

# QuEChERS Combined with an Agilent 7000 Series Triple Quadrupole GC/MS System for the Analysis of Over 200 Pesticide Residues in Leek and Garlic

## Application Note

Food

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### Abstract

This application note describes a simple and high-throughput method for the analysis of 213 pesticides in leek and garlic using an Agilent QuEChERS kit combined with Agilent gas chromatography-triple quadrupole mass spectrometry (GC-MS/MS). A matrix-matched standard calibration method was used to avoid quantitation bias from matrix interference. It shows that the method has a linearity more than two orders of magnitude (2–400 µg/kg), with linear regression coefficients ( $R^2$ ) of 0.99 or above for the majority of pesticides. The limits of quantification (LOQs) ranged between 2 and 10 µg/kg, and the majority of the pesticides had an LOQ of 2 µg/kg, which is below the regulatory maximum residue limits. A spiking test showed that most recoveries at 2, 5, 10, 20, 50, 100, and 200 µg/kg were in the range of 80–120% ( $n = 6$ ) with associated RSDs below 20%. Leek and garlic samples were analyzed for method application. This method can be applied for routine analysis of these pesticide residues in leek and garlic.



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## Introduction

Leek and garlic are important vegetables in China. China is by far the largest producer of garlic, producing two thirds of the world's total with 409 million cwt in 2012, and ranked fifth in leek production, with 127,308 tons, in 2010. The protective effect of garlic on the development of cancer has been reported in *in vitro* and *in vivo* experimental studies. Leeks are reported to have similar anticancer medical value [1].

Leek and garlic are known as troublesome matrices in pesticide analysis. They each contain large amounts of sulfur-containing compounds that may cause significant interferences, especially when using a single mass spectrometry (MS) detector. To overcome this problem, some sample preparation methods, such as supercritical fluid extraction and microwave heating combined with AgNO<sub>3</sub>-loaded solid-phase extraction (SPE) column cleanup or gel permeation chromatography [2], have been reported to remove interferences from sulfur compounds. However, these methods require a large amount of solvent and time-consuming operation steps.

The quick, easy, cheap, effective, rugged, and safe (QuEChERS) method for pesticide multiresidue analysis has been accepted worldwide since it was first introduced by Anastassiades, *et al.* [3] in 2003. Most of the applications were dedicated to pesticide multiresidue analysis in food matrices. This topic has been thoroughly reviewed in recent years by the researchers.

Previously, several studies have reported multiresidue pesticide analysis based on the QuEChERS procedure combined with GC-MS/MS detection for fruit and vegetables including leek and garlic [1,4,5]. However, there are over 200 pesticides not analyzed in these reports. This application note describes a recently published study of QuEChERS combined with GC-MS/MS for the analysis of 213 pesticide residues in leek and garlic [6]. It is a rapid multiresidue method based on QuEChERS sample preparation, combined with GC-MS/MS detection, attempting to detect more than 200 pesticide targets in leek and garlic.

## Materials and Methods

Acetonitrile and ethyl acetate of HPLC grade; QuEChERS extraction salt packets with ceramic homogenizer (p/n 5982-5650CH), QuEChERS dispersive SPE kit (p/n 5982-5056 for garlic and 5982-5256 for leek).

The standard pesticides were bought from Chemservice (West Chester, PA, USA) and Dr. Ehrenstorfer (Ausberg, Germany). Stock standard solution of 5 µg/mL (mixture of pesticides divided into two groups), and internal standard solution (Heptachlor epoxide B, 1 µg/mL) were prepared in ethyl acetate, and stored at -20 °C until use.

## Instrument conditions

### GC conditions

GC system	Agilent 7890A, coupled with an Agilent 7693 autosampler
Column	Agilent VF-1701ms, 30 m × 0.25 mm, 0.25 µm (p/n CP9151)
Oven temperature	40 °C hold 1 minute, at 40 °C/min to 120 °C, at 5 °C/min to 240 °C, at 12 °C/min to 300 °C, hold 6 minutes
Carrier gas	Helium
Flow rate	1.0 mL/min
Injection port temperature	280 °C
Injection volume	1.0 µL
Injection mode	Splitless, purge on after 1.5 minutes

### MS conditions

MS system	Agilent 7000B Triple Quadrupole GC/MS System*
Ion source	EI
Ionization voltage	70 eV
Ion source temperature	280 °C
Quadrupole temperature	Q1 150 °C, Q2 150 °C
Interface temperature	280 °C
Solvent delay	3.0 minutes

The specific MRM transitions for all the test pesticides and other parameters are given in the appendix.

