C146-E334

SHIMADZU
Excellence in Science

Technical Report

Residual Pesticides Analysis of Botanical Ingredients Using Gas Chromatography Triple Quadrupole Mass Spectrometry

Riki Kitano[1], Tairo Ogura[1], Nicole Lock[1], Robert Clifford[1], Julie Kowalski[2], Jack Cochran[2], Dan Li[2]

Abstract:

Dietary supplements, which are consumed worldwide, are made from various botanical ingredients. To be safe from pesticides exposure, residual pesticides must be monitored by chromatographic instrumentation. Issues arise however, due to the fact these botanical samples are dried and cause large interferences in the chromatography. This study shows that the modified QuEChERS method combined with GC-MS/MS achieves consistent pesticides monitoring in botanical ingredients.

Keywords: botanical, QuEChERS, pesticides, GC-MS/MS

1. Introduction

The use of dietary supplements is increasing in the United States. These dietary supplements are made from various dried botanicals and residual pesticides and because of that, they have to be monitored to ensure their quality and prevent exposure. The ingredients are dried which requires the standard QuEChERS methods[1)] to be modified to overcome this difficulty. Gas chromatography is best technique to separates multi components including coextracted interferences, and because the triple quadruple mass spectrometry (GC-MS/MS) is highly sensitive this allows analysis of trace level contamination. In this study, we analyzed over 200 compounds simultaneously using a triple quadrupole gas chromatograph mass spectrometer with the modified QuEChERS method.

2. Materials and Methods

Pesticides standards, internal standards, quality control standards and QuEChERS kit were obtained from Restek:

- GC Multiresidue Pesticide Kit (Cat.#: 32562)
- GCMS Internal Standard Mix (Cat.#: 33267)
- SV Internal Standard Mix (Cat.#: 31206)
- Q-sep QuEChERS Extraction Kit (Original) (Cat.#: 23991)

The total number of targets was 232 compounds (220 pesticides, 6 internal standards, and 6 quality control standards).Ginseng, which can be purchased in any store, was used as a matrix. Using this ginseng, matrix-matched calibration standards (1 to 200 ng/mL) and fortified samples (each two 10 and 50 ng/g) were prepared. Calibration curves were generated by internal standard method, weighted 1/C and the internal standard was PCB52.

A MRM analytical method was created using the Smart Pesticides Database (Shimadzu). This database has retention indices for all registered compounds, and retention times can be predicted by running an *n*-alkane sample mixture (AART: Automatic Adjustment of Retention Time). According to estimated retention times, Smart MRM® creates an optimum data acquisition time program (Fig. 1).

2-1. Extraction and Clean-up Procedure

- Weigh 1.0 ± 0.05 g ground ginseng powder into 50 mL polypropylene centrifuge tube.

- Add 10 mL HPLC-grade water and vortex the tube vigorously.
- Add 10 mL of the ACN/IS Extraction Solvent.
- Allow the tube to sit for 15 min.
- Add 4 g anhydrous $MgSO_4$ and 1 g NaCl.

- Shake the tube vigorously on a mechanical shaker for 30 min.
- Centrifuge the 50 mL tubes at 3000–4500 rpm × 5 min.

- Condition the GCB/PSA (0.25 g/0.5 g) SPE columns with ~250 mg anhydrous $Na2SO_4$ on top using 3 column volumes of acetone.
- Insert a collection rack consisting of 15 mL disposable glass centrifuge tube on a SPE vacuum manifold.
- Add 1.25 mL of the ACN extract.
- Rinse with 1 mL acetone.
- Elute with 12 mL of 3:1 v/v acetone:toluene.

- Evaporate (50 °C) the eluent to ~100 µL gently.
- Add 500 µL of toluene to the Blank/fortified samples, calibration standard solutions to matrix matched calibration standards.

- Add 20 µL quality control standards (12.5 µg/mL) and ~50 mg of anhydrous $MgSO_4$ to all samples.
- Vortex for 5 sec.
- Centrifuge the tubes at 3000 g × 5 min.

- Transfer the toluene extract using a Pasteur pipette to ALS GC vials.

1 Shimadzu Scientific Instruments, Inc.
2 Restek Corporation

Fig. 1 Data Acquisition Time Program Created by Smart MRM

2-2. Analytical Condition

System Configuration	
GC-MS/MS	: GCMS-TQ8040 (Shimadzu)
Auto Injector	: AOC-20i + 20s (Shimadzu)
Column	: SH-Rxi-5MS 30 m × 0.25 mm I.D., df = 0.25 µm (Shimadzu, P/N: 221-75940-30) with Rxi guard column 5 m × 0.25 mm I.D. (Restek, Cat.#: 10029)
Glass Liner	: Sky Liner, Splitless Single Taper Gooseneck w/Wool (Restek, Cat.#: 23336.5)
Software	: GCMSsolution Ver. 4.42
GC	
Injection Temp.	: 250 °C
Oven Temp.	: 90 °C (1 min), 30 °C/min to 130 °C, 10 °C/min to 330 °C (2 min)
Total GC Time	: 24.33 min
Carrier Gas Control	: Linear Velocity (55 cm/sec)
Injection Mode	: Splitless with high pressure injection (250 kPa, 1.5 min)
Injection Volume	: 2 µL
MS	
Interface Temp.	: 290 °C
Ion Source Temp.	: 230 °C
Ionization Mode	: EI (Ionization voltage: 70 eV)
Acquisition Mode	: MRM (2 transitions for each compound)
Resolution	: Unit (Q1) – Low (Q3)
Loop Time	: 0.4 sec

3. Result and Discussion

3-1. Matrix Matched Calibration

The chromatogram in Fig. 2 shows a 10 ng/mL matrix matched calibration standard. Of the 232 compounds, 230 could be detected in ±0.1 min of estimated retention time by AART. The remaining two compounds, 1,4-Dichlorobenzene-d4 and Naphthalene-d8 of six quality control standards, had eluted before 4 min. Although retention times were shifted, they were with identified within about ±0.2 min of estimated.

Calibration curves were generated from matrix matched calibration standards, then back calculation and linearity were evaluated.

Back calculation was performed by calculating the concentration of each calibration point, and if the concentration exceeds ±20% of theoretical value, the calibration point would be interpolated with the nearest two points. Over 93% of the compounds with concentration of 1 ng/mL were within ±20% of theoretical calculations and all compounds of concentration 20 to 200 ng/mL were within ±20% (Fig. 3).

This modified QuEChERS method contains dilution steps, and samples will be diluted by quarter. To quantify 10 ng/g concentration, 2 ng/g or lower calibration point are required. Even at low concentrations, the calibration curves show good linearity (Fig. 4) and all coefficients of determination (220 pesticides) were greater than 0.99.

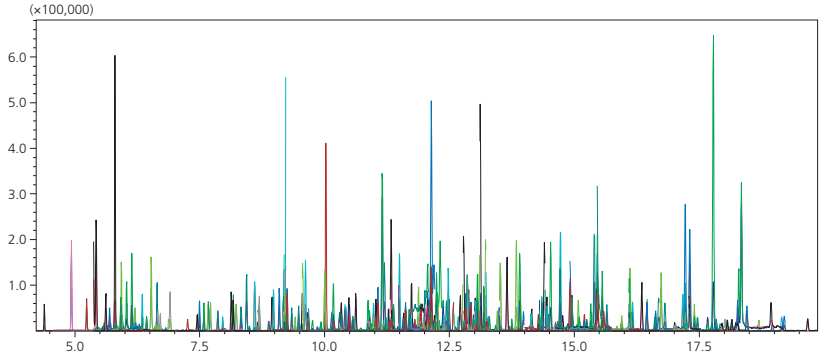


Fig. 2 MRM Chromatogram of 10 ng/mL Matrix Matched Calibration Standard (Internal standards and quality control standards are not displayed.)

