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Application Note

LC-MS/MS Analysis of Pesticide Residues in Rice and Unexpected Detection of Residues in an Organic Rice Sample

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Abstract

In this application note, we describe a rapid, multi-residue method for the determination of over 200 pesticides in basmati rice using a simple QuEChERS sample preparation procedure, followed by UltraPerformance LC (UPLC) coupled to tandem quadrupole mass spectrometry. The LC-MS/MS method presented in this study can also be employed for multi-analyte screening and quantification, providing a single method for more cost-effective analysis of pesticides in rice.

Benefits

- Rapid, multi-residue method for the determination of over 200 pesticides in rice.
- Obtain qualitative and quantitative information in a single injection.
- Standard addition capability in TargetLynx Software enables automated data processing.

Introduction

Rice is one of the most widely consumed foods in the world and its demand has increased in recent decades. Basmati rice is a variety of long grain rice which is traditionally cultivated in northern part of India. To improve its production yield, the use of various pesticides in various stages of cultivation has increased. Due to the adverse effects of these pesticides on human health and to the environment, the use of pesticides must be controlled and monitored.

According to the Horizon Scan database,[1] managed by the UK Food and Environment Research Agency, the most commonly found pesticides in rice exported from India between 2011 to 2014 were acephate, buprofezin, carbendazim, imidacloprid, isoprothiolane, methamidophos, pirimiphos-methyl, triazophos, tricyclazole, malathion and propiconazole. These pesticides were detected above the reporting limit.

A multi-residue pesticide method has been developed for the determination of pesticides in basmati rice. In this work, over 200 pesticides were analyzed using a simple QuEChERS sample preparation procedure, followed by UltraPerformance LC (UPLC) coupled to tandem quadrupole mass spectrometry.

Experimental

UPLC conditions

UPLC system:	ACQUITY UPLC H-Class
Column:	ACQUITY UPLC BEH C_{18}, 1.7 µm, 2.1 x 100 mm
Column temp.:	45 °C
Injection volume:	10 µL
Flow rate:	0.45 mL/min
Mobile phase A:	10 mM ammonium acetate (pH 5) in water
Mobile phase B:	10 mM ammonium acetate (pH 5) in methanol
Weak needle wash:	50/50 water/methanol (v/v)
Strong needle wash:	90/10 methanol/water (v/v)
Seal wash:	90/10 water/methanol

Gradient

Time (min)	Flow rate (mL/min)	%A	%B	Curve
Initial	0.45	98.0	2.0	6.0

Time (min)	Flow rate (mL/min)	%A	%B	Curve
0.25	0.45	98.0	2.0	6.0
12.25	0.45	1.0	99.0	6.0
13.0	0.45	1.0	99.0	6.0
13.01	0.45	98.0	2.0	6.0
17.0	0.45	98.0	2.0	6.0

MS conditions

MS system:	Xevo TQD
Ionization mode:	ESI+
Capillary voltage:	1 kV
Desolvation temp.:	500 °C
Desolvation gas flow:	1000 L/hr
Source temp.:	150 °C

Standards

A mix of 204 pesticides was prepared from Waters LC Multi-residue Pesticide Standards Kit, p/n: 186007574. The additional two pesticides Pirimiphos-methyl and triazophos were purchased from Sigma Aldrich. A mixture of all pesticides at 5 µg/mL was prepared in acetonitrile.

Samples

A variety of rice samples was purchased from local supermarkets. Jasmine rice (Sample A), three types of basmati rice (Samples B, C, and E), and organic brown rice (Sample D) were analyzed in this study.

The sample preparation protocol used was slightly modified from a previously published protocol by Lucia Pareja et al..[2]

Briefly, rice samples were ground to a fine powder using a food processor and 7.5 grams were weighed in a 50-mL centrifuge tube. The rice samples were then homogenized in 7.5 mL of water. The tube was shaken for 30 minutes using hand-motion shaker (Eberbach corporation, EL680.Q). Subsequently 15 mL of 1% glacial acetic acid in acetonitrile was added as an extraction solvent, followed by the addition of QuEChERS AOAC[3] material (Waters DisQuE QuEChERS pouches, p/n: 186006812). The tube was shaken for 4 minutes on a motion shaker and centrifuged (Eppendorf, 5810 R) at 3700 RPM for 5 minutes. A 4-mL aliquot of the supernatant was removed and dried under a stream of nitrogen. The dried extract was then redissolved in 2 mL of 50:50 water:acetonitrile and filtered through an Acrodisc Minispike Syringe (0.45 µm) PTFE Filter (WAT200559) before LC-MS/MS analysis.

To study linearity, solvent and matrix matched standards (MMS) calibration curves were created by spiking the pesticide mix from 0.00125 mg/kg to 0.32 mg/kg (1.25 ppb to 320 ppb) in solvent and the rice sample respectively.

For the standard addition work, six test portions of ground Basmati rice (Sample B) were spiked with the pesticide mix from 0.00125 mg/kg to 0.32 mg/kg. Each of the pre-spiked samples were then extracted as described above and analyzed to create a standard addition curve.

Results and Discussion

All of the pesticides were successfully detected at or below their EU maximum residue level (MRL) in the spiked rice sample. According to the EU legislation, the MRL level for the most commonly found pesticides in rice exported from India were between 0.01 to 8 mg/kg.

The vast majority of the pesticides (~85%) showed linearity with R^2 values >0.99. The remaining pesticides showed R^2 values above 0.98 in both the matrix-matched and solvent calibration curves. Examples of two pesticides in both solvent and matrix are shown in Figure 1.