\* The Agilent 7000C and 7010 GC-MS/MS are available, and have demonstrated better results.

## Sample preparation

Weigh 10 g of minced sample into a 50-mL centrifuge tube. Add 10 mL of acetonitrile and a ceramic homogenizer, followed by the QuEChERS extraction salt packet. Immediately, seal the tube and shake vigorously by hand for one minute. Centrifuge the tube for five minutes at 4,000 rpm.

Transfer 6 mL of the supernatant into a 15-mL QuEChERS dispersive centrifuge tube. Vortex the extract with the sorbent for one minute, then centrifuge the tube for five minutes at 4,000 rpm.

Transfer 3 mL of the supernatant into a 10-mL glass centrifuge tube, and add 75  $\mu$ L of internal standard solution. Evaporate the supernatant to dryness under a stream of nitrogen in a 40 °C water bath.

Redissolve the residue in 1.5 mL of ethyl acetate, and filter through a PTEE filter (0.22  $\mu$ m) for GC-MS/MS analysis.

## Method validation

A recovery study was carried out to determine the method accuracy and precision. For each blank matrix, seven levels 2, 5, 10, 20, 50, 100, and 200  $\mu$ g/kg were fortified. After fortification, the spiked samples were left at room temperature for 30 minutes prior to extraction. To avoid quantitative errors, matrix-matched calibration standards were used to calculate the analyte recoveries. Solvent-based standards were also analyzed to assess the matrix effects. The limits of quantification (LOQs) for each pesticide was based on the recovery results, and defined as the lowest validated spike level meeting the requirement of recovery and relative standard deviation (RSD) for different fortification levels.

## Results and Discussion

### Matrix effect

The matrix effect (ME) is regarded as a signal suppression or enhancement of the analyte due to the coelution of matrix components. It is reported that signal enhancement is always observed in GC-MS/MS analysis. The enhancement appears because matrix components block active sites (silanols, metal ions, and so forth) present in the column or inlet. Suppression or enhancement can vary considerably from matrix to matrix

and differ substantially in pure solvent and matrix. Matrix effects also depend heavily on the chemical properties of the analyte and sample preparation procedure. Therefore, it is essential to take into account the matrix effects. The matrix effect is calculated by the equation:

$$ME (\%) = \left( \frac{m_{matrix} - m_{solvent}}{m_{solvent}} - 1 \right) \times 100$$

Where *ME* is matrix effect, and  $m_{matrix}$  and  $m_{solvent}$  are the slopes of the calibration curves obtained in the matrix and solvent, respectively.

Soft matrix effects (suppression or enhancement of 0–20%) are negligible. However, if the pesticides suffer medium (suppression or enhancement of 20–50%) or strong (suppression or enhancement >50%) matrix effects, it is necessary to use certain methods to overcome the influence of the matrix.

It is indicated that there was no statistical difference between leek and garlic in respect to ME. Most of the pesticides exhibited matrix enhancement effects. Approximately 37% of the pesticides exhibited soft matrix effects, 30% exhibited medium matrix effects, and 32% exhibited strong matrix effects. The results proved that the matrix-matched calibration standards were indispensable for accurate quantification by GC-MS/MS.