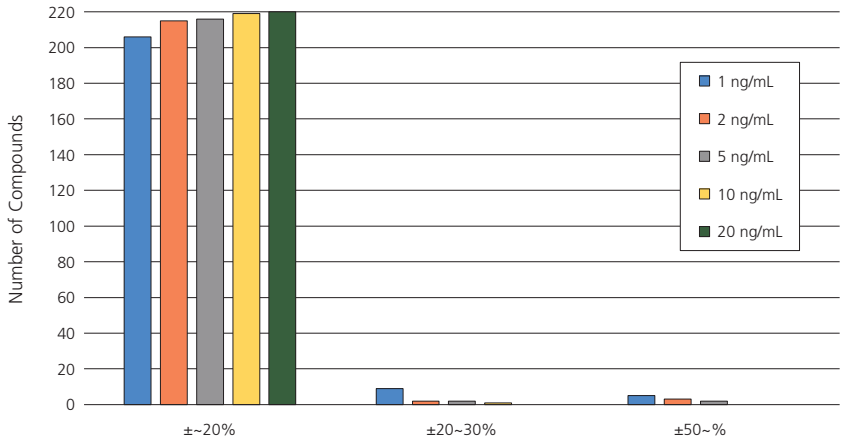


Fig. 3 Difference Between Back Calculation and Theoretical Concentration

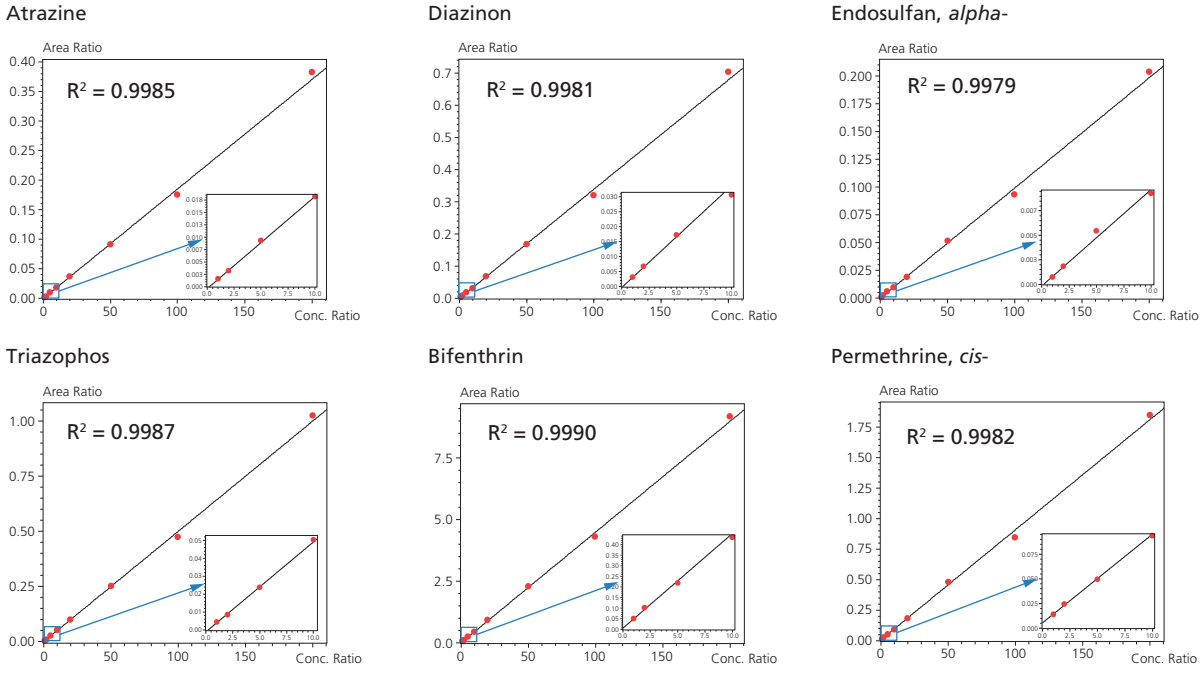


Fig. 4 Calibration Curves of Representative Six Compounds

3-2. Recovery of Fortified Sample

Each two 10 ng/g and 50 ng/g fortified samples were prepared (10 ng/g-1, 10 ng/g-2, 50 ng/g-1, 50 ng/g-2) and these recovery rates were evaluated from the average of three successive data points for each samples.

Of the compounds, 85% showed good recovery within the range of 70 to 120% on 10 ng/g-1 and 50 ng/g-2. As mentioned previously, fortified samples were diluted to 2.5 ng/mL and 12.5 ng/mL. Since calibration curves showed good linearity at low concentration and modified QuEChERS method suppressed interference, good recovery result were achieved. (Some compounds were not quantified correctly because the matrix for the calibration standards originally contained them. Y-intercept were lifted up and this shift might cause incorrect quantification, especially at low concentration. From the standard addition method, 16 pesticides were detected with more than 10 ng/g in matrix.)

In this study, recovery results were rechecked and combined with qualitative information, the relative ion ratio. Ion ratios between the target and reference were compared to that of the 100 ng/g standard and evaluated according to SANCO/12571/2013[2]. This mentions that the use of relative ratio ±30% as a criteria is recommended.

Table 1 shows recovery and relative ion ratio for all compounds and Fig. 5 shows the combination map of recovery and relative ion ratio, which was generated from this table. Of the compounds, 76% in the 10 ng/g-1 were within ±30% on relative ion ratio with good recovery of between 70 to 120%. For 50 ng/g-2, 83% of the compounds were within a ±30% relative ion ratio. And here, compounds which showed poor recovery and/or over ±30% relative ion ratio were examined (Page 7).

Table 1 Recovery and Relative Ion Ratio; Relative ion ratios were calculated by those of 100 ng/mL standard solution.

