8	84	1.9	93	2.7	110	7.7	111	1.4	112	6.0	128	8.4
61 Carbaryl	78	4.8	124	12.3	78	11.7	115	2.4	120	3.0	146	6.6	111	5.5
62 <i>p,p'</i> -Dichloro-diphenyldichloroethane	82	1.1	87	2.2	89	1.8	103	6.1	85	2.6	118	1.0	114	6.3
63 <i>p,p'</i> -Dichloro-diphenyldichloroethylene	82	3.0	82	3.3	81	1.3	85	5.0	80	5.1	115	4.1	87	6.0
64 Paclobutrazol	84	2.2	83	4.8	92	1.7	118	4.4	110	6.3	150	4.8	81	3.2
65 Phenthoate	78	2.5	80	3.5	89	4.8	111	7.6	103	7.4	135	5.8	114	5.8
66 Parathion	83	1.1	77	9.5	96	9.4	136	9.4	118	1.9	158	6.1	135	1.0
67 Pendimethalin	85	4.5	79	2.9	96	7.1	129	5.1	114	0.6	154	4.2	138	8.7
68 Permethrin	92	1.6	95	2.9	98	3.5	108	6.3	112	2.2	109	0.0	119	0.7
69 Phosalone	87	0.9	95	5.9	103	8.9	131	9.4	115	4.2	97	1.2	151	4.6
70 Pirimicarb	76	1.4	78	2.0	80	1.5	82	5.8	79	1.9	114	0.1	89	5.7
71 Pirimiphos-methyl	76	1.2	82	2.9	84	2.2	103	5.7	98	7.1	131	2.9	112	5.1
72 Pretilachlor	88	1.3	83	2.7	101	4.3	134	3.6	120	1.3	164	4.9	146	6.1
73 Propiconazole	85	3.6	86	1.1	92	1.8	143	0.4	113	3.4	115	2.3	136	8.9
74 Prothiofos	80	2.1	83	4.8	85	1.6	104	6.8	97	3.9	130	6.1	114	7.3
75 Pyraclofos	141	4.2	157	5.1	161	14.6	164	8.6	151	7.0	185	1.7	199	8.4
76 Pyridaben	89	0.4	96	2.6	103	3.8	116	1.8	117	1.3	113	1.5	130	4.5
77 Pyrifenox	79	0.4	81	3.5	85	3.6	119	1.4	100	0.7	138	4.1	130	5.5
78 Pyrimidifen	94	1.2	101	3.9	107	0.8	120	1.3	125	2.6	138	4.8	132	3.6
79 Pyriproxyfen	87	1.6	89	3.6	93	1.4	119	1.2	117	1.6	120	5.3	130	5.4
80 Quinalphos	80	2.7	84	2.8	89	3.3	114	5.3	93	6.4	117	3.4	102	7.2
81 Silafluofen	90	0.4	92	0.4	92	1.0	119	2.4	115	1.3	120	0.9	128	7.1
82 Tebuconazole	82	1.0	84	2.0	93	1.1	132	1.8	111	3.3	133	5.5	136	1.2
83 Tebufenpyrad	89	2.6	91	2.2	92	4.8	114	3.3	111	2.5	129	4.8	130	8.6
84 Tefluthrin	76	0.9	79	1.6	80	2.3	86	5.0	78	1.4	114	4.4	79	8.1
85 Terbufos	75	2.9	78	3.7	81	2.9	111	3.9	101	2.9	129	0.3	108	3.9
86 Thenylchlor	88	1.6	85	3.6	98	3.6	117	3.2	95	3.0	119	1.4	129	6.5
87 Thiobencarb	77	1.9	78	1.6	81	1.6	95	4.1	75	2.6	105	2.4	86	8.8
88 Thiometon	74	2.6	75	2.2	79	2.7	93	2.7	84	2.1	120	1.9	88	7.8
89 Tolclofos-methyl	76	1.4	80	1.9	82	0.9	91	7.7	84	2.6	118	2.2	88	5.7
90 Triadimenol	79	4.8	82	5.6	91	2.2	86	6.1	114	2.1	116	3.0	117	8.4
91 Tricyclazole	94	3.3	100	2.2	111	2.3	110	5.9	84	8.7	92	4.6	108	2.9
92 α -Benzene hexachloride	76	2.4	79	1.0	79	1.7	73	2.4	56	1.3	107	0.1	69	8.8
93 α -Chlorfenvinphos	91	3.5	95	1.3	94	3.8	119	0.7	116	2.5	160	6.8	117	2.6
94 β -Benzene hexachloride	80	3.4	83	6.6	83	4.5	90	4.3	99	2.5	113	3.3	89	7.9
95 β -Chlorfenvinphos	91	3.5	95	1.3	94	3.9	119	0.7	119	2.0	153	3.2	130	9.4
96 γ -Benzene hexachloride	76	0.9	77	4.5	76	5.2	84	1.4	63	4.7	114	0.7	72	2.8
97 δ -Benzene hexachloride	66	6.4	82	2.6	75	4.6	68	13.7	53	5.7	76	4.2	56	8.8

Mean: average recovery; RSD: relative standard deviation; ND: not detected

4 Conclusions

The GC/MS system for the analysis of multiresidual pesticides in agricultural products was developed in this study. This article also provides some references for the analysis of residual pesticides in some other products. The MSPD

technique reduced the interferences of the matrix to some degree. The results showed that most pesticides were recovered within an acceptable recovery range of 70%–120% and the RSD values were usually less than 10%. The method can be successfully applied to accurately and rapidly analyze a broad range of target pesticides in a diverse range of foods.

This may become a routine method for determining residual pesticides in products.

Fig. 2 Chromatogram of pesticides spiked into rice (a), apple (b) and cabbage (c)

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