### Linearity and LOQs

Due to the matrix effect, matrix-matched standard was used for quantification. Linearity was determined in all matrices, and the linear range was between 2 and 400  $\mu$ g/kg (internal standard method was applied with concentration of 50  $\mu$ g/L). In the two matrices, the correlation coefficient of detection ( $R^2$ ) for all of the pesticides was equal to or higher than 0.99, which would guarantee accurate quantification. There were 213 pesticides spiked at 2, 5, 10, 20, 50, 100, and 200,  $\mu$ g/kg ( $n = 6$ ) in leek and garlic matrix for recovery and RSD analysis. The LOQs for the pesticides were determined based on the recovery and RSD results, and defined as the selected lowest validated spike level meeting the requirement of recovery and RSD for different fortification levels, as described in Document No. SANCO/12495/2013 [7]. The LOQs for the 213 pesticides ranged between 2 and 10  $\mu$ g/kg. In leek, 196 out of 213 pesticides had LOQs at 2  $\mu$ g/kg, and in garlic, 176 out of 213 pesticides had LOQs of 2  $\mu$ g/kg.

## Recovery and precision

The proposed modified QuEChERS method was evaluated for 213 pesticides in leek and garlic. A recovery study was carried out to determine the method's accuracy by comparing the real concentration of each pesticide measured. This was accomplished by performing the complete procedure with a known pesticide concentration initially fortified to the blank matrix at seven levels 2, 5, 10, 20, 50, 100, and 200  $\mu\text{g}/\text{kg}$  at replicates  $n = 6$ . For recovery experiments, most of the 213 pesticides were in the range of 80–120% with the RSD below 20%. Figure 1 shows the recoveries of all pesticides in leek and garlic at 10 and 100  $\mu\text{g}/\text{kg}$ .

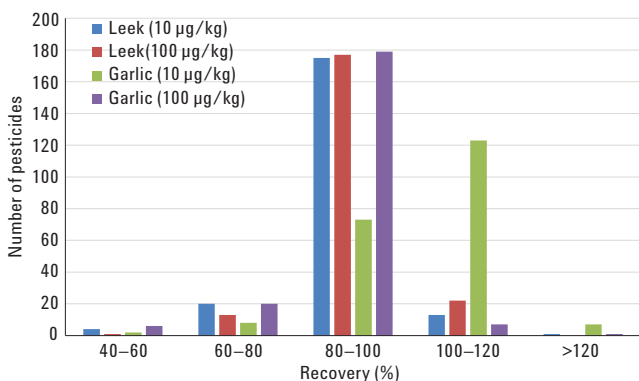


Figure 1. The distribution of recoveries in leek and garlic at 10 and 100  $\mu\text{g}/\text{kg}$ .

## Real sample analysis

To prove the effectiveness of the proposed method, eight leek and garlic samples from three local markets were analyzed according to the method described above. No pesticide was detected in the garlic samples, whereas 17 pesticides were detected in the eight leek samples. Table 1 shows the maximum residue limit (MRL) of the detected pesticides, and the concentrations of leek samples according to China, Japan, the US Environmental Protection Agency (EPA), and the European Union (EU). All the pesticides detected were insecticides and fungicides. The detection frequency for chlorpyrifos, procymidone, and triazophos was very high. The concentrations of chlorpyrifos, procymidone, pyrimethanil, and triazophos in some samples exceeded the linearity range, and up to 3.33, 2.66, 1.73, and 4.27  $\text{mg}/\text{kg}$  were detected, respectively. The residue levels of chlorpyrifos (sample 5) and procymidone (samples 1 and 4) exceeded the MRLs in China. It should be noted that the following government-banned pesticides, isazofos, phorate, and phorate sulfone (the metabolite of phorate) were detected in several samples.

Table 1. Pesticide Concentrations (mg/kg) of Real Leek Samples from Local Markets

