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

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Introduction

The use of dietary supplements is increasing in the United States. These dietary supplements are made from various dried botanicals, which may contain residual pesticides from agricultural practices, 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 the best technique to separate multicomponents including coextracted interferences, and because the triple quadruple mass spectrometer (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.

Materials and Methods

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

- GC Multiresidue Pesticide Kit (cat.# 32562)
- QuEChERS Internal Standard Mix for GC-MS Analysis (cat.# 33267)
- SV Internal Standard Mix (cat.# 31206)
- Q-sep QuEChERS Extraction Kit (Original cat.# 23991 discontinued; see cat.# 25848)

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 an internal standard method, weighted 1/C and the internal standard was PCB52.

An 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 (Figure 1).

Figure 1: Data Acquisition Time Program Created by Smart MRM

Extraction and Clean-up Procedure

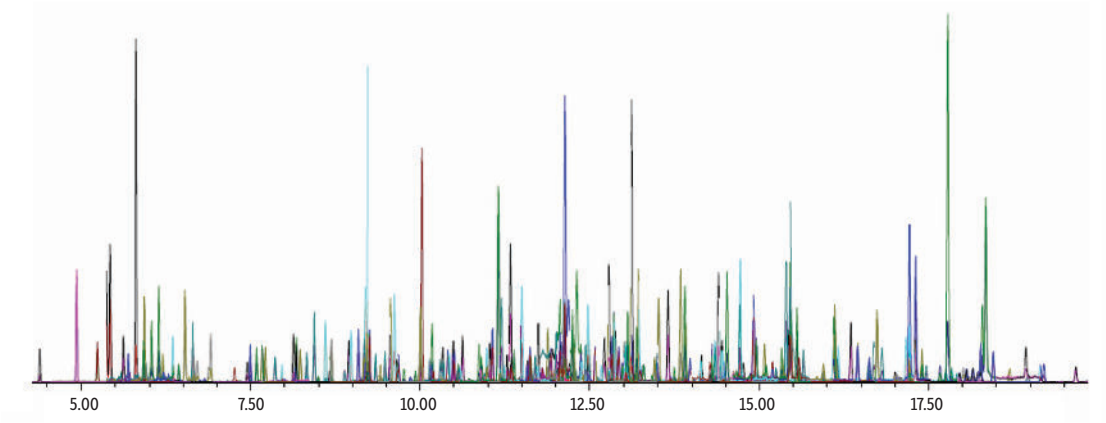
- Weigh 1.0 ± 0.05 g ground ginseng powder into 50 mL polypropylene centrifuge tube.
- Add 10 mL HPLC-grade water and vortex the tube vigorously.
- Add 10 mL of the ACN/IS Extraction Solvent.
- Allow the tube to sit for 15 min.
- Add 4 g anhydrous $MgSO_4$ and 1 g NaCl.
- Shake the tube vigorously on 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 Na_2SO_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.

Result and Discussion

Matrix Matched Calibration

The chromatogram in Figure 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.