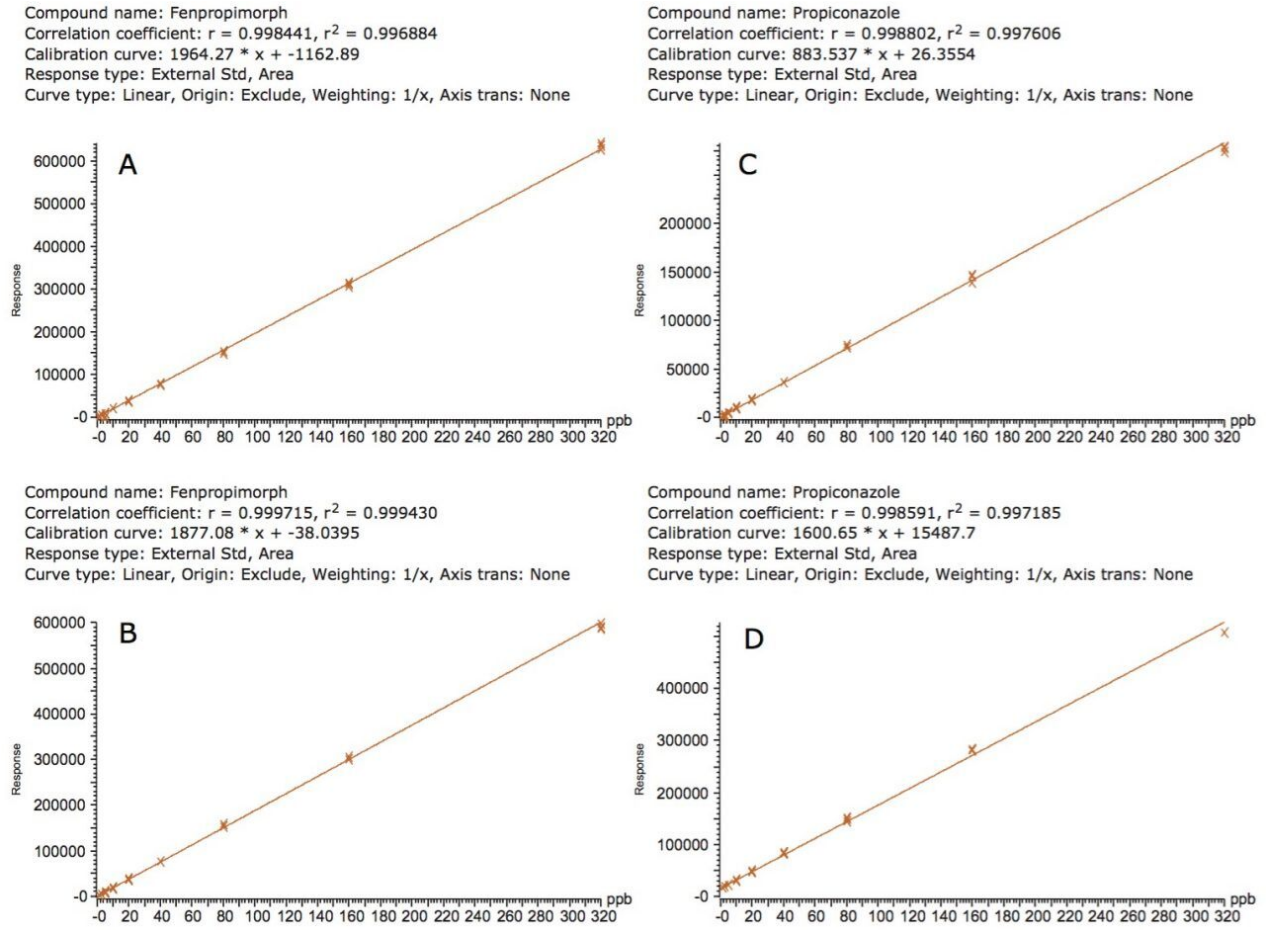

Figure 1. Example calibration curves of fenpropimorph and propiconazole in solvent (A and C, respectively) and the rice matrix (B and D, respectively).

Recovery

To assess the method recovery, rice samples were spiked at 0.01 mg/kg and extracted according to the described protocol. The samples were spiked in triplicate and each extract was analyzed in triplicate (total = nine injections). The recoveries of all of the pesticides together, along with standard deviations are shown in Figure 2.

As shown in Figure 2, the recoveries for most of the pesticides (92%) fell within the acceptable tolerance of 70% to 120% range (SANCO/12571/2014). Only 5% showed recoveries between 50% and 70%, and another 3% showed recoveries between 25% and 50%. The relative standard deviations (RSDs) were less than 20% except for nine compounds which had RSDs higher than 20%.

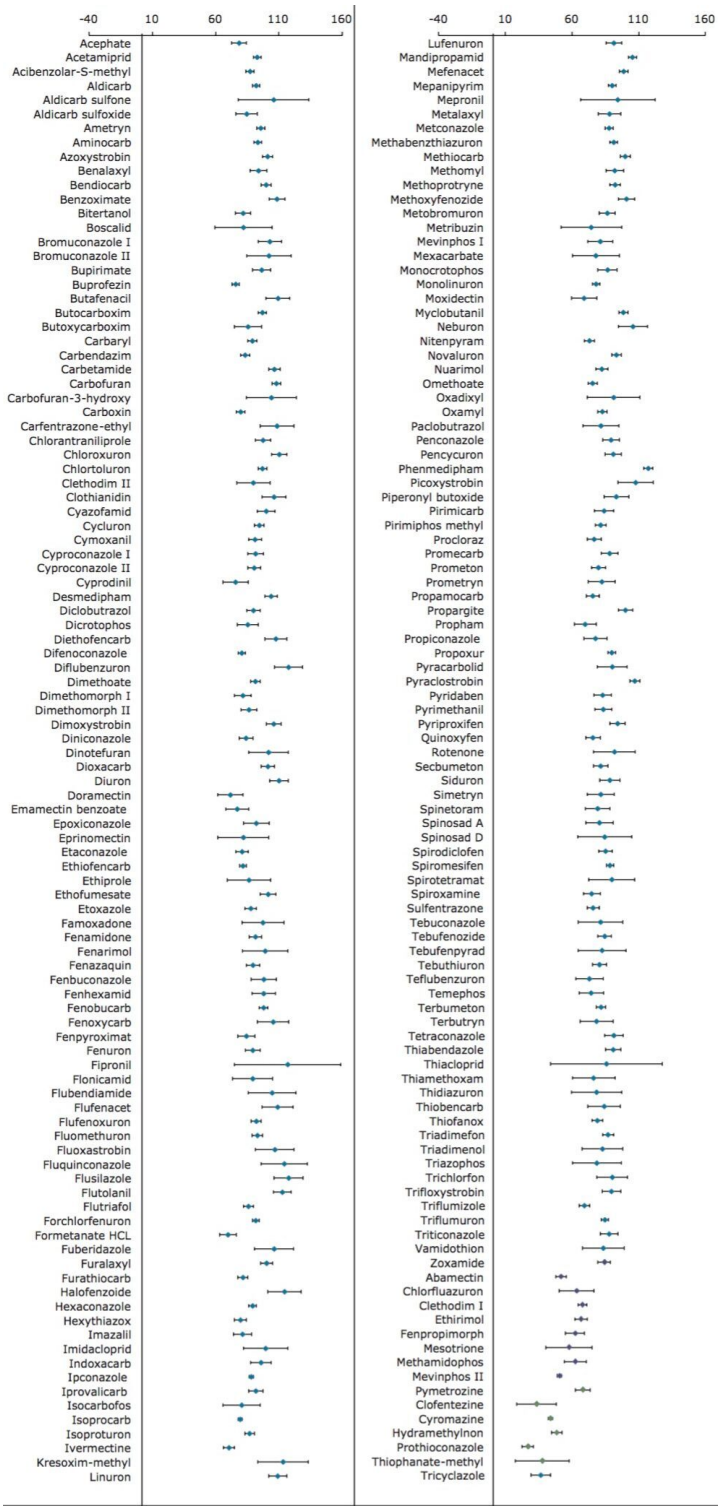

Figure 2. Percentage recovery of all the pesticides in a basmati rice sample at 0.01 mg/kg.