Pesticide	MRL(mg/kg)				Leek samples (mg/kg)							
	China	Japan	EPA	EU	1	2	3	4	5	6	7	8
Bifenthrin	–	0.5	0.05	0.05	ND	ND	ND	0.00815	ND	ND	ND	ND
Chlorpyrifos	0.1	0.2	0.1	0.5	< LOQ	0.0158	0.0139	0.0120	3.33 <sup>a</sup>	0.0237	0.0192	<LOQ
Cypermethrin	0.05	5	6	0.5	ND	0.353	0.272	ND	0.0275	ND	ND	ND
Cyprodinil	–	4	4	0.05	0.0426	0.00222	0.00635	ND	ND	ND	ND	ND
Difenoconazol	–	6	6	0.5	0.0185	0.00251	0.00399	ND	ND	ND	LOQ	ND
Fipronil	0.02	0.002	–	0.01	ND	ND	ND	0.0131	ND	ND	ND	ND
Isazofos	P	B	B	B	0.00204	<LOQ	ND	ND	ND	<LOQ	ND	ND
Metalaxyl	–	0.2	10	0.2	<LOQ	ND	ND	0.00576	ND	ND	ND	ND
Myclobutanil	–	1	–	0.02	ND	ND	ND	ND	0.110	ND	<LOQ	ND
Phorate	P	0.3	–	B	0.00317	0.0153	0.00267	ND	ND	ND	ND	ND
Phorate sulfone	–	–	–	–	0.120	0.0158	0.0151	ND	ND	ND	ND	ND
Procymidone	0.2	5	–	0.01	0.456 <sup>a</sup>	0.0358	0.0615	2.66 <sup>a</sup>	0.0460	0.00623	ND	ND
Profenofos	–	0.05	–	B	ND	0.136	0.254	0.00299	ND	ND	ND	ND
Pyrimethanil	–	3	3	1	1.74 <sup>a</sup>	0.737 <sup>a</sup>	1.09 <sup>a</sup>	0.0130	ND	ND	ND	ND
Triadimefon	–	0.1	–	0.1	ND	ND	ND	ND	0.0201	ND	ND	ND
Triadimenol	–	0.2	–	0.1	<LOQ	<LOQ	ND	ND	0.0644	ND	<LOQ	ND
Triazophos	–	–	–	B	4.27 <sup>a</sup>	0.154	0.447 <sup>a</sup>	0.00562	<LOQ	<LOQ	ND	ND

<sup>a</sup> These samples were diluted to make sure that the concentrations were within the linearity range.

ND = not detected

B = banned pesticide

P =prohibited to use in fruit and vegetable

## Comparison of the proposed method to other works

The presence of pesticide residues in bulb vegetables has been reported in some works recently. This proposed method shows the following advantages over previous methods:

- Over 200 pesticides were discovered in leek and garlic, whereas other published works found many fewer pesticides.
- In the extraction procedure, a ceramic homogenizer was used to improve extraction efficiency and repeatability.
- Seven spiking levels were carried out to ensure reliable method validation.
- Less extraction time and a simpler extraction procedure were needed for sample extraction.
- Very low LOQs (2 µg/kg) were obtained for the majority of pesticides.

## Conclusions

The proposed method was successful and reliable for the multiresidue analysis of 213 pesticides in leek and garlic with satisfactory recovery, precision, and accuracy, demonstrating the suitability of the method for the routine analysis of pesticide residue in leek and garlic. Compared to other works relating to pesticide residue analysis in leek and other bulb vegetables, this method presents many advantages in respect to the target number, LOQ, sample extraction procedure, and method validation.

## References

1. L. J. Qu, *et al.* "Rapid determination of organophosphorous pesticides in leeks by gas chromatography–triple quadrupole mass spectrometry" *Food Chem.* **122(1)**, 327-332 (2010).
2. S. Shuling, M. Xiaodong, L. Chongjiu. "Multi-residue determination method of pesticides in leek by gel permeation chromatography and solid phase extraction followed by gas chromatography with mass spectrometric detector" *Food Control* **18(5)**, 448-453 (2007).
3. M. Anastassiades, *et al.* "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(2)**, 412-431 (2003).
4. N. Chamkasem, *et al.* "Analysis of 136 Pesticides in Avocado Using a Modified QuEChERS Method with LC-MS/MS and GC-MS/MS" *J. Agr. Food Chem.* **61(10)**, 2315-2329 (2013).
5. D. Lu, *et al.* "A fast and easy GC-MS/MS method for simultaneous analysis of 73 pesticides residues in vegetables and fruits" *Anal. Methods-UK* **5(5)**, 1721-4732 (2013).
6. Z. He, *et al.* "Multiresidue analysis of 213 pesticides in leek and garlic using QuEChERS-based method and gas chromatography-triple quadrupole mass spectrometry" *Anal. Bioanal. Chem.* **407**, 2637-2643 (2015).
7. E. C. DG-SANCO. "Method validation and quality control procedures for pesticides residues analysis in food and feed" In, vol. SANCO/12571/2013 (2014).