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

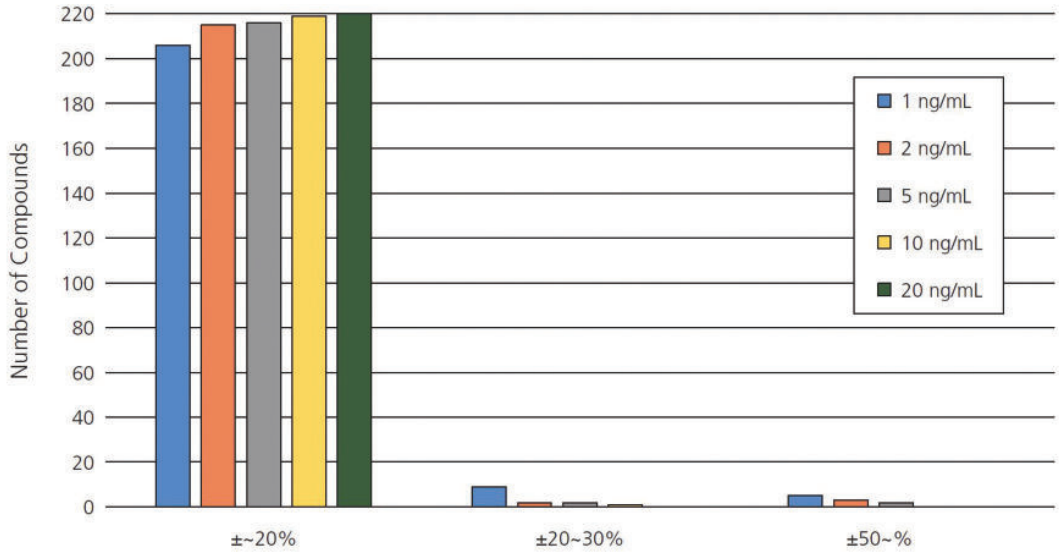


Column	Rxi-5ms, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13423) using Rxi guard column 5 m, 0.25 mm ID (cat.# 10029) with SilTite µ-Union connector kit (cat.# 23885)
Sample	GC Multiresidue pesticide kit (cat.# 32562)
Diluent:	Toluene
Conc.:	10 ng/mL
Injection	
Inj. Vol.:	2 µL pulsed splitless
Liner:	Topaz 3.5 mm ID single taper inlet liner w/wool (cat.# 23336)
Inj. Temp.:	250 °C
Pulse Pressure:	36 psi (248.2kPa)
Pulse Time:	1.5 min
Oven	
Oven Temp.:	90 °C (hold 1 min) to 130 °C at 30 °C/min to 330 °C at 10 °C/min (hold 2 min)
Carrier Gas	He, constant linear velocity
Linear Velocity:	55 cm/sec
Detector	Shimadzu GCMS TQ8040
Transfer Line Temp.:	290 °C
Analyzer Type:	Quadrupole
Source Temp.:	230 °C
Electron Energy:	70 eV
Ionization Mode:	EI
Instrument	Shimadzu GCMS-TQ8040
Notes	Matrix-matched calibration standards were prepared in ginseng. Calibration curves were generated by the internal standard method using PCB52 as the internal standard. An MRM analytical method was created using the Shimadzu pesticides database. Although the multiresidue pesticides kit mixes are formulated to ensure maximum long-term stability and reliability as packaged, stability may become an issue when a large number of compounds with different chemical functionalities are combined together into a single mix. This should be taken into consideration for quantitative analysis.
Acknowledgement	Chromatogram provided by Shimadzu. Publication 3655-11615-10ANS (C146-E334), First Edition, December 2016. Residual Pesticides Analysis of Botanical Ingredients Using Gas Chromatography Triple Quadrupole Mass Spectrometry. Riki Kitano, Tairo Ogura, Nicole Lock, Robert Clifford, Julie Kowalski, Jack Cochran, Dan Li