ID	Compound Name	Transitions				Recovery (Average of n = 3)				Relative Ion Ratio (Average of n = 3)			
		Target	CE	Reference	CE	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2
1	2,3,5,6-Tetrachloroaniline	228.9 > 158.0	18	230.9 > 158.0	22	72.5	67.0	61.4	68.8	93.6	97.2	98.9	101.1
2	2,4'-Methoxychlor	227.1 > 121.1	16	228.1 > 122.1	16	83.0	101.1	86.5	80.2	94.7	105.0	104.6	106.7
3	2-Phenylphenol	170.1 > 141.1	24	170.1 > 115.1	28	72.7	72.3	63.2	69.0	113.1	112.9	99.1	102.1
4	3,4-Dichloroaniline	161.0 > 99.0	22	161.0 > 126.0	14	74.5	66.2	56.4	64.4	103.8	105.5	105.8	106.0
5	4,4'-Dichlorobenzophenone	139.0 > 111.0	14	139.0 > 75.0	26	77.4	83.2	73.5	73.8	97.5	104.7	102.0	100.3
6	4,4'-methoxychlor olefin	308.0 > 238.1	16	310.0 > 238.1	20	84.4	94.8	85.7	83.0	99.4	101.0	100.0	98.4
7	Acequinocyl deg.	342.2 > 188.1	14	342.2 > 160.1	22	105.3	269.8	201.1	115.2	61.2	62.8	90.7	64.0
8	Acetochlor	223.1 > 132.1	22	223.1 > 147.1	10	77.5	88.3	77.4	77.1	109.0	104.1	102.8	106.2
9	Acrinathrin	289.1 > 93.0	14	181.1 > 152.1	26	102.5	121.4	91.0	82.4	87.0	94.7	92.3	95.1
10	Alachlor	188.1 > 160.1	10	188.1 > 132.1	18	74.1	87.2	77.8	74.1	114.7	107.2	97.3	103.5
11	Aldrin	262.9 > 191.0	34	262.9 > 193.0	28	86.0	66.9	65.2	74.3	79.8	101.1	104.6	93.2
12	Allidochlor	132.1 > 56.0	8	132.1 > 49.0	24	71.8	64.5	59.0	65.9	123.7	118.4	113.4	117.2
13	Anthraquinone	208.1 > 180.1	10	208.1 > 152.1	22	0.0	126.7	47.4	48.0	97.0	83.6	90.4	94.0
14	Atrazine	200.1 > 104.1	18	200.1 > 122.1	8	77.8	94.0	83.3	78.6	105.3	110.1	92.5	100.6
15	Azinphos-ethyl	160.1 > 132.1	4	160.1 > 77.0	18	91.4	115.5	94.5	86.5	102.1	94.8	100.0	98.7
16	Azinphos-methyl	160.1 > 132.1	6	160.1 > 77.0	20	84.1	124.7	93.0	83.3	89.6	103.3	98.2	100.0
17	Benfluralin	292.1 > 264.0	8	292.1 > 160.0	22	78.4	77.0	64.5	70.7	97.7	89.1	96.8	95.3
18	BHC, *alpha*-	180.9 > 144.9	16	218.9 > 182.9	8	62.5	50.4	58.2	66.1	99.0	104.0	99.2	104.3
19	BHC, *beta*-	180.9 > 144.9	16	218.9 > 182.9	8	69.6	87.6	76.8	71.8	102.2	102.9	98.9	104.1
20	BHC, *delta*-	180.9 > 144.9	16	218.9 > 182.9	8	28.6	87.6	69.0	67.8	102.5	104.3	106.4	104.2
21	BHC, *gamma*-	180.9 > 144.9	16	218.9 > 182.9	8	64.1	73.3	62.0	69.8	94.8	93.9	103.1	102.8
22	Bifenthrin	181.1 > 166.1	12	181.1 > 179.1	12	84.6	99.2	89.3	81.4	97.0	104.4	116.7	105.9
23	Bioallethrin	123.1 > 81.1	10	136.1 > 93.1	14	81.7	100.7	83.6	71.9	463.4	563.8	200.4	228.1
24	Biphenyl	154.1 > 128.1	22	154.1 > 115.1	24	90.0	67.9	56.2	65.1	105.5	105.1	106.3	106.0
25	Bromfenvinfos-methyl	294.9 > 109.0	16	296.9 > 109.0	16	85.5	98.5	84.2	77.6	103.5	92.2	100.9	102.7
26	Bromfenvinphos	266.9 > 159.0	14	268.9 > 161.0	16	78.4	97.8	88.2	79.2	102.1	94.7	100.9	102.3
27	Bromophos	330.9 > 315.9	14	328.9 > 313.9	18	70.9	86.2	77.2	76.4	104.6	98.6	97.2	98.6
28	Bromophos-ethyl	358.9 > 302.9	16	302.9 > 284.9	18	78.0	86.9	76.5	75.8	91.9	89.6	98.5	98.4
29	Bromopropylate	340.9 > 182.9	18	340.9 > 184.9	20	85.8	104.5	94.3	85.6	101.4	100.9	101.0	99.2
30	Bupirimate	273.1 > 108.1	16	273.1 > 193.1	8	94.4	102.8	92.2	87.0	82.0	98.3	98.1	92.8
31	Captafol	79.0 > 77.0	14	79.0 > 51.0	20	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
32	Captan	149.1 > 105.1	4	149.1 > 79.1	14	90.7	95.3	83.4	75.3	232.6	240.2	118.7	135.5
33	Carbophenothion	341.9 > 157.0	14	341.9 > 199.0	8	81.9	97.6	84.2	79.2	58.6	66.3	80.4	91.6
34	Carfentrazone-ethyl	340.1 > 312.1	14	312.1 > 151.1	24	85.3	100.6	94.0	87.3	81.5	81.8	101.9	94.7
35	Chlorbenside	125.0 > 99.0	18	127.0 > 89.0	18	80.3	83.4	73.5	71.8	86.9	93.5	96.8	99.3
36	Chlordane, *cis*-	374.8 > 265.9	26	372.8 > 265.9	22	76.4	84.2	76.6	77.1	94.8	99.2	105.3	101.1
37	Chlordane, *trans*-	374.8 > 265.9	26	372.8 > 265.9	22	76.6	77.3	75.9	72.9	96.3	106.5	103.2	102.7
38	Chlorfenapyr	247.1 > 227.0	16	247.1 > 200.0	24	84.3	114.9	87.1	85.0	51.3	71.3	112.2	98.6