## Appendix

### Acquisition and chromatographic parameters for the selected pesticides

Pesticides	t <sub>R</sub> (min)	MRM1	CE1(V)	MRM2	CE2(V)
<b>Group A</b>					
Dichlorvos	8.52	109→79	5	184.9→93	10
Disulfoton sulfoxide	9.09	153→96.9	10	213→97	20
Methamidophos	10.12	141→95	5	95→79	10
Dichlorobenzonitrile(2,6- (Dichlobenil) )	10.71	171→100	25	171→136.1	15
Mevinphos	12.46	127→109	10	127→95	15
Methacrifos	12.8	207.9→180.1	5	207.9→93	10
Molinate	13.33	126.2→55.1	10	126.2→83.1	5
Cycloate	14.89	154.1→83.1	5	83→55.1	5
Isoproc carb	14.94	121→77.1	20	136→121.1	10
Acephate	15.32	142→96	5	136→94	10
Hexachlorobenzene	15.51	283.8→213.9	30	283.8→248.8	15
Ethoprophos	15.88	157.9→114	5	157.9→97	15
Ethalfuralin	16.21	275.9→202.1	15	315.9→275.9	10
Chlordimeform	16.42	151.9→117.1	10	195.9→181	5
Propoxur	16.97	110→63	25	110→64	15
Sulfotep	16.97	237.8→145.9	10	201.8→145.9	10
BHC- <i>alpha</i>	17.55	217→181	5	218.9→183	5
Atrazine-desethyl	18.4	172→94	15	187→172	5
Terbufos	18.46	230.9→175	10	230.9→129	20
Triallate	18.69	268→184.1	20	142.9→83	15
Profluralin	18.72	317.9→199	15	317.9→54.8	10
Tebupirimfos	19.06	233.9→110.1	15	260.8→137.2	15
Dioxathion	19.19	152.9→96.9	10	271→96.9	30
Propazine	19.4	214.2→172.2	10	229.1→58.1	10

Pesticides	t <sub>R</sub> (min)	MRM1	CE1(V)	MRM2	CE2(V)
Dicloran	19.59	206.1→176	10	160.1→124.1	10
Propetamphos	19.7	138→110	10	138→64	15
Iprobenfos	20.21	203.9→91	5	121.9→121	15
Dichlofenthion	20.35	278.9→222.9	15	222.9→204.9	15
Pirimicarb	20.39	238→166.2	10	166→55.1	20
Dimethoate	20.82	86.9→46	15	142.9→111	10
Monocrotophos	20.87	127.1→109	10	127.1→95	15
Acetochlor	21.21	174→146.1	10	222.9→147.2	5
Alachlor	21.59	188.1→160.2	10	160→132.1	10
Pirimiphos-methyl	21.82	290→125	20	232.9→151	5
Paraoxon-methyl	21.88	229.9→136.1	5	229.9→106.1	15
Vinclozolin	22.04	187→124	20	197.9→145	15
Metribuzin	22.23	198→82	15	198→55	30
Metalaxyl	22.3	234→146.1	20	220→192.1	5
Thiobencarb	22.57	100→72	5	124.9→89	15
Metolachlor	22.95	238→162.2	10	162.2→133.2	15
Formothion	22.97	170→93	5	197.9→92.9	10
Bromophos	23.38	330.8→315.8	15	328.8→313.8	15
Fenthion	23.5	278→169	15	278→109	15
Paraoxon	23.56	148.9→119	5	108.9→81	10
Triadimefon	24.11	208→181.1	5	208→111	20
Parathion	24.25	290.9→109	10	138.9→109	5
Isofenphos-methyl	24.34	199→121	10	241.1→199.1	10
Isofenphos	24.68	212.9→121.1	10	212.9→185.1	5
Quinalphos	24.89	146→118	10	146→91	30
Penconazole	25.15	248→192.1	15	248→157.1	25
Phorate Sulfone	25.46	153→97	10	124.9→96.9	5
Fosthiazate	25.58	195→103	5	195→60	20
DDE-p,p'	25.73	246.1→176.2	30	315.8→246	15
Fenothiocarb	25.86	160.1→72.1	10	72→56	10
Terbufos sulfone	26.12	198.9→143	10	152.9→96.9	10
DEF (Tribufos)	26.17	202→147	5	169→57.1	5
Mepanipirim	26.18	223.2→222.2	10	222.2→207.2	15
Bromacil	26.31	205→188	15	207→190	15
triadimenol	26.39	168→70	10	128→65	25
Bromfenvinfos	26.45	266.9→159.1	15	268.9→161.1	15
Pretilachlor	26.57	262→202	5	162.1→132.2	20
DDD-o,p'	26.78	235→165.2	20	237→165.2	20
Ditalimfos	26.88	130→102.1	10	148→130.1	10
Kresoxim-methyl	26.89	116→89	15	116→63	30
Oxadiazon	26.94	174.9→112	15	174.9→76	35
DDT-o,p'	27.36	235→165.2	20	237→165.2	20
Cyflufenamid	27.62	188.1→88	35	118.1→89	25
Mephosfolan	27.69	196→139.9	15	196→59.9	30
Bupirimate	27.85	272.9→193.1	5	272.9→108	15