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

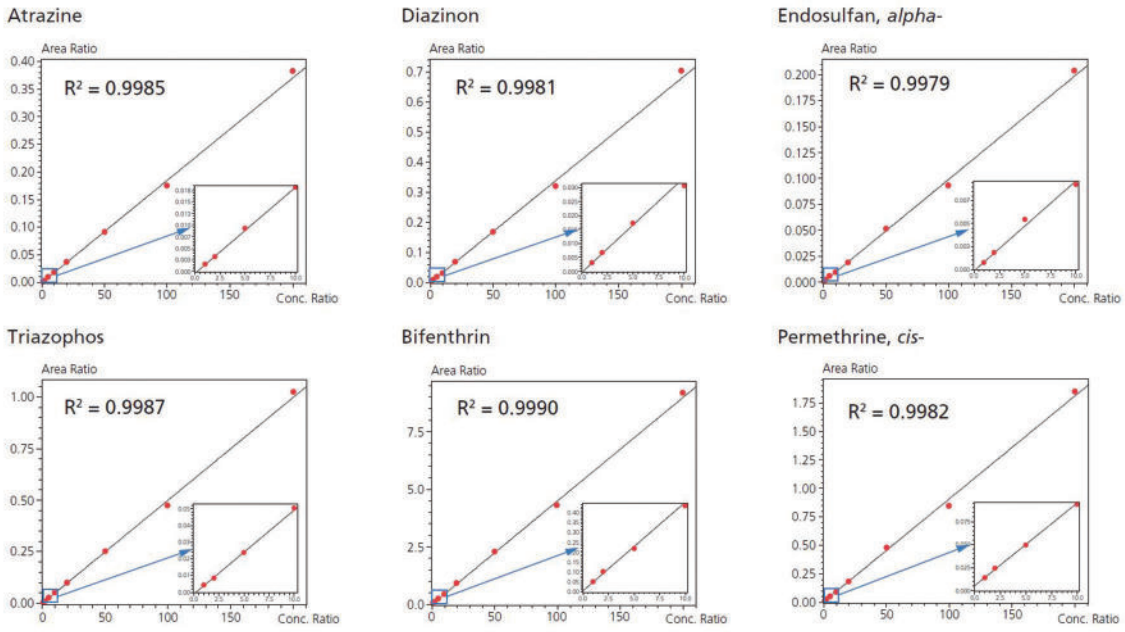
Back calculation was performed by calculating the concentration of each calibration point, and if the concentration exceeds ±20% of theoretical value, the calibration point would be interpolated with the nearest two points. Over 93% of the compounds with concentration of 1 ng/mL were within ±20% of theoretical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

Figure 3: Difference Between Back Calculation and Theoretical Concentration



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 (Figure 4) and all coefficients of determination (220 pesticides) were greater than 0.99.

Figure 4: Calibration Curves of Representative Six Compounds



Recovery of Fortified Samples

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 sample.

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 results 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 criterion is recommended.

Table I shows recovery and relative ion ratio for all compounds and Figure 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.

Table I: 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
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
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

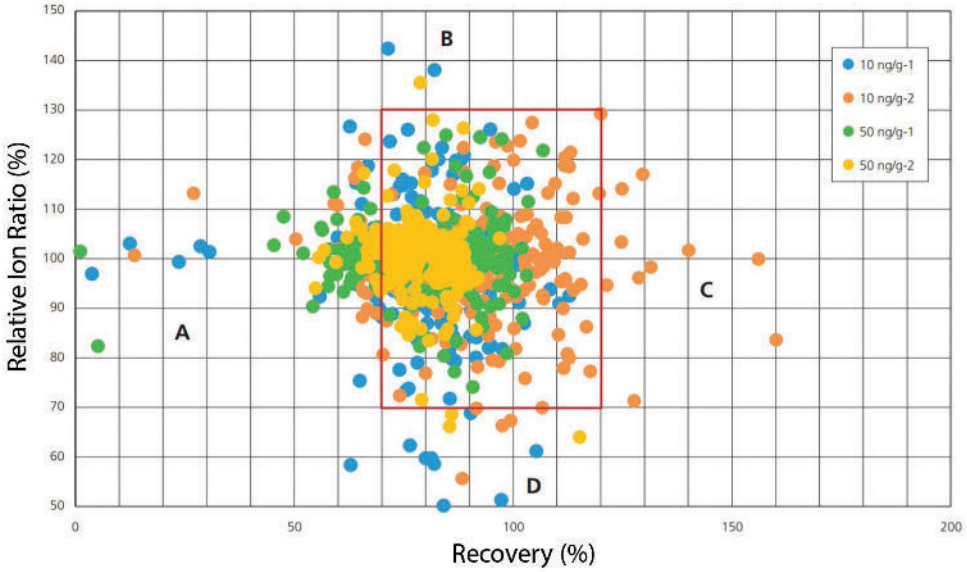
127	Isazofos	257.0 > 162.0	8	257.0 > 119.0	18	80.0	87.4	74.4	74.7	109.5	107.1	96.0	102.6
128	Isodrin	192.9 > 157.0	20	262.9 > 192.9	28	76.4	73.7	67.4	71.2	88.2	99.0	110.1	98.6
129	Isopropalin	280.1 > 238.1	8	280.1 > 133.1	18	76.5	88.1	73.6	71.4	89.8	82.7	97.9	106.1
130	Lenacil	153.1 > 136.1	14	153.1 > 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
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 (Figure 5) were classified to four groups.