Sample analysis: matrix-matched calibration

For the remaining samples, quantification was performed against a matrix-matched calibration

curve. Piperonyl butoxide was detected in Sample C (0.0236 mg/kg) and Sample D (0.0054 mg/kg). Other pesticides were detected at concentrations <LOQ (e.g. imidacloprid in all samples, carbendazim in Sample C and Sample D, and piperonyl butoxide in Sample E. Figure 3 shows the MRM chromatogram for piperonyl butoxide in Samples C, D, and E, along with the solvent standard at 0.01 mg/kg.

Sample D was labeled as organic brown rice and was found to have low but detectable levels of two pesticides. The USDA National Organic Program4 includes a list of synthetic compounds that may or may not be used in organic crop production. This list does not include piperonyl butoxide or imidacloprid. The pesticides detected however were not at concentrations that exceeded the U.S. Food and Drug Administration (FDA) or the Environmental Protection Agency (EPA) regulatory tolerances and as such, no action level has been exceeded.

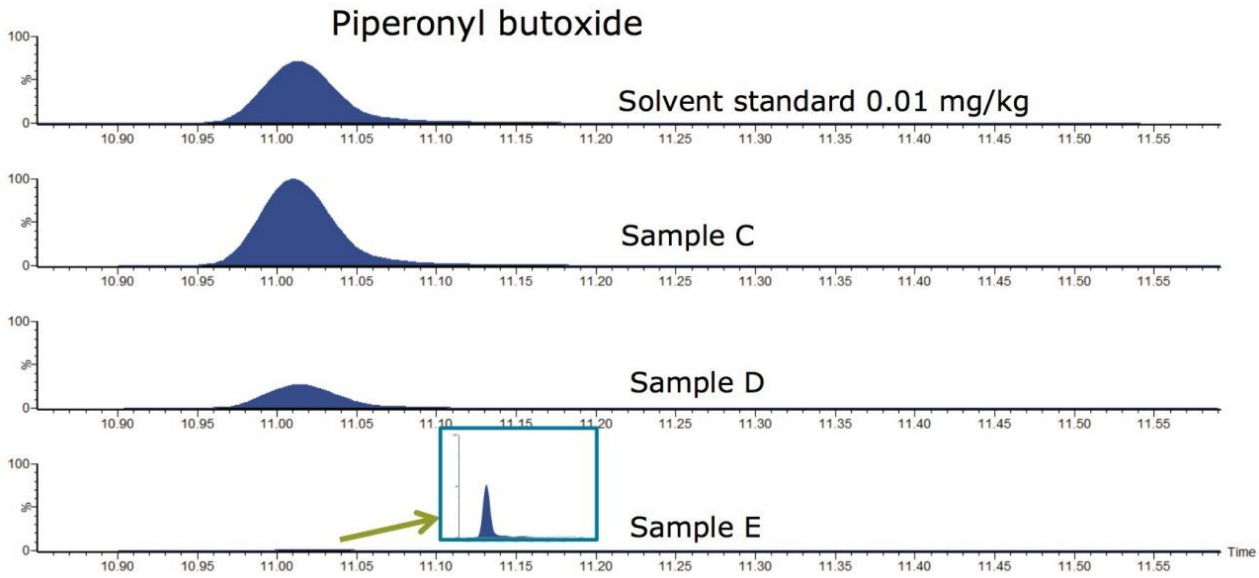

Figure 3. MRM chromatograms of piperonyl butoxide detected in Samples C, D, and E, along with the solvent standard at 0.01 mg/kg.

All of the incurred pesticide residues detected were identified from comparison of the retention time and ion ratio data with that from reference standards and found to be within the tolerance limit specified by the European Commission (Document No. SANCO/12571/2014).

Sample analysis: standard addition

A total of five rice samples were extracted and analyzed using the described method. One of the basmati rice samples (Sample B) was found to contain seven different pesticides. For accurate quantification of this

sample, a standard addition approach was taken. The incurred residues were then automatically quantified using TargetLynx. Figure 4 shows the quantification of buprofezin using the new standard addition feature in TargetLynx.