39	Chlorfenson	175.0 > 111.0	12	301.9 > 175.0	8	77.0	89.1	81.4	76.4	99.5	100.6	100.5	98.0
40	Chlorfenvinphos, (E)-	323.0 > 267.0	16	267.0 > 159.0	18	110.3	114.8	74.7	72.9	270.4	484.8	103.9	99.9
41	Chlorfenvinphos, (Z)-	323.0 > 267.0	16	267.0 > 159.0	18	85.5	96.6	82.7	78.5	105.8	101.5	104.8	101.2
42	Chlorobenzilate	251.0 > 139.0	14	139.0 > 75.0	26	87.5	106.9	88.9	80.3	98.3	92.0	100.1	101.1
43	Chloroneb	206.0 > 141.0	20	193.0 > 113.0	18	65.4	59.8	59.8	68.6	111.1	110.8	107.9	105.9
44	Chlorothalonil	263.9 > 168.0	24	263.9 > 228.8	18	N.D.	N.D.	5.2	N.D.	N.D.	N.D.	82.3	N.D.
45	Chlorpropham	213.1 > 171.1	6	127.1 > 92.0	18	77.7	81.7	72.5	73.3	99.6	101.7	99.0	101.4
46	Chlorpyrifos	313.9 > 257.9	14	313.9 > 285.9	8	71.2	77.8	74.9	74.2	100.5	93.8	93.6	95.7
47	Chlorpyrifos-methyl	285.9 > 93.0	22	287.9 > 93.0	22	77.0	89.3	73.1	75.3	92.9	91.7	102.6	98.0
48	Chlorthal-dimethyl	298.9 > 220.9	24	300.9 > 222.9	26	78.8	81.9	77.3	73.7	90.7	95.7	96.1	99.9
49	Chlorthiophos-1	256.9 > 239.0	14	256.9 > 193.0	22	91.4	108.5	84.2	83.5	8.4	49.4	77.2	68.6
50	Chlorthiophos-2	324.9 > 268.9	14	268.9 > 205.0	18	76.1	96.8	84.6	80.0	73.8	79.3	95.8	101.5
51	Chlorthiophos-3	324.9 > 268.9	14	268.9 > 205.0	18	78.4	100.2	86.8	77.8	92.6	85.9	100.8	97.8
52	Chlozolinate	258.9 > 188.0	14	330.9 > 258.9	6	75.6	82.2	75.9	76.1	91.7	98.3	103.8	104.3
53	Clomazone	204.1 > 107.0	20	204.1 > 78.0	26	72.7	72.2	70.9	74.9	100.7	99.6	101.8	98.6
54	Coumaphos	362.0 > 109.0	16	362.0 > 226.0	14	90.1	110.4	98.2	89.7	84.4	84.7	94.5	94.9
55	Cycloate	154.2 > 72.0	6	215.1 > 154.2	4	69.8	66.7	61.2	69.5	89.3	89.9	93.3	94.0
56	Cyfluthrin-1	226.1 > 206.1	14	163.1 > 127.1	6	92.8	111.7	98.2	92.1	105.2	118.8	100.9	96.1
57	Cyfluthrin-2	226.1 > 206.1	14	163.1 > 127.1	6	92.5	113.1	91.9	90.2	124.6	121.5	105.1	97.6
58	Cyfluthrin-3	226.1 > 206.1	14	163.1 > 127.1	6	81.4	96.8	92.5	81.7	117.8	160.7	124.5	128.0
59	Cyfluthrin-4	226.1 > 206.1	14	163.1 > 127.1	6	76.5	95.7	106.8	88.7	167.1	159.4	121.8	126.4
60	Cyhalothrin, *lambda*-	208.1 > 181.1	8	197.1 > 141.0	12	88.4	108.0	92.1	85.5	99.2	113.2	99.5	96.9
61	Cypermethrin-1	163.1 > 127.1	6	163.1 > 109.1	22	96.0	113.8	94.6	89.9	108.8	93.7	117.4	111.3
62	Cypermethrin-2	163.1 > 127.1	6	163.1 > 109.1	22	89.6	119.7	98.0	88.7	99.2	113.1	94.8	100.1
63	Cypermethrin-3	163.1 > 127.1	6	163.1 > 109.1	22	75.9	124.9	103.1	96.9	126.1	114.1	96.6	104.1
64	Cypermethrin-4	163.1 > 127.1	6	163.1 > 109.1	22	76.6	100.1	84.6	81.5	115.3	119.9	124.9	120.1
65	Cyprodinil	224.1 > 197.1	22	224.1 > 131.1	14	82.0	88.9	80.0	71.2	138.1	117.5	100.5	112.6
66	DDD, *o,p*'-	235.0 > 165.0	24	235.0 > 199.0	16	83.6	96.0	79.8	75.3	87.0	86.6	100.1	99.1
67	DDD, *p,p*'-	235.0 > 165.0	24	235.0 > 199.0	16	80.2	94.6	83.6	78.2	104.4	98.8	102.6	104.3
68	DDE, *o,p*'-	246.0 > 176.0	30	248.0 > 176.0	28	73.7	83.1	75.7	71.7	101.7	97.9	99.0	100.3
69	DDE, *p,p*'-	246.0 > 176.0	30	317.9 > 248.0	24	76.9	99.7	76.2	73.5	101.1	93.8	98.8	98.3
70	DDT, *o,p*'-	235.0 > 165.0	24	235.0 > 199.0	16	79.0	89.1	78.0	74.3	93.9	97.6	100.1	101.2
71	DDT, *p,p*'-	235.0 > 165.0	24	235.0 > 199.0	16	75.2	95.1	80.7	75.8	96.8	95.0	102.9	97.7
72	Deltamethrin	252.9 > 93.0	20	252.9 > 171.9	8	83.8	109.4	92.2	85.3	99.9	99.3	102.5	99.7
73	Di-allate-1	234.1 > 150.0	20	234.1 > 192.1	14	75.3	66.5	63.3	70.7	93.8	93.0	100.9	103.3
74	Di-allate-2	234.1 > 150.0	20	234.1 > 192.1	14	72.4	65.1	63.0	70.5	88.9	107.7	100.0	94.1
75	Diazinon	304.1 > 179.1	10	304.1 > 162.1	8	76.4	70.3	69.5	74.4	62.3	80.6	97.9	86.4
76	Dichlobenil	170.9 > 100.0	24	170.9 > 136.0	14	71.2	65.3	58.2	65.4	96.5	97.4	96.5	98.2
77	Dichlofluanid	223.9 > 123.1	8	223.9 > 77.0	28	65.0	71.1	57.8	56.9	75.4	87.4	94.4	101.7
78	Dicloran	206.0 > 176.0	10	206.0 > 124.0	24	78.1	76.6	69.7	77.0	79.0	87.7	100.6	91.0