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



Group A showed low recovery; this group consisted mainly of compounds which have a 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.

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 amounts 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.

References

[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

Topaz 3.5 mm ID Single Taper Inlet Liner w/ Wool for Shimadzu 17A, 2010, 2014, and 2030 GCs equipped with split/splitless inlets

ID x OD x Length	qty.	cat.#
Single Taper, Premium Deactivation, Borosilicate Glass with Quartz Wool 3.5 mm x 5.0 mm x 95 mm	5-pk.	23336

Pesticide Residue Cleanup SPE Cartridges

- Convenient, multiple adsorbent beds in a single cartridge.
- For use in multiresidue pesticide analysis to remove matrix interferences.
- Excellent for cleanup of dietary supplement extracts.

SPE Cartridge	qty.	cat.#
6 mL Combo SPE Cartridge Packed with 500 mg CarboPrep 90/500 mg Aminopropyl, Polyethylene Frits	30-pk.	26193
6 mL Combo SPE Cartridge Packed with 250 mg CarboPrep 90/500 mg PSA, Polyethylene Frits	30-pk.	26488
6 mL Combo SPE Cartridge Packed with 500 mg CarboPrep 90/500 mg PSA, Polyethylene Frits	30-pk.	26194
6 mL SPE Cartridge Packed with 500 mg PSA, Polyethylene Frits	30-pk.	26195
6 mL Combo SPE Cartridge Packed with 200 mg CarboPrep 200 and 400 mg PSA, PTFE Frits	30-pk.	26127
6 mL Combo SPE Cartridge Packed with 250 mg CarboPrep 200 and 500 mg PSA, PTFE Frits	30-pk.	26128
6 mL Combo SPE Cartridge Packed with 500 mg CarboPrep 200 and 500 mg PSA, PTFE Frits	30-pk.	26129

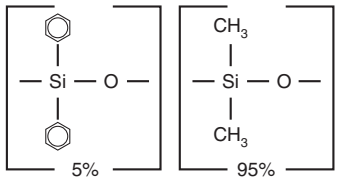
PSA–primary and secondary amine

Rxi-5ms Columns (fused silica)
low-polarity phase; Crossbond diphenyl dimethyl polysiloxane

- General-purpose columns for semivolatiles, phenols, amines, residual solvents, drugs of abuse, pesticides, PCB congeners (e.g., Aroclor mixes), solvent impurities.
- Most inert column on the market.
- Tested and guaranteed for ultra-low bleed; improved signal-to-noise ratio for better sensitivity and mass spectral integrity.
- Temperature range: -60 °C to 330/350 °C.
- Equivalent to USP G27 and G36 phases.

similar **phases**

HP-5ms Semivolatiles, HP-5ms, HP-5msUI, DB-5, Ultra-2, CP-Sil 8 CB, ZB-5, ZB-5msi



Description	temp. limits	qty.	cat.#
Rxi-5ms 30 m, 0.25 mm ID, 0.25 µm	-60 to 330/350 °C	ea.	13423
Rxi-5ms 30 m, 0.25 mm ID, 0.25 µm	-60 to 330/350 °C	6-pk.	13423-600

Rxi Guard/Retention Gap Columns (fused silica)

- Extend column lifetime.
- Excellent inertness—obtain lower detection limits for active compounds.
- Sharper chromatographic peaks by utilizing retention gap technology.
- Maximum temperature: 360 °C.