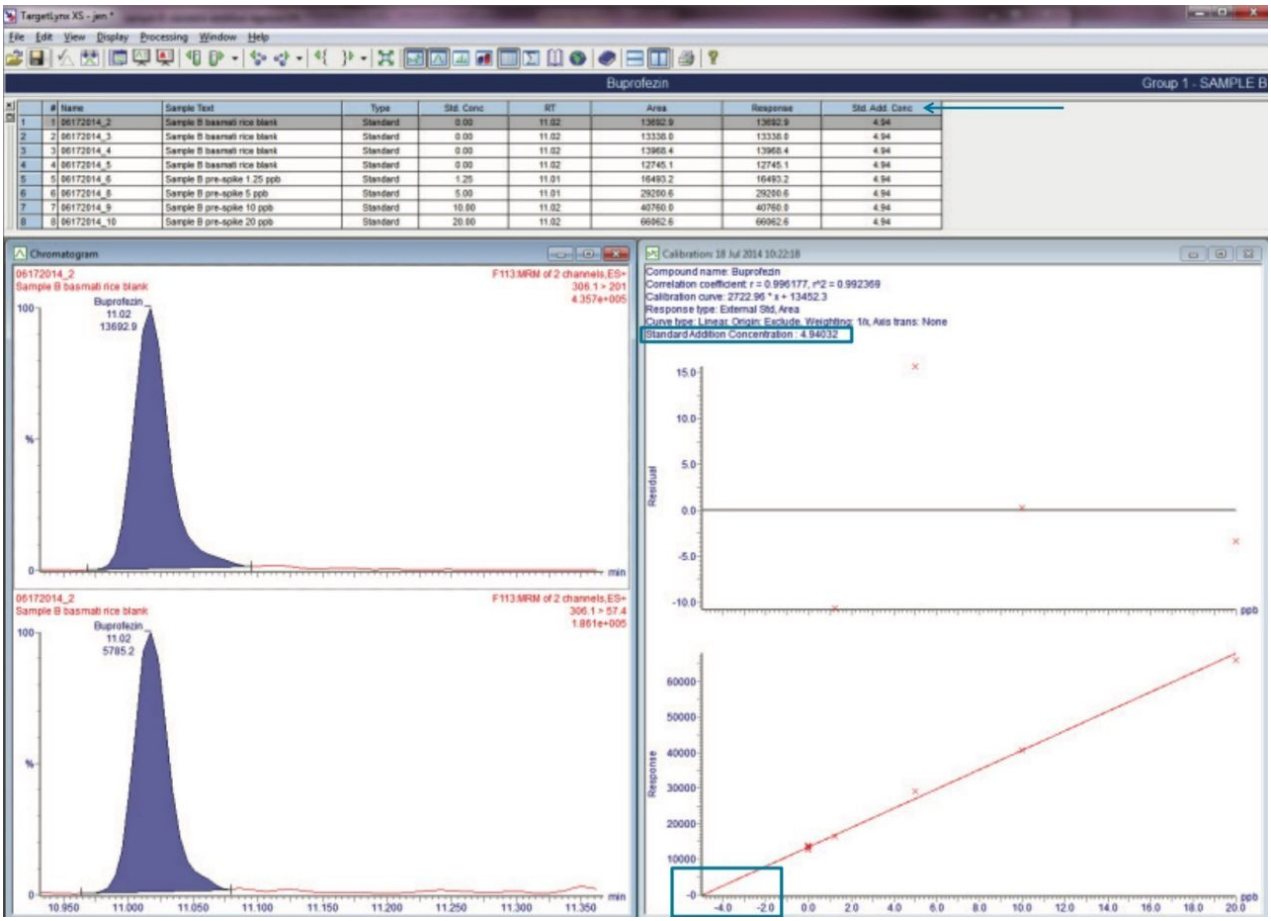

Figure 4. Quantification of buprofezin in Sample B using the standard addition feature of TargetLynx Software.

Using the standard addition method, seven pesticides were determined in basmati rice (Sample B) four of which were at concentrations greater than the lowest calibration point (0.00125 mg/kg); tricyclazole (0.0113 mg/kg), propiconazole (0.0099 mg/kg), buprofezin (0.005 mg/kg), and triazophos (0.0015 mg/kg). Acetamiprid, carbendazim, and imidacloprid were detected at concentrations below the lowest calibration point (0.00125 mg/kg). The detection of these compounds corresponds to those that have been listed in the Horizon Scan database. All the pesticides detected in basmati rice Sample B were at concentrations that were below the European MRLs.

Conclusion

All of the pesticides of interest were successfully analyzed using a simple QuEChERS sample preparation procedure and UPLC-MS/MS determination in rice samples.

- This method can easily detect all of the listed pesticides at 0.01 mg/kg, which is at or below the maximum residue level limit.
- More than 90% of the pesticides have shown recoveries within the range of 70% to 120% using a modified QuEChERS approach.
- TargetLynx enables efficient and automated data processing including a new standard addition capability.
- The LC-MS/MS method presented in this study can also be employed for multi-analyte screening and quantification, providing a single method for more cost-effective analysis of pesticides in rice.

References

1. http://services.leatherheadfood.com/foodline/horizonscan.aspx
2. L Pareja, V Cesio, H Heinzen, and A R Fernandez-Alba. Evaluation of various QuEChERS based methods for the analysis of herbicides and other commonly used pesticides in polished rice by LC-MS/MS. *Talanta.* 83(5): 1613–1622, 2011.
3. DisQuE Dispersive Sample Preparation. Waters brochure no. 720003048en. June, 2012.
4. Electronic Code of Federal Regulations (eCFR) Title 7, Agriculture, Part 205. National Organic Program. U.S. Government Publishing Office. 2015.

Appendix

Name	Ion mode	Precursor	CV	Product	CE	RT
Abamectin	ES+	890.6	10	567.4	11	12.05
Abamectin	ES+	890.6	10	305.2	25	12.05
Acephate	ES+	184.1	20	143.0	10	2.19
Acephate	ES+	184.1	20	49.0	20	2.19
Acetamiprid	ES+	223.0	35	126.0	20	4.92
Acetamiprid	ES+	223.0	35	56.1	15	4.92
Acibenzolar-S-methyl	ES+	210.9	40	135.9	30	8.53
Acibenzolar-S-methyl	ES+	210.9	40	69.0	35	8.53
Aldicarb	ES+	213.1	30	89.1	20	5.76
Aldicarb	ES+	213.1	30	116.1	10	5.76
Aldicarb sulfone	ES+	223.0	30	86.00	15	3.04
Aldicarb sulfone	ES+	223.0	30	148.0	10	3.04
Aldicarb sulfoxide	ES+	207.0	20	132.0	5	2.80
Aldicarb sulfoxide	ES+	207.0	20	89.0	15	2.80
Ametryn	ES+	228.1	40	186.1	20	8.57
Ametryn	ES+	228.1	40	68.1	35	8.57
Aminocarb	ES+	209.0	30	137.0	25	6.21
Aminocarb	ES+	209.0	30	152.0	15	6.21
Azoxystrobin	ES+	404.0	25	372.0	25	8.67
Azoxystrobin	ES+	404.0	25	329.0	30	8.67
Benalaxyl	ES+	326.1	25	148.0	20	10.00
Benalaxyl	ES+	326.1	25	91.0	30	10.00
Bendiocarb	ES+	224.1	25	167.0	10	6.78
Bendiocarb	ES+	224.1	25	109.0	15	6.78
Benzoximate	ES+	364.0	15	199.1	10	10.28
Benzoximate	ES+	364.0	15	105.0	25	10.28
Bitertanol	ES+	338.1	15	99.1	15	10.30
Bitertanol	ES+	338.1	15	70.1	10	10.30
Boscalid	ES+	342.9	40	307.0	45	8.89
Boscalid	ES+	342.9	40	139.9	20	8.89
Bromuconazole I	ES+	376.0	40	158.9	30	9.17
Bromuconazole I	ES+	376.0	40	70.1	20	9.17
Bromuconazole II	ES+	376.0	40	158.9	45	9.83
Bromuconazole II	ES+	376.0	40	70.1	40	9.83
Bupirimate	ES+	317.0	40	166.0	25	9.69
Bupirimate	ES+	317.0	40	108.0	25	9.69
Buprofezin	ES+	306.1	25	201.0	10	11.02
Buprofezin	ES+	306.1	25	57.4	25	11.02
Butafenacil	ES+	492.0	25	331.0	25	9.31
Butafenacil	ES+	492.0	25	180.0	35	9.31
Butocarboxim	ES+	213.0	25	75.0	15	5.71
Butocarboxim	ES+	213.0	25	116.0	10	5.71
Butoxycarboxim	ES+	223.0	25	106.0	10	3.00
Butoxycarboxim	ES+	223.0	25	166.0	5	3.00
Carbaryl	ES+	202.0	20	145.0	10	7.17
Carbaryl	ES+	202.0	20	117.0	25	7.17
Carbendazim	ES+	192.1	35	160.1	15	5.17
Carbendazim	ES+	192.1	35	132.1	30	5.17
Carbetamide	ES+	237.0	20	192.0	10	6.29
Carbetamide	ES+	237.0	20	118.0	15	6.29
Carbofuran	ES+	222.1	25	165.1	10	6.78
Carbofuran	ES+	222.1	25	123.0	20	6.78
Carbofuran-3-hydroxy	ES+	238.0	30	181.0	10	4.89