ID	Compound Name	Transitions				Recovery (Average of n = 3)				Relative Ion Ratio (Average of n = 3)			
		Target	CE	Reference	CE	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2
79	Dieldrin	276.9 > 241.0	8	262.9 > 193.0	34	80.2	80.3	89.0	85.0	154.2	185.9	102.7	91.4
80	Dimethachlor	197.1 > 148.1	10	199.1 > 148.1	10	78.4	90.6	77.9	76.8	99.6	89.1	100.2	101.6
81	Diphenamid	239.1 > 167.1	8	239.1 > 72.0	16	94.7	96.0	88.3	79.8	126.1	123.6	112.2	115.5
82	Diphenylamine	169.1 > 66.0	24	169.1 > 77.0	28	78.2	74.6	65.6	71.1	85.3	99.9	98.3	100.6
83	Disulfoton	186.0 > 153.0	6	186.0 > 97.0	16	71.4	66.2	64.6	78.9	142.4	124.1	107.9	85.8
84	Edifenphos	173.0 > 109.0	10	310.0 > 173.0	14	82.6	103.7	91.0	83.6	97.9	94.6	100.3	98.2
85	Endosulfan ether	240.9 > 205.9	16	238.9 > 203.9	16	62.7	64.4	67.2	69.5	126.7	95.3	104.8	101.2
86	Endosulfan sulfate	271.8 > 236.9	18	386.8 > 252.9	16	83.8	88.4	87.0	85.1	42.5	55.6	83.3	85.8
87	Endosulfan, *alpha*-	194.9 > 160.0	8	194.9 > 125.0	24	74.0	80.0	75.6	80.7	77.6	76.9	92.3	83.4
88	Endosulfan, *beta*-	194.9 > 160.0	8	194.9 > 125.0	24	83.1	99.4	81.6	81.7	102.1	67.3	93.2	100.2
89	Endrin	262.9 > 193.0	28	262.9 > 228.0	22	84.1	89.9	77.2	75.3	50.2	49.4	85.3	88.2
90	Endrin aldehyde	249.8 > 214.9	26	344.9 > 244.9	16	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
91	Endrin ketone	316.9 > 244.9	20	314.9 > 242.9	18	73.4	91.5	84.1	88.0	108.9	95.4	107.6	101.9
92	EPN	169.1 > 140.9	8	169.1 > 77.0	22	90.3	107.4	89.2	82.3	94.3	102.9	100.2	98.8
93	Ethalfluralin	276.0 > 202.0	18	316.1 > 276.0	10	73.3	67.7	63.0	67.1	95.2	96.4	100.9	100.6
94	Ethion	230.9 > 129.0	24	230.9 > 174.9	14	81.4	97.1	84.2	77.9	118.3	108.6	101.8	102.6
95	Etofenprox	163.1 > 135.1	10	163.1 > 107.1	18	87.1	101.1	95.6	87.3	97.4	103.1	98.8	97.8
96	Etridiazole	210.9 > 182.9	10	210.9 > 139.9	22	70.0	60.1	58.0	66.9	88.2	102.0	102.0	100.4
97	Fenamiphos	303.1 > 195.1	8	288.1 > 260.1	6	88.7	104.6	99.3	87.1	120.9	106.0	96.8	100.1
98	Fenarimol	251.0 > 139.0	14	330.0 > 139.0	8	83.9	106.7	96.5	89.4	97.4	92.6	99.3	96.0
99	Fenchlorphos	284.9 > 269.9	16	286.9 > 271.9	18	80.5	81.4	71.5	74.8	89.8	83.8	99.1	97.2
100	Fenitrothion	277.0 > 260.0	6	260.0 > 125.1	12	78.0	89.3	79.3	75.8	104.9	99.2	111.4	109.6
101	Fenpropathrin	265.1 > 210.1	12	265.1 > 89.0	28	90.3	102.8	96.8	88.2	68.9	75.9	90.9	94.4
102	Fenson	141.0 > 77.0	16	267.9 > 141.0	6	78.3	88.0	77.9	74.5	91.3	92.7	100.8	96.8
103	Fenthion	278.0 > 169.0	14	278.0 > 125.0	20	80.4	81.9	78.2	76.0	93.9	96.0	102.2	104.1
104	Fenvalerate-1	225.1 > 147.1	10	419.1 > 225.1	6	82.2	100.7	94.6	88.9	95.5	92.4	103.2	99.3
105	Fenvalerate-2	225.1 > 147.1	10	419.1 > 225.1	6	75.7	106.7	93.1	86.5	73.5	70.0	86.2	89.7
106	Fipronil	366.9 > 212.9	30	368.9 > 214.9	30	92.8	113.2	99.5	82.8	93.8	96.2	88.0	96.7
107	Fluazifop-P-butyl	282.1 > 91.0	18	383.1 > 282.1	14	77.4	94.0	89.0	80.6	101.9	96.4	99.6	100.5
108	Fluchloralin	306.0 > 264.0	8	326.0 > 63.0	16	76.4	78.4	76.9	75.1	95.8	96.0	90.4	96.5
109	Flucythrinate-1	157.1 > 107.1	12	199.1 > 107.1	22	86.9	111.9	95.6	87.3	119.8	120.4	107.1	105.2
110	Flucythrinate-2	157.1 > 107.1	12	199.1 > 107.1	22	89.4	111.0	96.0	86.6	105.1	108.4	102.6	104.6
111	Fludioxonil	248.0 > 127.0	26	248.0 > 154.0	20	94.1	110.3	96.5	86.1	91.9	101.1	98.1	96.0
112	Fluquinconazole	340.0 > 298.0	20	340.0 > 313.0	14	85.2	107.0	98.9	88.9	97.7	98.5	101.4	101.1
113	Fluridone	328.1 > 259.0	24	328.1 > 127.0	24	91.5	117.7	100.3	91.4	80.1	77.3	93.4	95.7
114	Flusilazole	233.1 > 165.1	14	233.1 > 152.1	14	78.1	93.3	84.9	83.2	105.9	91.5	94.8	95.4
115	Flutolanil	173.0 > 95.0	26	281.1 > 173.0	12	84.1	108.0	93.4	84.7	104.1	101.1	100.0	99.8
116	Flutriafol	219.1 > 123.1	14	219.1 > 95.0	28	82.9	109.6	89.8	84.2	120.1	115.2	103.9	103.9