Nominal ID	Nominal OD	5-Meter cat.#	5-Meter/6-pk. cat.#	10-Meter cat.#	10-Meter/6-pk. cat.#
0.25 mm	0.37 ± 0.04 mm	10029	10029-600	10059	10059-600
0.32 mm	0.45 ± 0.04 mm	10039	10039-600	10064	10064-600
0.53 mm	0.69 ± 0.05 mm	10054	10054-600	10073	10073-600

SilTite µ-Union Connectors

- Reliably create permanent connections between fused silica analytical columns, guard columns, and retention gaps.
- SilTite FingerTite technology provides easy installation and a permanent leak-tight connection.
- Deactivated metal and zero-dead-volume design ensure optimal peak shapes.
- Robust connection is stable through extreme temperature and pressure cycling, making it ideal for use with mass spectrometers.
- Kits contain two SilTite µ-Union connectors, five double-taper ferrules, and installation tools.

Press-fit style connectors work well for many applications, but the connector seal can be broken by extreme temperature and pressure cycling. In these situations, SilTite µ-Unions are a better alternative as they create a permanent connection between analytical columns, guard columns, and retention gaps. SilTite FingerTite technology provides easy installation and a reliable, leak-tight connection. Data quality can be improved by using these connectors because their zero-dead-volume design and deactivated metal construction ensure optimal peak shapes. Robust SilTite µ-Unions are recommended for mass spectrometry work and any application with extreme temperature and pressure changes. Connector kits are available in six configurations that are designed to securely connect columns of either the same or different inner diameters. Each kit contains two SilTite µ-Union connectors, five double-taper ferrules, and installation tools.

When connecting columns of different inner diameters (ID), always connect the column with the largest ID first. The ferrules are very small, making it difficult to see the size difference between the two ferrule orifices and making it easy to install the smaller ID column into the wrong orifice. Since the larger ID column (e.g., 0.32 mm) will not fit into the smaller ferrule orifice (e.g., 0.25 mm), installing the larger column first ensures the correct end of the ferrule will be used for both columns.

Description	Fits Column ID	qty.	cat.#
SilTite µ-Union Connector Kit	0.32 mm to 0.32 mm	kit	23882
SilTite µ-Union Connector Kit	0.32 mm to 0.53 mm	kit	23883
SilTite µ-Union Connector Kit	0.53 mm to 0.53 mm	kit	23884
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.18/0.25 mm	kit	23885
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.32 mm	kit	23886
SilTite µ-Union Connector Kit	0.18/0.25 mm to 0.53 mm	kit	23887

Empty Centrifuge Tubes, Polypropylene

Description	qty.	cat.#
Empty 50 mL Centrifuge Tube, Polypropylene w/Blue Cap	50-pk.	25846

GC Multiresidue Pesticide Kit

- Accurately identify and quantify pesticide residues by GC-MS/MS in fruits, vegetables, botanicals, and herbals such as tea, ginseng, ginger, echinacea, and dietary supplements.
- Comprehensive 203-compound kit covers food safety lists by the FDA, USDA, and other global governmental agencies; individual ampuls also sold separately.

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

Description	# of Components	Type	Conc. in Solvent and Volume	qty.	cat.#
GC Multiresidue Pesticide Kit			Contains 1 mL each of these mixtures.	kit	32562
GC Multiresidue Pesticide Standard #1	16	Organophosphorus Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32563
GC Multiresidue Pesticide Standard #2	40	Organochlorine Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32564
GC Multiresidue Pesticide Standard #3	25	Organonitrogen Compounds	100 µg/mL each in toluene:acetonitrile (99:1), 1 mL/ampul	ea.	32565
GC Multiresidue Pesticide Standard #4	28	Organonitrogen Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32566
GC Multiresidue Pesticide Standard #5	34	Organonitrogen Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32567
GC Multiresidue Pesticide Standard #6	18	Synthetic Pyrethroid Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32568
GC Multiresidue Pesticide Standard #7	10	Herbicide Methyl Esters	100 µg/mL each in toluene, 1 mL/ampul	ea.	32569
GC Multiresidue Pesticide Standard #8	24	Organophosphorus Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32570
GC Multiresidue Pesticide Standard #9	8	Organophosphorus Compounds	100 µg/mL each in toluene, 1 mL/ampul	ea.	32571

QuEChERS Standards

Pesticide analysis is fast and simple using QuEChERS methods. Use these cost-effective QuEChERS standards for even greater lab efficiency. Standards are compatible with all major methods, including mini-multiresidue, AOAC, and European procedures. Save time with convenient mixes or make your own blend using our full line of single-component solutions.