<b>Pesticides</b>	<b>t<sub>R</sub>(min)</b>	<b>MRM1</b>	<b>CE1(V)</b>	<b>MRM2</b>	<b>CE2(V)</b>
Oxyfluorfen	28.08	252→196	20	252→146	30
Chlorthiophos	28.21	324.8→268.9	10	296.8→268.9	5
Flutolanil	28.21	173→145.1	15	280.9→173	10
DDD-p,p'	28.41	234.9→165.1	20	236.9→165.2	20
Carbophenothion	28.45	153→96.9	10	199→143	10
Quinoxifen	28.76	237→208.1	30	271.9→237.1	10
Aclonifen	28.85	212.1→182.2	10	264.1→194.2	15
Trifloxystrobin	28.88	116→89	15	116→63	30
Piperonyl butoxide	29.18	176.1→103.1	25	176.1→131.1	15
Edifenphos	29.53	172.9→109	5	201→109	10
Fensulfothion	29.6	140→125	10	291.8→156	15
Triazophos	29.71	161.2→134.2	5	161.2→106.1	10
Bifenthrin	29.76	181.2→165.2	25	181.2→166.2	10
Tebufenpyrad	30.28	275.9→171.1	10	332.9→171	15
Bromopropylate	30.46	183→155	15	185→157	15
Epoxiconazole	30.5	192→138.1	10	192→111	25
Tetramethrin	30.63	164→107.1	10	164→77.1	25
Tebuconazole	30.64	250→125	20	125→89	15
Pyriproxyfen	30.95	136.1→96	15	136.1→78.1	20
Piperophos	30.99	320→122	10	140→98.1	10
EPN	31.07	169→141.1	5	169→77.1	25
Hexazinone	31.08	171→71.1	10	171→85.1	10
Fenamidone	31.17	238→237.2	10	268→180.2	20
Tetradifon	31.47	226.9→199	15	158.9→131	10
Anilofos	31.54	225.9→184	5	225.9→157	10
pyrazophos	32.09	221→193.1	10	232→204.1	10
Fenarimol	32.2	251→139.1	10	219→107.1	10
Permethrin	32.2	183.1→168.1	10	183.1→153	10
Pyridaben	32.53	147.2→117.1	20	147.2→132.2	10
Cypermethrin	33.88	163→127	5	163→91	10
Boscalid	34.6	140→112	10	140→76	25
Fenvalerate	35.17	167→125.1	5	224.9→119	15
Deltamethrin	36.75	252.9→93	15	181→152.1	25
<b>Group B</b>					
Ethiolate	6.82	100→72	5	161→72	15
Naled	8.41	144.9→109	15	108.9→79	5
Biphenyl	9.88	154.1→153.1	15	153.1→152.1	15
Etridiazole	11.26	211.1→183	10	183→140	15
Chloroneb	13.19	206→191.1	10	208→193.1	10
Tecnazene (TCNB)	14.52	260.9→203	10	214.9→179	10
Thionazin	15.53	143→79	10	175→79	10