117	Fluvalinate-1, *tau*-	250.1 > 55.0	18	250.1 > 200.1	16	86.7	112.8	91.9	85.5	79.4	79.9	90.8	90.4
118	Fluvalinate-2, *tau*-	250.1 > 55.0	18	250.1 > 200.1	16	90.4	112.0	89.1	83.9	89.6	95.5	102.5	104.7
119	Folpet	259.9 > 130.0	14	261.9 > 130.0	18	65.2	82.9	72.8	67.0	97.2	89.1	99.1	98.3
120	Fonofos	246.0 > 137.1	6	246.0 > 109.1	18	76.6	74.7	65.8	71.4	106.8	97.6	102.1	101.2
121	Heptachlor	271.8 > 236.9	20	273.8 > 238.9	16	68.6	65.4	65.0	68.6	92.3	102.0	104.0	101.2
122	Heptachlor-exo-epoxide	352.8 > 262.9	14	352.8 > 316.9	10	81.1	80.4	71.8	79.1	33.7	39.0	88.6	71.5
123	Hexachlorobenzene	283.8 > 248.8	24	283.8 > 213.8	28	45.7	28.8	48.1	62.3	101.4	100.7	102.7	99.3
124	Hexazinone	171.1 > 71.0	16	171.1 > 85.0	16	93.2	112.9	96.1	88.2	105.3	101.3	104.0	105.2
125	Iodofenphos	376.9 > 361.8	22	376.9 > 331.8	32	80.0	86.8	75.8	74.2	83.8	102.3	97.3	97.1
126	Iprodione	314.0 > 245.0	12	314.0 > 56.0	22	110.5	156.0	102.0	89.9	91.0	99.9	102.2	107.0
127	Isazofos	257.0 > 162.0	8	257.0 > 119.0	18	80.0	87.4	74.4	74.7	109.5	107.1	96.0	102.6
128	Isodrin	192.9 > 157.0	20	262.9 > 192.9	28	76.4	73.7	67.4	71.2	88.2	99.0	110.1	98.6
129	Isopropalin	280.1 > 238.1	8	280.1 > 133.1	18	76.5	88.1	73.6	71.4	89.8	82.7	97.9	106.1
130	Lenacil	153.1 > 136.1	14	153.1 > 82.1	16	85.1	103.2	97.5	85.7	109.0	108.9	124.1	111.8
131	Leptophos	376.9 > 361.9	24	374.9 > 359.9	24	86.8	102.6	89.0	82.6	95.4	99.8	101.9	103.0
132	Linuron	248.0 > 61.0	16	250.0 > 61.0	16	68.3	84.7	77.7	77.9	98.3	83.1	105.8	108.6
133	Malathion	173.1 > 99.0	14	158.1 > 125.0	10	79.9	87.3	77.6	75.3	95.2	97.4	101.3	100.9
134	Metalaxyl	249.2 > 190.1	8	249.2 > 146.1	22	83.5	88.7	89.5	81.1	103.0	122.4	104.0	100.7
135	Metazachlor	209.1 > 132.1	18	211.1 > 132.1	20	83.3	93.2	83.8	80.1	97.8	100.1	101.6	104.8
136	Methacrifos	208.0 > 180.0	8	240.0 > 208.0	4	75.6	68.7	62.9	67.2	93.3	89.0	95.3	101.8
137	Methoxychlor	227.1 > 169.1	24	227.1 > 212.1	14	82.8	101.8	90.8	83.5	109.0	108.5	103.5	103.2
138	Metolachlor	238.1 > 162.1	12	238.1 > 133.1	26	77.0	89.0	77.9	75.4	104.8	96.6	101.4	102.3
139	Mevinphos-1	192.0 > 127.0	12	127.0 > 95.0	18	75.7	70.7	64.1	71.4	95.8	104.8	103.6	102.2
140	MGK 264-1	164.1 > 93.0	10	164.1 > 80.0	24	112.8	102.9	86.2	81.3	92.5	104.4	108.1	104.7
141	MGK 264-2	164.1 > 98.0	12	164.1 > 67.0	8	74.8	89.7	79.3	78.2	116.0	100.8	93.7	96.0
142	Mirex	271.8 > 236.8	18	273.8 > 238.8	18	71.4	80.4	73.6	71.2	99.9	97.3	100.7	99.6
143	Myclobutanil	179.1 > 125.0	14	179.1 > 152.0	8	83.7	104.4	90.8	84.1	122.4	127.5	112.5	108.8
144	N-(2,4-dimethylphenyl) formamide	149.1 > 106.1	16	149.1 > 121.1	6	86.6	88.5	73.0	71.4	334.2	367.6	157.5	169.6
145	Nitralin	316.1 > 274.0	8	274.0 > 169.0	12	101.1	116.0	95.4	87.2	98.7	104.0	106.2	97.4
146	Nitrofen	202.0 > 139.0	24	282.9 > 253.0	12	83.4	91.9	85.9	77.0	89.0	95.2	99.6	104.3
147	Nonachlor, *cis*-	406.8 > 299.9	24	406.8 > 334.9	16	80.0	91.3	79.8	77.0	59.7	48.5	84.3	87.0
148	Nonachlor, *trans*-	406.8 > 299.9	24	406.8 > 334.9	16	74.8	86.0	80.8	78.2	58.4	72.4	82.3	84.6
149	Norflurazon	303.0 > 145.0	22	145.0 > 95.0	18	94.7	111.0	98.7	85.0	92.7	95.4	99.2	100.8
150	Oxadiazon	258.0 > 175.0	8	302.0 > 175.0	14	78.8	88.7	84.7	76.9	95.1	101.4	100.4	103.2
151	Oxyfluorfen	361.0 > 300.0	14	361.0 > 317.0	6	101.4	106.2	90.9	80.4	91.2	97.7	101.4	102.1
152	Paclobutrazol	236.1 > 125.0	14	236.1 > 167.0	10	92.2	116.7	92.8	86.1	98.0	86.2	100.9	100.1
153	Parathion	291.1 > 137.0	6	291.1 > 81.0	24	99.8	96.8	80.7	76.0	100.1	115.2	104.9	108.1
154	Parathion-methyl	263.0 > 109.0	14	263.0 > 246.0	6	85.5	91.6	74.2	77.4	71.7	69.8	96.2	93.7
155	Pebulate	161.1 > 128.1	6	128.1 > 57.0	6	64.1	59.1	56.9	66.7	115.3	111.1	101.3	101.7
156	Penconazole	248.1 > 157.1	26	159.1 > 123.1	22	90.9	92.6	75.3	75.4	90.0	94.4	97.6	97.5