Name	Ion mode	Precursor	CV	Product	CE	RT
Carbofuran-3-hydroxy	ES+	238.0	30	163.0	15	4.89
Carboxin	ES+	236.0	30	143.0	15	7.03
Carboxin	ES+	236.0	30	87.0	25	7.03
Carfentrazone-ethyl	ES+	412.0	40	346.0	24	9.80
Carfentrazone-ethyl	ES+	412.0	40	266.0	18	9.80
Chlorantraniliprole	ES+	484.0	25	453.0	15	8.35
Chlorantraniliprole	ES+	484.0	25	286.0	15	8.35
Chlorfluazuron	ES+	539.8	42	158.0	15	11.73
Chlorfluazuron	ES+	539.8	42	382.9	20	11.73
Chloroxuron	ES+	291.1	35	72.0	20	9.28
Chloroxuron	ES+	291.1	35	164.1	15	9.28
Chlortoluron	ES+	213.0	30	72.0	15	7.53
Chlortoluron	ES+	213.0	30	46.0	15	7.53
Clethodim I	ES+	360.0	25	164.0	20	8.21
Clethodim I	ES+	360.0	25	268.1	10	8.21
Clethodim II	ES+	360.0	25	164.0	20	9.57
Clethodim II	ES+	360.0	25	268.1	10	9.57
Clofentezine	ES+	303.0	25	138	15	10.23
Clofentezine	ES+	303.0	25	102.0	35	10.23
Clothianidin	ES+	250.0	25	169.0	10	4.44
Clothianidin	ES+	250.0	25	132.0	15	4.44
Cyazofamid	ES+	325.0	25	107.9	15	9.56
Cyazofamid	ES+	325.0	25	261.0	10	9.56
Cycluron	ES+	199.0	35	89.1	15	7.96
Cycluron	ES+	199.0	35	69.2	20	7.96
Cymoxanil	ES+	199.0	20	128.0	10	5.10
Cymoxanil	ES+	199.0	20	111.0	20	5.10
Cyproconazole I	ES+	292.2	35	70.2	20	9.04
Cyproconazole I	ES+	292.2	35	125.1	30	9.04
Cyproconazole II	ES+	292.2	35	70.2	20	9.30
Cyproconazole II	ES+	292.2	35	125.1	30	9.30
Cyprodinil	ES+	226.0	40	93.0	35	9.89
Cyprodinil	ES+	226.0	40	108.0	25	9.89
Cyromazine	ES+	167.0	35	60.2	20	2.19
Cyromazine	ES+	167.0	35	108.1	20	2.19
Desmedipham	ES+	301.0	30	182.0	10	8.21
Desmedipham	ES+	301.0	30	136.0	25	8.21
Diclobutrazol	ES+	328.0	35	70.0	20	9.85
Diclobutrazol	ES+	328.0	35	158.9	35	9.85
Dicrotophos	ES+	238.0	25	112.0	10	4.25
Dicrotophos	ES+	238.0	25	193.0	10	4.25
Diethofencarb	ES+	268.0	20	226.0	10	8.53
Diethofencarb	ES+	268.0	20	124.0	30	8.53
Difenoconazole	ES+	406.0	40	251.1	25	10.47
Difenoconazole	ES+	406.0	40	111.1	35	10.47
Diflubenzuron	ES+	311.1	20	158.0	10	9.69
Diflubenzuron	ES+	311.1	20	141.0	15	9.69
Dimethoate	ES+	230.1	20	125.0	10	4.72
Dimethoate	ES+	230.1	20	199.0	20	4.72
Dimethomorph I	ES+	388.1	45	300.9	20	8.82
Dimethomorph I	ES+	388.1	45	165.0	30	8.82
Dimethomorph II	ES+	388.1	45	300.9	20	9.10
Dimethomorph II	ES+	388.1	45	165.0	30	9.10
Dimoxystrobin	ES+	327.1	20	116.1	20	9.79

Name	Ion mode	Precursor	CV	Product	CE	RT
Dimoxystrobin	ES+	327.1	20	205.2	10	9.79
Diniconazole	ES+	326.1	40	70.2	25	10.45
Diniconazole	ES+	326.1	40	159.0	30	10.45
Dinotefuran	ES+	203.0	20	129.0	10	2.78
Dinotefuran	ES+	203.0	20	113.0	10	2.78
Dioxacarb	ES+	224.1	20	123.1	15	4.78
Dioxacarb	ES+	224.1	20	167.1	10	4.78
Diuron	ES+	233.0	35	72.1	20	7.98
Diuron	ES+	233.0	35	46.3	15	7.98
Doramectin	ES+	916.6	20	331.2	23	12.31
Doramectin	ES+	916.6	20	593.4	14	12.31
Emamectin benzoate	ES+	886.6	10	158.0	35	11.45
Emamectin benzoate	ES+	886.6	10	126.0	30	11.45
Epoxiconazole	ES+	330.0	35	121.0	20	9.48
Epoxiconazole	ES+	330.0	35	101.0	35	9.48
Eprinomectin	ES+	914.6	20	186.0	35	11.91
Eprinomectin	ES+	914.6	20	144.0	41	11.91
Etaconazole	ES+	328.1	35	159.0	25	9.43
Etaconazole	ES+	328.1	35	205.0	15	9.43
Ethiofencarb	ES+	226.1	20	107.0	15	7.33
Ethiofencarb	ES+	226.1	20	164.0	10	7.33
Ethiprole	ES+	414.1	15	350.9	25	8.87
Ethiprole	ES+	414.1	15	396.9	10	8.87
Ethirimol	ES+	210.1	40	140.0	20	7.87
Ethirimol	ES+	210.1	40	98.0	25	7.87
Ethofumesate	ES+	287.1	35	121.1	15	8.60
Ethofumesate	ES+	287.1	35	259.1	10	8.60
Etoxazole	ES+	360.1	31	57.2	25	11.42
Etoxazole	ES+	360.1	31	141.0	25	11.42
Famoxadone	ES+	392.2	20	331.1	10	10.18
Famoxadone	ES+	392.2	20	238.0	15	10.18
Fenamidone	ES+	312.1	25	92.0	25	8.80
Fenamidone	ES+	312.1	25	236.1	15	8.80
Fenarimol	ES+	331.0	40	81.0	30	9.45
Fenarimol	ES+	331.0	40	268.0	25	9.45
Fenazaquin	ES+	307.2	20	57.2	20	11.80
Fenazaquin	ES+	307.2	29	161.0	15	11.80
Fenbuconazole	ES+	337.0	35	70.1	20	9.67
Fenbuconazole	ES+	337.0	35	125.0	30	9.67
Fenhexamid	ES+	302.1	45	97.2	25	9.37
Fenhexamid	ES+	302.1	45	55.3	35	9.37
Fenobucarb	ES+	208.0	25	94.9	15	8.53
Fenobucarb	ES+	208.0	25	152.0	10	8.53
Fenoxycarb	ES+	302.1	25	88.0	20	9.76
Fenoxycarb	ES+	302.1	25	116.1	10	9.76
Fenpropimorph	ES+	304.2	50	147.1	30	11.66
Fenpropimorph	ES+	304.2	50	57.2	30	11.66
Fenpyroximat	ES+	422.2	15	366.1	20	11.56
Fenpyroximat	ES+	422.2	15	138.1	30	11.56
Fenuron	ES+	165.0	30	71.9	15	4.48
Fenuron	ES+	165.0	30	45.9	15	4.48
Fipronil	ES+	453.9	20	368.1	25	9.75
Flonicamid	ES+	230.0	40	203.0	15	3.42
Flonicamid	ES+	230.0	40	148.0	25	3.42