QuEChERS Internal Standard Mix for GC-MS Analysis (6 components)

Ready to use for QuEChERS extractions—no dilutions necessary.

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

PCB 18 (37680-65-2), 50 µg/mL	Triphenylphosphate (115-86-6), 20 µg/mL
PCB 28 (7012-37-5), 50 µg/mL	Tris(1,3-dichloroisopropyl)phosphate (13674-87-8), 50 µg/mL
PCB 52 (35693-99-3), 50 µg/mL	
Triphenylmethane (519-73-3), 10 µg/mL	
In acetonitrile, 5 mL/ampul	cat.# 33267 (ea.)

SV Internal Standard Mix (6 components)

- High purity for consistent results.
- Free quality assurance data package available online.
- Highest concentrations commercially available.

Certified reference materials (CRMs) manufactured and QC-tested in ISO-accredited labs satisfy your ISO requirements.

Acenaphthene-d10 (15067-26-2)	Naphthalene-d8 (1146-65-2)
Chrysene-d12 (1719-03-5)	Perylene-d12 (1520-96-3)
1,4-Dichlorobenzene-d4 (3855-82-1)	Phenanthrene-d10 (1517-22-2)
2,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31206 (ea.)
2,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31206.15 (15-pk.)
2,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31206.25 (25-pk.)
4,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31006 (ea.)
4,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31006.15 (15-pk.)
4,000 µg/mL each in methylene chloride, 1 mL/ampul	cat.# 31006.25 (25-pk.)

Q-sep QuEChERS Extraction Salts

- Free-flowing salts transfer easily and completely.
- Easy-open packets eliminate the need for a second empty tube for salt transfer.
- Convenient slim packets fit perfectly into tubes to prevent spills.
- Ready-to-use tubes, no glassware required.
- Pre-weighed, ultra-pure extraction salts.
- Ideal for original unbuffered, AOAC (2007.01), and European (EN 15662) QuEChERS methods.

QuEChERS methods are fast, easy, and cost-effective, and Restek Q-sep products make QuEChERS procedures even easier. No specialized glassware is required when you're using Q-sep extraction packets and tubes. Free-flowing extraction salts and salt packets that fit easily into the extraction tubes make transferring the salts to your sample mess-free and easy.

Description	Method	Material	qty.	cat.#
Q-sep QuEChERS Extraction Kit	original unbuffered	4 g $MgSO_4$, 1 g NaCl with 50 mL Centrifuge Tube	50 packets & 50 tubes	25848
Q-sep QuEChERS Extraction Salt Packets Only	original unbuffered	4 g $MgSO_4$, 1 g NaCl	50 packets	25847
Q-sep QuEChERS Extraction Kit	European EN 15662	4 g $MgSO_4$, 1 g NaCl, 1 g TSCD, 0.5 g DHS with 50 mL Centrifuge Tube	50 packets & 50 tubes	25850
Q-sep QuEChERS Extraction Salt Packets Only	European EN 15662	4 g $MgSO_4$, 1 g NaCl, 1 g TSCD, 0.5 g DHS	50 packets	25849
Q-sep QuEChERS Extraction Kit	AOAC 2007.01	6 g $MgSO_4$, 1.5 g NaOAc with 50 mL Centrifuge Tube	50 packets & 50 tubes	25852
Q-sep QuEChERS Extraction Salt Packets Only	AOAC 2007.01	6 g $MgSO_4$, 1.5 g NaOAc	50 packets	25851
Empty 50 mL Centrifuge Tube, Polypropylene w/Blue Cap			50-pk.	25846

TSCD – trisodium citrate dihydrate
DHS – disodium hydrogen citrate sesquihydrate
NaOAc – sodium acetate

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