Pesticides	t <sub>R</sub> (min)	MRM1	CE1(V)	MRM2	CE2(V)
Diphenylamine	16.08	169→168.2	15	168→167.2	15
Fenobucarb	16.26	121→77	20	121→103.1	15
Benfluralin	16.65	292→264	5	292→206	10
phorate	16.98	260→75	5	230.9→128.9	25
Chlorpropham	17.26	153→125.1	10	153→90	25
Pentachloronitrobenzene	17.69	236.9→118.9	25	236.9→142.9	30
Omethoate	18.2	155.9→110	5	109.9→79	15
Atraton	18.44	211→169.1	5	169→154.1	5
Diazinon	18.5	137.1→84	10	137.1→54	20
Clomazone	18.65	204.1→107.1	20	125→89	15
Dicrotofos	18.72	127→109	15	127→95	15
pyrimethanil	19	198→183	15	198→118	35
BHC- <i>gamma</i>	19.19	217→181.1	5	181→145	15
Carbofuran	19.38	164.2→149.1	10	149.1→121.1	5
Etrimfos	19.39	181→153.1	5	168→153.1	5
Atrazine	19.52	214.9→58.1	10	214.9→200.2	5
Simazine	19.62	201.1→173.1	5	201.1→186.2	5
Terbuthylazine	19.78	228.9→173.1	5	172.9→172	5
Monolinuron	20	214→61	10	155→127	10
Isazofos	20.27	161→119.1	5	161→146	5
Pentachloroaniline	20.38	262.8→192	20	264.9→194	20
Pronamide	20.44	173→145	15	175→147	15
Chlorpyrifos-methyl	20.82	285.9→92.9	20	287.9→92.9	20
Aldrin	21.17	262.9→192.9	35	254.9→220	20
Ronnel (Fenchlorphos)	21.32	285→269.9	15	286.9→272	15
Desmetryn	21.39	213→58.1	10	213→171.2	5
Tolclofos-methyl	21.44	265→250	15	265→93	25
Prometryn	21.88	226→184.2	10	199→184.1	5
BHC-beta	22.1	217→181.1	5	181→145	15
Chlorpyrifos	22.36	198.9→171	15	196.9→169	15
Ametryn	22.37	227→170.1	10	227→58.1	10
Terbutryn	22.37	241.1→170.2	15	185→170.1	5
Trichloronat	22.71	296.8→268.9	10	298.8→270.9	10
Dipropetryn	22.79	255.1→222.1	10	255.1→180.1	20
BHC-delta	22.99	217→181.1	5	181→145	15
Pirimiphos-ethyl	23.1	318.1→166.1	10	318.1→182	10
Phosphamidon	23.13	127→95	15	127→109	10
Malathion	23.3	172.9→99	15	157.8→125	5
Fenitrothion	23.5	277→260	5	277.1→109	15
Methoprene	23.6	153→111.1	5	111.1→55	15
Ethofumesate	23.76	206.9→161.1	5	161→105.1	10
Cyprodinil	23.78	225.2→224.3	10	224.2→208.2	20
Isofenphos oxon	24.1	229→200.9	10	229→121	25
Pendimethalin	24.15	251.8→162.2	10	251.8→161.1	15
DDE-o,p'	24.43	246→176.2	30	248→176.2	30