Name	Ion mode	Precursor	CV	Product	CE	RT
Flubendiamide	ES+	683.0	10	408.0	5	9.90
Flubendiamide	ES+	683.0	10	274.0	30	9.90
Flufenacet	ES+	364.0	25	152.1	20	9.37
Flufenacet	ES+	364.0	25	194.1	10	9.37
Flufenoxuron	ES+	489.1	40	158.0	22	11.49
Flufenoxuron	ES+	489.1	40	141.0	46	11.49
Fluomethuron	ES+	233.2	35	72.2	20	7.39
Fluomethuron	ES+	233.2	35	46.4	20	7.39
Fluoxastrobin	ES+	459.0	35	427.0	15	9.37
Fluoxastrobin	ES+	459.0	35	188.0	35	9.37
Fluquinconazole	ES+	376.0	40	306.9	25	9.29
Fluquinconazole	ES+	376.0	40	348.8	20	9.29
Flusilazole	ES+	316.0	40	247.0	20	9.74
Flusilazole	ES+	316.0	40	165.0	25	9.74
Flutolanil	ES+	324.1	35	262.1	20	8.99
Flutolanil	ES+	324.1	35	65.0	35	8.99
Flutriafol	ES+	302.1	35	70.2	15	7.90
Flutriafol	ES+	302.1	35	123.1	30	7.90
Forchlorfenuron	ES+	248.1	30	129.0	15	8.08
Forchlorfenuron	ES+	248.1	30	93.0	35	8.08
Formetanate HCL	ES+	222.0	30	165.0	15	2.65
Formetanate HCL	ES+	222.0	30	46.0	25	2.65
Fuberidazole	ES+	185.0	45	157.0	20	6.27
Fuberidazole	ES+	185.0	45	156.0	25	6.27
Furalaxyl	ES+	302.1	25	95	25	8.65
Furalaxyl	ES+	302.1	25	242.1	15	8.65
Furathiocarb	ES+	383.2	25	194.9	15	10.87
Furathiocarb	ES+	383.2	25	252.0	10	10.87
Halofenzoide	ES+	331.1	10	104.9	15	8.76
Halofenzoide	ES+	331.1	10	275.0	5	8.76
Hexaconazole	ES+	314.0	35	70.1	20	10.23
Hexaconazole	ES+	314.0	35	159.0	25	10.23
Hexythiazox	ES+	353.0	25	168.1	25	11.25
Hexythiazox	ES+	353.0	25	228.1	15	11.25
Hydramethylnon	ES+	495.1	30	323.2	30	10.89
Hydramethylnon	ES+	495.1	30	151.1	35	10.89
Imazalil	ES+	297.0	40	159.0	20	9.65
Imazalil	ES+	297.0	40	69.0	20	9.65
Imidacloprid	ES+	256.1	30	175.1	20	4.41
Imidacloprid	ES+	256.1	30	209.1	15	4.41
Indoxacarb	ES+	528.0	10	150.0	25	10.58
Indoxacarb	ES+	528.0	10	203.0	30	10.58
Ipconazole	ES+	334.2	40	70.0	25	10.70
Ipconazole	ES+	334.2	40	125.0	25	10.70
Iprovalicarb	ES+	321.1	20	119.1	20	9.16
Iprovalicarb	ES+	321.1	20	203.1	10	9.16
Isocarbofos	ES+	291.1	20	121.1	30	8.06
Isocarbofos	ES+	291.1	20	231.1	15	8.06
Isoprocarb	ES+	194.1	25	95.1	15	7.72
Isoprocarb	ES+	194.1	25	137.1	10	7.72
Isoproturon	ES+	207.0	35	72.0	15	7.88
Isoproturon	ES+	207.0	35	46.1	15	7.88
Ivermectine	ES+	892.6	20	551.4	25	12.63
Ivermectine	ES+	892.6	20	569.4	14	12.63