ID	Compound Name	Transitions				Recovery (Average of n = 3)				Relative Ion Ratio (Average of n = 3)			
		Target	CE	Reference	CE	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2	10 ng/g-1	10 ng/g-2	50 ng/g-1	50 ng/g-2
157	Pendimethalin	252.1 > 162.1	10	252.1 > 191.1	8	84.0	84.2	73.9	73.8	89.5	98.9	102.2	104.0
158	Pentachloroaniline	262.9 > 191.9	22	264.9 > 193.9	18	23.6	83.6	52.0	72.4	99.4	99.0	101.1	98.8
159	Pentachloroanisole	279.9 > 236.8	26	279.9 > 264.8	12	69.0	65.8	60.2	67.9	102.8	93.3	101.7	101.4
160	Pentachlorobenzene	249.9 > 214.9	18	249.9 > 176.9	26	59.9	27.0	47.6	62.0	104.3	113.2	108.4	104.3
161	Pentachlorobenzonitrile	274.8 > 239.8	18	272.8 > 202.9	30	69.3	65.6	63.2	68.7	90.1	88.2	96.3	95.7
162	Pentachlorothioanisole	295.8 > 262.9	14	295.8 > 245.8	30	55.8	75.7	62.7	69.7	92.4	96.3	94.9	93.5
163	Permethrine, *cis*-	183.1 > 153.1	14	183.1 > 168.1	14	86.6	112.1	96.8	87.5	101.7	108.3	99.5	100.2
164	Permethrine, *trans*-	183.1 > 153.1	14	183.1 > 168.1	14	96.0	131.5	97.0	88.4	100.4	98.3	103.6	102.4
165	Perthane	223.2 > 167.1	14	223.2 > 193.1	28	85.3	92.7	81.9	78.1	97.1	106.8	102.3	100.3
166	Phenothrin-1	183.1 > 153.1	14	183.1 > 168.1	14	N.D.	N.D.	N.D.	84.8	N.D.	N.D.	111.5	92.4
167	Phenothrin-2	183.1 > 153.1	14	183.1 > 168.1	14	100.7	113.7	95.7	86.7	101.4	112.1	105.1	100.1
168	Phorate	260.0 > 75.0	8	231.0 > 129.0	24	74.3	63.8	61.9	69.7	92.7	116.3	99.8	101.6
169	Phosalone	182.0 > 102.0	14	182.0 > 111.0	14	86.3	101.6	93.9	84.4	117.0	123.8	101.2	104.5
170	Phosmet	160.0 > 77.0	24	160.0 > 105.0	18	86.7	105.4	92.6	83.0	100.8	100.1	103.5	101.2
171	Piperonyl butoxide	176.1 > 131.1	12	176.1 > 117.1	20	84.1	112.8	92.1	85.9	104.9	118.6	102.2	102.3
172	Pirimiphos ethyl	304.1 > 168.1	12	318.1 > 166.1	12	78.1	94.3	79.1	73.8	83.5	84.4	97.4	105.8
173	Pirimiphos-methyl	290.1 > 125.0	22	290.1 > 233.1	12	80.4	88.9	78.5	76.1	93.3	92.5	101.2	99.4
174	Pretilachlor	262.1 > 202.1	10	238.1 > 162.1	10	77.0	95.2	86.0	79.7	109.6	79.5	93.4	102.5
175	Prochloraz	180.1 > 138.1	12	180.1 > 69.0	20	68.1	110.1	85.4	81.5	106.2	102.1	91.1	91.2
176	Procymidone	283.0 > 96.0	10	285.0 > 96.0	10	12.4	140.1	78.8	79.4	103.0	101.7	104.7	101.8
177	Prodiamine	321.1 > 279.1	6	321.1 > 203.1	10	86.1	94.7	81.6	78.3	88.5	88.3	93.6	99.0
178	Profenofos	338.9 > 268.9	18	336.9 > 266.9	14	87.1	93.5	90.0	85.6	107.6	107.6	93.3	91.1
179	Profluralin	318.1 > 199.1	16	318.1 > 55.0	22	66.3	64.9	65.7	74.8	105.2	95.9	97.6	91.7
180	Propachlor	176.1 > 57.0	8	176.1 > 77.0	24	74.9	76.4	67.3	71.4	114.9	106.5	103.8	102.7
181	Propanil	217.0 > 161.0	10	160.9 > 126.0	18	93.5	105.3	91.4	78.7	100.7	106.9	95.8	104.6
182	Propargite	173.1 > 135.1	16	173.1 > 107.1	24	88.0	98.7	90.8	85.5	120.0	122.8	74.1	66.1
183	Propisochlor	223.1 > 132.1	20	223.1 > 147.1	8	83.7	90.8	79.4	77.9	91.0	100.1	98.4	101.0
184	Propyzamide	172.9 > 109.0	26	172.9 > 74.0	28	83.0	91.4	77.1	76.5	98.7	100.0	108.7	106.3
185	Prothiofos	266.9 > 238.9	10	309.0 > 238.9	14	74.3	88.9	78.0	73.9	102.4	94.4	102.7	101.2
186	Pyraclofos	194.0 > 138.0	22	360.1 > 194.0	14	91.6	111.4	97.0	87.0	84.1	89.9	99.3	100.1
187	Pyrazophos	221.1 > 193.1	12	221.1 > 149.1	14	88.7	111.9	97.3	86.1	101.7	96.0	99.9	103.7
188	Pyridaben	147.1 > 117.1	22	147.1 > 132.1	14	87.5	106.4	92.5	84.1	99.2	105.1	102.0	102.5
189	Pyridaphenthion	340.0 > 199.1	8	199.1 > 92.0	16	100.2	120.0	95.0	89.3	114.1	129.2	109.5	107.5
190	Pyrimethanil	198.1 > 118.1	28	198.1 > 158.1	18	74.4	82.5	75.3	73.2	95.2	99.1	96.4	98.5
191	Pyriproxyfen	136.1 > 96.0	14	226.1 > 186.1	14	81.3	91.9	92.8	84.4	59.7	78.1	88.1	84.8
192	Quinalphos	146.1 > 118.0	10	146.1 > 91.0	24	71.7	86.6	79.6	72.9	204.6	155.7	122.4	117.8
193	Quintozene	294.8 > 236.8	16	264.8 > 236.8	10	61.7	0.0	18.5	71.1	100.1	100.7	101.5	100.3
194	Resmethrin-1	171.1 > 128.1	12	171.1 > 143.1	6	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
195	Resmethrin-2	171.1 > 143.1	6	171.1 > 128.1	14	85.8	96.4	85.8	78.1	86.0	97.9	95.6	100.0
196	Sulfotep	322.0 > 294.0	4	322.0 > 202.0	10	79.1	71.8	65.5	71.8	94.6	98.9	98.0	95.0
197	Sulprofos	322.0 > 156.0	8	156.0 > 108.0	28	80.7	97.3	86.0	83.2	101.1	95.9	97.3	95.1
198	Tebuconazole	250.1 > 125.1	22	250.1 > 153.1	12	76.8	82.9	68.1	71.6	112.5	96.6	98.9	98.1
199	Tebufenpyrad	333.1 > 171.1	20	333.1 > 276.1	8	87.7	105.2	96.7	84.7	97.6	97.3	94.6	95.6
200	Tecnazene	260.9 > 202.9	14	202.9 > 85.0	24	78.8	59.9	59.7	72.4	93.9	99.1	96.8	95.8
201	Tefluthrin	177.0 > 127.1	16	177.0 > 137.1	16	79.0	81.2	67.5	72.5	92.6	94.9	101.0	98.3
202	Terbacil	161.0 > 88.0	20	117.0 > 76.0	8	97.5	111.6	86.5	82.0	81.8	77.9	92.5	96.1
203	Terbufos	231.0 > 128.9	26	231.0 > 174.9	14	78.8	80.6	65.2	70.3	111.6	100.1	102.8	104.7
204	Terbuthylazine	229.1 > 173.1	6	214.1 > 71.0	16	94.9	91.0	77.6	77.1	82.6	91.6	99.7	103.3
205	Tetrachlorvinphos	328.9 > 109.0	20	330.9 > 109.0	22	87.4	102.5	87.3	81.0	96.0	93.6	99.9	96.6
206	Tetradifon	355.9 > 159.0	18	355.9 > 228.9	12	84.1	96.6	93.6	88.5	87.9	103.1	102.9	96.7
207	Tetramethrin-1	164.1 > 107.1	14	164.1 > 77.0	22	N.D.	N.D.	100.5	94.8	N.D.	N.D.	106.5	114.1
208	Tetramethrin-2	164.1 > 107.1	14	164.1 > 77.0	22	103.2	129.6	98.4	88.4	115.2	117.0	107.9	113.8
209	THPI	151.1 > 79.0	18	151.1 > 77.0	28	79.4	85.0	81.5	78.2	104.7	104.1	103.6	108.0
210	Tolclofos-methyl	264.9 > 93.0	24	264.9 > 219.9	22	72.8	78.6	72.6	73.8	98.8	100.9	102.3	103.6
211	Tolylfluanid	238.0 > 137.1	14	181.1 > 138.1	10	66.8	79.8	65.9	64.1	118.7	117.3	114.3	107.4
212	Transfluthrin	163.1 > 127.1	6	163.1 > 143.1	16	84.2	85.7	77.1	77.6	106.3	115.0	97.7	99.0
213	Triadimefon	208.1 > 111.0	22	208.1 > 127.0	14	88.4	98.2	86.7	80.5	99.5	103.9	97.9	104.7
214	Triadimenol	168.1 > 70.0	10	128.1 > 65.0	22	N.D.	N.D.	101.2	94.2	N.D.	N.D.	80.9	85.6
215	Tri-allate	268.1 > 184.0	20	270.1 > 186.0	20	80.0	75.6	68.6	74.6	102.4	85.9	94.6	98.1
216	Triazophos	257.0 > 162.0	8	257.0 > 134.0	22	89.7	112.4	94.8	86.4	95.9	80.8	90.3	88.2
217	Tricyclazole	189.0 > 161.9	12	189.0 > 135.0	18	91.5	95.7	84.5	81.8	105.9	118.7	105.2	97.3
218	Triflumizole	278.1 > 73.0	6	206.1 > 186.1	8	87.2	88.2	71.5	76.8	83.2	110.9	97.0	99.2
219	Trifluralin	306.1 > 264.1	8	306.1 > 160.1	22	79.9	73.6	64.4	72.5	92.7	100.3	100.8	101.1
220	Vinclozolin	285.0 > 212.0	12	212.0 > 172.0	16	86.3	92.8	79.9	80.7	84.3	96.4	104.2	103.3
QC-1	1,4-Dichlorobenzene-d4	150.0 > 78.0	24	115.1 > 78.0	12	—	—	—	—	—	—	—	—
QC-2	Acenaphthene-d10	164.0 > 160.0	30	164.0 > 134.0	38	—	—	—	—	—	—	—	—
QC-3	Chrysene-d12	240.0 > 236.0	30	240.0 > 212.0	24	—	—	—	—	—	—	—	—
QC-4	Naphthalene-d8	136.0 > 84.0	22	136.0 > 82.0	28	—	—	—	—	—	—	—	—
QC-5	Perylene-d12	264.0 > 263.0	34	264.0 > 262.0	24	—	—	—	—	—	—	—	—
QC-6	Phenanthrene-d10	188.0 > 160.0	24	187.0 > 159.0	18	—	—	—	—	—	—	—	—
IS-1	2,2',5-Trichlorobiphenyl	255.9 > 186.0	26	257.9 > 186.0	26	—	—	—	—	—	—	—	—
IS-2	2,4,4'-Trichlorobiphenyl	255.9 > 186.0	26	257.9 > 186.0	26	—	—	—	—	—	—	—	—
IS-3	2,2',5,5'-Tetrachlorobiphenyl	257.0 > 222.0	12	292.0 > 220.0	26	—	—	—	—	—	—	—	—
IS-4	Triphenylmethane	244.1 > 167.1	16	244.1 > 165.1	26	—	—	—	—	—	—	—	—
IS-5	Triphenylphosphate	215.1 > 168.1	16	325.1 > 169.1	20	—	—	—	—	—	—	—	—
IS-6	Tris(1,3-dichloroisopropyl)phosphate	379.0 > 159.0	12	381.0 > 159.0	12	—	—	—	—	—	—	—	—