Pesticides	t <sub>R</sub> (min)	MRM1	CE1(V)	MRM2	CE2(V)
Bromophos-ethyl	24.66	358.7→302.8	15	302.8→284.7	15
Propanil (DCPA)	24.7	161→99	30	161→90	25
isocarbophos	24.85	135.9→108	15	135.9→69	30
Chlorfenvinphos	24.88	266.9→159.1	15	322.8→266.8	10
Chlordane-trans	24.97	372.8→265.8	15	271.7→236.9	15
Tetraconazole	25.39	336→217.9	20	170.9→136	10
Butachlor	25.53	236.9→160.2	5	176.1→147.1	10
Prothiofos	25.78	266.9→239	5	266.9→221	20
Tetrachlorvinphose	26.1	328.9→109	22	330.9→109	22
Dieldrin	26.17	277→241	5	262.9→193	35
Beflubutamid	26.27	221→193.1	5	176.1→91.1	10
Methidathion	26.4	144.9→85	5	144.9→58.1	15
Procymidone	26.50	96→67.1	10	96→53.1	15
profenofos	26.57	207.9→63	30	338.8→268.7	15
napropamide	26.77	128→72.1	5	128→100.1	10
Butamifos	26.89	285.9→202	15	200→92	10
Hexaconazole	26.96	231→175	10	256→82.1	10
Chlorfenson	27.22	175→111	10	111→75	15
Paclobutrazol	27.35	236→125.1	10	125.1→89	20
Fluazifop-butyl	27.44	281.9→238	20	281.9→91	20
Isoprothiolane	27.52	162.1→85	20	162.1→134	5
Phosfolan	27.69	196→140	10	168→140	5
methoprotryne	27.83	256→212.1	15	256→170.1	25
Chlorobenzilate	27.97	251.1→139.1	15	139.1→111	10
Nitrofen	28.17	202→139.1	20	282.9→253	10
Disulfoton sulfone	28.33	213→153	5	213→96.9	15
Ethion	28.45	230.9→129	20	230.9→175	10
Fluorodifen	28.68	190→126.1	10	190→75	20
Diniconazole	28.87	267.9→232.1	10	269.9→232.1	10
fipronil	28.97	366.8→212.8	25	368.8→214.8	25
Myclobutanil	28.98	179→125.1	10	179→90	30
Cyproconazole	29.01	139→111	15	139→75	30
DDT-p,p'	29.02	235→165.2	20	237→165.2	20
Benalaxyl	29.05	148→77	35	148→105.1	20
Methoxychlor- o,p'	29.11	227.1→121.1	10	227.1→91.1	35
Diclofop-methyl	29.51	339.9→252.9	10	253→162.1	15
Propiconazole	29.53	172.9→74	45	258.8→69	10
Fenthion sulfone	30.07	309.9→105	10	135.9→92	10
Fludioxonil	30.28	248→154.1	20	248→182.1	10
Oxadixyl	30.35	163→132.1	5	163→117.1	25
Etoxazole	30.45	141→63.1	30	141→113	15
Famphur	30.57	218→109	15	217→92.9	10
Fenpropathrin	30.69	264.9→210	10	207.9→181	5
Leptophos	30.96	171→77.1	15	154.9→77.1	15
Pyridaphenthion	31.03	340→199	5	204→203.1	5

<b>Pesticides</b>	<b>t<sub>R</sub>(min)</b>	<b>MRM1</b>	<b>CE1(V)</b>	<b>MRM2</b>	<b>CE2(V)</b>
Phosmet	31.33	160→77.1	20	160→133.1	10
Bifenox	31.49	340.9→309.9	10	189.1→126	20
Acrinathrin	31.88	207.8→181.1	10	181→127	30
Cyhalothrin ( <i>lambda</i> )	31.88	208→181	5	181.1→152	25
Phosalone	31.89	182→111	15	182→102.1	15
Mefenacet	32.05	192→136.1	15	192→109.1	30
Azinphos-ethyl	32.56	132→77.1	15	160→77.1	20
Fluquinconazole	32.98	340→298	15	108→57	15
Coumaphos	33.75	361.9→109	15	210→182	10
Cyfluthrin	33.88	162.9→127	5	198.9→170.1	25
Flucythrinate	34.31	156.9→107.1	15	198.9→157	10
Fenbuconazole	34.78	197.9→129	5	128.9→102.1	15
Fluvalinate- <i>tau</i>	35.83	250→55	40	250→200	40
Difenoconazole	36.42	322.8→264.8	15	264.9→202	20

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