Name	Ion mode	Precursor	CV	Product	CE	RT
Kresoxim-methyl	ES+	314.1	15	116.0	15	9.82
Kresoxim-methyl	ES+	314.1	15	206.0	5	9.82
Linuron	ES+	249.1	30	160.1	20	8.60
Linuron	ES+	249.1	30	182.1	15	8.60
Lufenuron	ES+	511.1	27	158.1	22	11.19
Lufenuron	ES+	511.1	27	141.2	40	11.19
Mandipropamid	ES+	412.0	30	328.0	15	8.95
Mandipropamid	ES+	412.0	30	125.0	35	8.95
Mefenacet	ES+	299.0	25	148.0	15	9.16
Mefenacet	ES+	299.0	25	120.0	25	9.16
Mepanipyrim	ES+	224.1	50	106.0	25	9.22
Mepanipyrim	ES+	224.1	50	77.0	35	9.22
Mepronil	ES+	270.1	35	119.0	25	8.99
Mepronil	ES+	270.1	35	91.0	35	8.99
Mesotrione	ES+	340.1	35	228.1	15	3.03
Mesotrione	ES+	340.1	35	104.0	30	3.03
Metalaxyl	ES+	280.1	25	220.1	15	7.98
Metalaxyl	ES+	280.1	25	192.1	20	7.98
Metconazole	ES+	320.1	35	70.0	25	10.24
Metconazole	ES+	320.1	35	125.0	30	10.24
Methabenzthiazuron	ES+	222.0	30	165.0	15	7.72
Methabenzthiazuron	ES+	222.0	30	150.0	30	7.72
Methamidophos	ES+	142.0	30	93.9	15	1.67
Methamidophos	ES+	142.0	30	124.9	15	1.67
Methiocarb	ES+	226.0	25	121.0	20	8.73
Methiocarb	ES+	226.0	25	169.0	10	8.73
Methomyl	ES+	163.0	15	88.0	10	3.39
Methomyl	ES+	163.0	15	106.0	10	3.39
Methoprotryne	ES+	272.2	45	170.2	30	8.54
Methoprotryne	ES+	272.2	45	198.2	25	8.54
Methoxyfenozide	ES+	369.1	15	149.1	15	9.06
Methoxyfenozide	ES+	369.1	15	313.2	10	9.06
Metobromuron	ES+	259.1	30	170.0	20	7.56
Metobromuron	ES+	259.1	30	148.1	15	7.56
Metribuzin	ES+	215.0	35	89.0	20	6.50
Metribuzin	ES+	215.0	35	131.0	20	6.50
Mevinphos I	ES+	225.1	20	127.1	15	4.84
Mevinphos I	ES+	225.1	20	193.1	10	4.84
Mevinphos II	ES+	225.1	25	127.1	15	5.53
Mevinphos II	ES+	225.1	25	193.1	5	5.53
Mexacarbate	ES+	223.2	30	166.1	15	8.93
Mexacarbate	ES+	223.2	30	151.0	25	8.93
Monocrotophos	ES+	224.1	25	127.1	15	3.89
Monocrotophos	ES+	224.1	25	98.1	10	3.89
Monolinuron	ES+	215.0	30	126.0	15	7.20
Monolinuron	ES+	215.0	30	99.0	30	7.20
Moxidectin	ES+	640.5	22	528.4	10	12.38
Moxidectin	ES+	640.5	22	498.3	10	12.38
Myclobutanil	ES+	289.1	35	124.9	30	9.20
Myclobutanil	ES+	289.1	35	150.9	25	9.20
Neburon	ES+	275.0	35	88.0	15	9.78
Neburon	ES+	275.0	35	57.0	20	9.78
Nitenpyram	ES+	271.1	30	125.9	30	3.25
Nitenpyram	ES+	271.1	30	224.9	10	3.25

Name	Ion mode	Precursor	CV	Product	CE	RT
Novaluron	ES+	493.0	30	158.0	15	10.75
Novaluron	ES+	493.0	30	141.1	30	10.75
Nuarimol	ES+	315.0	40	81.1	15	8.72
Nuarimol	ES+	315.0	40	252.0	20	8.72
Omethoate	ES+	214.1	25	125.1	20	2.52
Omethoate	ES+	214.1	25	183.1	10	2.52
Oxadixyl	ES+	279.0	25	219.0	10	6.34
Oxadixyl	ES+	279.0	25	132.0	30	6.34
Oxamyl	ES+	237.0	15	72.0	10	3.19
Oxamyl	ES+	237.0	15	90.0	10	3.19
Paclobutrazol	ES+	294.1	30	125.1	35	8.96
Paclobutrazol	ES+	294.1	30	70.2	20	8.96
Penconazole	ES+	284.0	30	70.1	15	9.95
Penconazole	ES+	284.0	30	159.0	25	9.95
Pencycuron	ES+	329.1	35	218.0	15	10.40
Pencycuron	ES+	329.1	35	125.0	25	10.40
Phenmedipham	ES+	301.0	30	168.0	10	8.42
Phenmedipham	ES+	301.0	30	136.0	20	8.42
Picoxystrobin	ES+	368.0	15	145.1	25	9.73
Picoxystrobin	ES+	368.0	15	205.1	10	9.73
Piperonyl butoxide	ES+	356.3	20	176.9	10	11.00
Piperonyl butoxide	ES+	356.3	20	119.0	35	11.00
Pirimicarb	ES+	239.1	35	72.0	20	7.54
Pirimicarb	ES+	239.1	35	182.1	15	7.54
Pirimiphos methyl	ES+	306.1	36	108.1	32	10.25
Pirimiphos methyl	ES+	306.1	36	164.1	22	10.25
Procloraz	ES+	376.0	35	307.1	15	10.27
Procloraz	ES+	376.0	35	70.1	25	10.27
Promecarb	ES+	208.1	25	109.0	15	8.90
Promecarb	ES+	208.1	25	151.0	10	8.90
Prometon	ES+	226.0	40	86.3	30	8.38
Prometon	ES+	226.0	40	184.3	20	8.38
Prometryn	ES+	242.0	40	158.0	25	9.29
Prometryn	ES+	242.0	40	200.1	20	9.29
Propamocarb	ES+	189.1	30	102.0	15	2.83
Propamocarb	ES+	189.1	30	144.0	10	2.83
Propargite	ES+	368.2	15	231.1	10	11.42
Propargite	ES+	368.2	15	175.1	15	11.42
Propham	ES+	180.0	15	138.0	10	7.20
Propham	ES+	180.0	15	120.0	15	7.20
Propiconazole	ES+	342.0	40	69.0	20	10.06
Propiconazole	ES+	342.0	40	159.0	25	10.06
Propoxur	ES+	210.0	20	111.0	15	6.67
Propoxur	ES+	210.0	20	168.0	5	6.67
Prothioconazole	ES+	344.0	25	326.0	10	10.05
Prothioconazole	ES+	344.0	25	189.0	20	10.05
Pymetrozine	ES+	218.0	35	105.0	20	3.66
Pymetrozine	ES+	218.0	35	79.0	35	3.66
Pyracarbolid	ES+	218.1	30	125.1	20	6.83
Pyracarbolid	ES+	218.1	30	97.1	30	6.83
Pyraclostrobin	ES+	388.1	25	163.0	25	10.18
Pyraclostrobin	ES+	388.1	25	193.9	10	10.18
Pyridaben	ES+	365.1	10	147.1	25	11.83
Pyridaben	ES+	365.1	10	309.1	10	11.83