Compounds outside the red box (Fig. 5) were classified to four groups.

Group A showed low recovery; this group consisted mainly of compounds which have low boiling point. They may have been lost in the evaporation step. Group B showed high relative ion ratios and this was caused by interference from matrix. Group C showed high recovery; this group consisted of 10 ng/g fortified sample. Some of these were in matrix originally and quantified incorrectly. Others caused by their transitions which had low response and low stability. Group D showed low relative ion ratio. It was necessary to set higher response transitions. By modifying some procedures and parameters, positions of these compounds may improve.

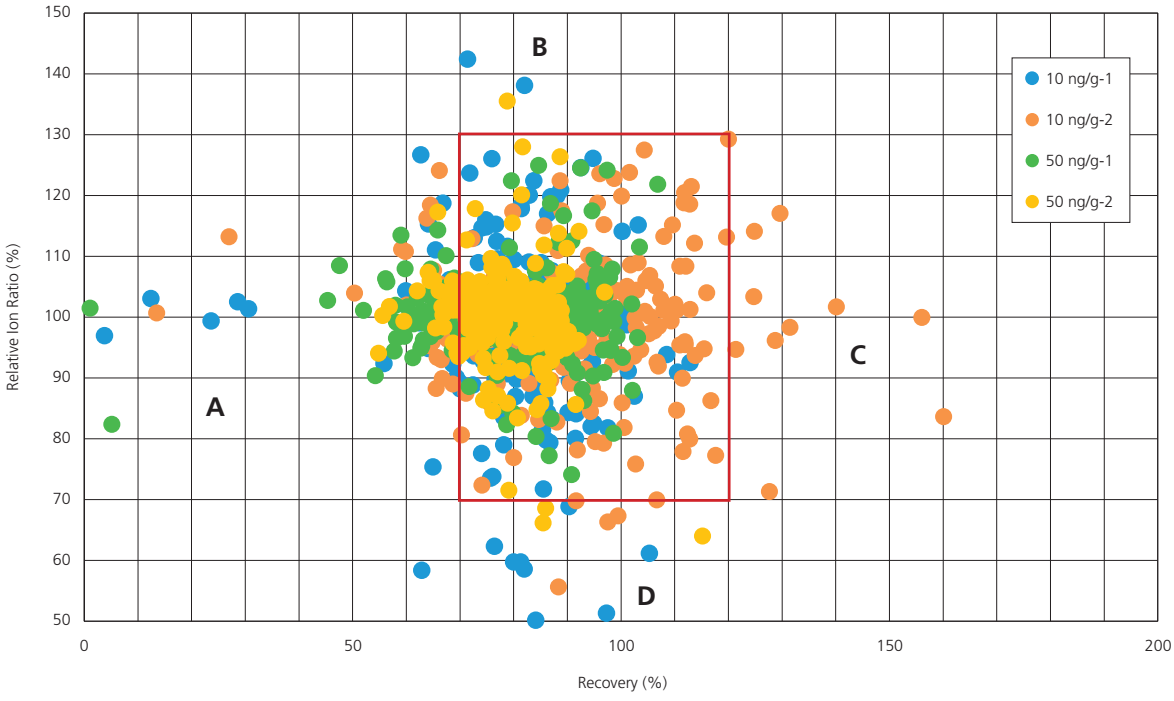


Fig. 5 Combination Map Between Recovery and Ion Ratio (Average of n = 3 for Each Fortified Samples)
Red box shows the area of 70–120% recovery and ±30% relative ion ratio.

4. Conclusion

This study shows that the modified QuEChERS method combined with GC-MS/MS achieved consistent pesticides monitoring in botanical ingredients.

Although dried sample could make a heavy and difficult matrix, the modified QuEChERS method, SPE column cleanup, and toluene dilution steps suppressed interference from matrix. The GC-MS/MS detected very low amount of pesticides even though the sample was diluted. This analytical method takes only 30 minutes in total run time and covers over 200 pesticides. It provides a high throughput solution in laboratories doing this type of analysis.

Reference

1) M. Anastassiades, S. J. Lehotay, D. Štajnbaher, F. J. Schenck, Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce, *J. AOAC Int.*, **86** (2003) 412–431
2) European Commission, Health & Consumer Protection Directorate-General, Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed, SANCO/12571/2013

Triple Quadrupole Gas Chromatograph Mass Spectrometer

GCMS-TQ8040

Smart Performance That Boosts Routine Analytical Work

GC-MS/MS is useful for measuring trace quantities of various chemical substances present in a variety of sample types. However, specifying several parameter settings and employing suitable methods are required when using this technique.
Nevertheless, GCMS-TQ8040 can dramatically increase the productivity by automating tedious method creation processes and simultaneously analyzing multiple components with high sensitivity.

Smart Productivity

- Includes a new firmware protocol.
- Simultaneously analyzes a wide range of compounds with high sensitivity and high accuracy.
- Twin Line MS system minimizes the replacement of columns.

Smart Operation

- Smart MRM automatically creates optimized methods.
- Automatically searches for optimal transitions.
- AART function automatically adjusts retention times.

Smart Performance

- Patented high-sensitivity ion source technology offers even higher sensitivity.
- OFF-AXIS Ion Optics reduces noise.
- Capable of performing high-sensitivity analysis even as a single GC-MS system.

GC/MS Residual Pesticides Database

Smart Pesticides Database Ver. 2

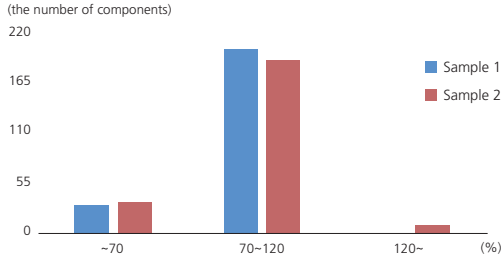
Supports the Simultaneous Analysis of 530 Residual Pesticides in Foods via GC-MS(/MS)

Smart Pesticides Database contains the retention indices and transitions for 530 pesticides. Ver. 2 additionally contains measurement ions for SIM mode, so it can be applied to both SIM and MRM analyses. Thanks to the retention indices contained in the database and the AART function, retention times can be revised automatically without the use of pesticide standards.
Furthermore, the Smart MRM/SIM function allows automatic creation of the optimal measurement programs for multicomponent simultaneous analysis using MRM and SIM modes. Lastly, the database can be customized to the GC conditions and the addition of new components.

Compound Name (E)	Ret. Index 1	Ret. Index 2	Ret. Index 3	Ion1				Ion2				Ion1			Ion2		
				Type	m/z	CE	Ratio	Type	m/z	CE	Ratio	Type	m/z	Ratio	Type	m/z	Ratio
Hymexazol	1201	1193	1191	T	99.0>71.0	8	100.00	Ref.1	99.0>54.0	26	6.13	T	99.0	100.00	Ref.1	71.0	18.40
Methamidophos	1240	1231	1229	T	141.0>95.0	8	100.00	Ref.1	141.0>79.0	22	31.28	T	141.0	100.00	Ref.1	94.0	337.84
Dichlorvos	1248	1244	1243	T	109.0>79.0	8	100.00	Ref.1	185.0>93.0	14	56.91	T	185.0	100.00	Ref.1	109.0	403.23
Nereistoxin	1285	1284	1274	T	149.1>71.1	8	100.00	Ref.1	149.1>102.1	6	67.94	T	70.0	100.00	Ref.1	149.0	51.20
Allidochlor	1290	1288	1283	T	132.1>56.0	8	100.00	Ref.1	138.1>96.0	6	26.15	T	138.0	100.00	Ref.1	173.0	6.98
Dichlobenil	1348	1345	1335	T	170.9>136.0	14	100.00	Ref.1	170.9>100.0	24	98.06	T	171.0	100.00	Ref.1	173.0	76.00
EPTC	1359	1358	1353	T	189.1>128.1	4	100.00	Ref.1	189.1>86.0	12	22.96	T	128.0	100.00	Ref.1	189.0	25.60
Biphenyl	1393	1391	1380	T	154.1>128.1	22	100.00	Ref.1	154.1>115.1	24	74.03	T	154.0	100.00	Ref.1	153.0	37.60
Propamocarb	1394	1393	1390	T	188.2>72.0	4	100.00	Ref.1	188.2>173.2	4	72.45	T	58.0	100.00	Ref.1	129.0	2.40
Mevinphos-1	1420	1420	1419	T	127.0>109.0	12	100.00	Ref.1	192.0>127.0	12	61.12	T	127.0	100.00	Ref.1	192.0	31.60

Verifying the Effectiveness of Recovery Tests of Health Foods

In conducting recovery tests in health foods for 220 pesticides, using the database, recommended pretreatment kit, pretreatment protocol, and certified standard substances, we succeeded in obtaining excellent ratios in over 80 percent of components.
Health foods contain many contaminants. Because it includes transitions for the separation of contaminants, the database minimizes their impact.
Additionally, the database can separate contaminants by allowing analysis with a different column, even if peaks of target pesticides and contaminants overlap. If the database is used in combination with the Twin Line MS system, analysis with different columns can be performed smoothly, without compromising the MS vacuum.



Distribution of rates for two health food samples which were spiked with a pesticide standard sample so as to obtain a final concentration of 2.5 ng/mL each.

First Edition: December, 2016

Shimadzu Corporation
www.shimadzu.com/an/

Printed in Japan 3655-11615-10ANS