Name	Ion mode	Precursor	CV	Product	CE	RT
Pyrimethanil	ES+	200.0	40	107.0	25	8.58
Pyrimethanil	ES+	200.0	40	82.0	25	8.58
Pyriproxifen	ES+	322.1	25	96.0	15	11.10
Pyriproxifen	ES+	322.1	25	227.1	10	11.10
Quinoxyfen	ES+	308.0	61	197.0	30	11.22
Quinoxyfen	ES+	308.0	61	161.9	35	11.22
Rotenone	ES+	395.0	46	213.1	25	9.70
Rotenone	ES+	395.0	46	192.1	20	9.70
Secbumeton	ES+	226.2	40	170.2	20	8.33
Secbumeton	ES+	226.2	40	100.2	25	8.33
Siduron	ES+	233.0	35	93.8	20	8.60
Siduron	ES+	233.0	35	137.0	15	8.60
Simetryn	ES+	214.0	40	124.0	20	7.69
Simetryn	ES+	214.0	40	95.9	25	7.69
Spinetoram	ES+	748.5	25	142.2	30	11.65
Spinetoram	ES+	748.5	25	98.1	35	11.65
Spinosad A	ES+	732.6	40	142.0	30	11.29
Spinosad A	ES+	732.6	40	98.1	35	11.29
Spinosad D	ES+	746.5	20	142.0	30	11.66
Spinosad D	ES+	746.5	20	98.1	35	11.66
Spirodiclofen	ES+	411.1	25	71.2	15	11.59
Spirodiclofen	ES+	411.1	25	313.0	10	11.59
Spiromesifen	ES+	371.1	20	273.1	5	11.37
Spiromesifen	ES+	371.1	20	255.1	25	11.37
Spirotetramat	ES+	374.0	35	302.0	30	9.37
Spirotetramat	ES+	374.0	35	330.0	15	9.37
Spiroxamine	ES+	298.0	35	144.0	20	9.35
Spiroxamine	ES+	298.0	35	100.0	30	9.35
Sulfentrazone	ES+	387.0	50	145.8	35	7.14
Sulfentrazone	ES+	387.0	50	307.0	30	7.14
Tebuconazole	ES+	308.0	35	70.1	20	9.99
Tebuconazole	ES+	308.0	35	125.0	35	9.99
Tebufenozide	ES+	353.1	15	133.0	20	9.73
Tebufenozide	ES+	353.1	15	297.1	10	9.73
Tebufenpyrad	ES+	334.0	40	117.0	25	10.94
Tebufenpyrad	ES+	334.0	40	145.0	25	10.94
Tebuthiuron	ES+	229.0	35	172.0	15	6.98
Tebuthiuron	ES+	229.0	35	116.0	25	6.98
Teflubenzuron	ES+	380.9	30	158.0	20	11.15
Teflubenzuron	ES+	380.9	30	140.9	30	11.15
Temephos	ES+	466.8	40	125.0	30	11.06
Temephos	ES+	466.8	40	418.9	20	11.06
Terbumeton	ES+	226.1	35	170.1	15	8.67
Terbumeton	ES+	226.1	35	114.1	25	8.67
Terbutryn	ES+	242.1	35	186.1	20	9.43
Terbutryn	ES+	242.1	35	91.0	25	9.43
Tetraconazole	ES+	372.0	40	70.1	20	9.50
Tetraconazole	ES+	372.0	40	159.0	25	9.50
Thiabendazole	ES+	202.0	50	175.0	25	5.97
Thiabendazole	ES+	202.0	50	131.0	30	5.97
Thiacloprid	ES+	253.0	35	126.0	20	5.46
Thiacloprid	ES+	253.0	35	90.1	35	5.46
Thiamethoxam	ES+	292.0	25	211.2	10	3.66
Thiamethoxam	ES+	292.0	25	132.0	20	3.66

Name	Ion mode	Precursor	CV	Product	CE	RT
Thidiazuron	ES+	221.0	30	101.9	15	6.85
Thidiazuron	ES+	221.0	30	93.9	15	6.85
Thiobencarb	ES+	258.1	25	125.0	15	10.30
Thiobencarb	ES+	258.1	25	89.0	35	10.30
Thiofanox	ES+	219.0	10	57.0	5	7.42
Thiofanox	ES+	219.0	10	76.0	5	7.42
Thiophanate-methyl	ES+	343.0	25	151.0	20	6.76
Thiophanate-methyl	ES+	343.0	25	93.0	35	6.76
Triadimefon	ES+	294.1	30	69.3	20	9.07
Triadimefon	ES+	294.1	30	197.2	15	9.07
Triadimenol	ES+	296.1	20	70.2	10	9.27
Triadimenol	ES+	296.1	20	227.1	10	9.27
Triazophos	ES+	314.1	31	118.9	35	9.25
Triazophos	ES+	314.1	31	161.9	18	9.25
Trichlorfon	ES+	257.0	35	109.0	15	4.64
Trichlorfon	ES+	257.0	35	79.0	30	4.64
Tricyclazole	ES+	190.0	50	163.0	20	5.71
Tricyclazole	ES+	190.0	50	136.0	25	5.71
Trifloxystrobin	ES+	409.0	34	186.0	16	10.55
Trifloxystrobin	ES+	409.0	34	145.0	40	10.55
Triflumizole	ES+	346.0	20	277.9	20	10.71
Triflumizole	ES+	346.0	20	73.1	15	10.71
Triflumuron	ES+	359.0	25	156.1	20	10.26
Triflumuron	ES+	359.0	25	139.1	30	10.26
Triticonazole	ES+	318.1	30	70.1	20	9.45
Triticonazole	ES+	318.1	30	124.9	30	9.45
Vamidothion	ES+	288.0	20	146.0	10	4.86
Vamidothion	ES+	288.0	20	118.0	25	4.86
Zoxamide	ES+	336.0	35	187.1	20	10.05
Zoxamide	ES+	336.0	35	159.0	35	10.05

Featured Products

ACQUITY UPLC H-Class System <https://www.waters.com/10138533>

Xevo TQD Triple Quadrupole Mass Spectrometry <https://www.waters.com/waters/en_US/Xevo-TQD-

Triple-Quadrupole-Mass-Spectrometry/nav.htm?cid=134608730>

MassLynx Mass Spectrometry Software <https://www.waters.com/waters/en_US/MassLynx-MS-Software/nav.htm?cid=513662>

TargetLynx Application Manager <https://www.waters.com/513791>

Quanpedia Database <https://www.waters.com/10148049>

Available for purchase online:

ACQUITY UPLC BEH C18, 1.7 µm, 2.1 mm X 100 mm Column < https://www.waters.com/waters/partDetail.htm?partNumber=186002352>

DisQuE Quecher, 1.5 g Sodium Acetate and 6 g MgSO4, 50 mL Pouch < https://www.waters.com/waters/partDetail.htm?partNumber=186006812>

LC Multiresidue Pesticide Standards Kit < https://www.waters.com/waters/partDetail.htm?partNumber=186007574>

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