

A novel comprehensive strategy for residual pesticide analysis in cannabis flower

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Abstract

Eight U.S. states have approved the recreational sale of cannabis, with an additional 35 states allowing some degree of medical use, or use of cannabidiol-containing products for limited medicinal purposes. Every state that has legalized use in some form has testing requirements for pesticide residues in cannabis flower and cannabis products before retail sale. Each state has its own testing requirements regarding the specific pesticides to be evaluated, and the action levels that the tested pesticides must not exceed. Pacific Agricultural Laboratory has validated workflows using LC/MS/MS and GC/MS/MS to cover these different requirements across a range of products. This Application Note describes a comprehensive pesticide screening and quantitation approach for up to 214 pesticide residues in cannabis flower. The methodology uses a single extraction and combined analyses using the Agilent 7010 Tandem Quadrupole and Agilent 6470 Tandem Quadrupole Mass Spectrometer systems. For GC/MS/MS, limits of quantitation (LOQs) were determined to be 0.1 mg/kg for 94 % of the comprehensive target list. For LC/MS/MS, LOQs were determined to be 0.1 mg/kg for 89 % of the comprehensive target list. Analyte recoveries were between 70–120 % for 212 of the 214 compounds.

Introduction

Quantifying pesticide residues in cannabis flower is a complex problem. The challenge is partially due to the concentration level disparities between naturally occurring cannabinoids, incurred pesticide residues, and other endogenous compounds such as terpenes, which typically are found at concentrations of 1 to 2 % by weight of the flower. The typical extraction process has the potential for low pesticide recoveries and deleterious effects on the analytical instrumentation caused by co-extracted material. Our approach to sample preparation exploits the benefits of highly sensitive instruments, allowing for significant sample dilution to decrease these matrix effects.

An acetonitrile extraction of the cannabis extract was used, followed by passing the extract through a SPE cartridge.

The protocol was based on the sample preparation procedure used to determine pesticides in dried hops¹ with an additional dilution step before analysis. The dilute eluate is then passed to both GC/MS/MS and LC/MS/MS workflows, as described below, for comprehensive pesticide residue analysis. More than 200 known pesticides were tested, going far beyond the often-referenced Oregon list of approved pesticides for cannabis. To help minimize matrix effects and provide acceptable recoveries for each target list, dispersive SPE cleanup (dSPE) techniques were developed for each platform.

The GC/MS/MS system was equipped with two columns of differing polarities, and used midcolumn backflush with the Agilent Purged Ultimate Union. The Agilent 7010 GC/MS/MS includes a High Efficiency Source (HES), which results in the creation of up to 20-times more ions compared to the Standard Agilent Inert

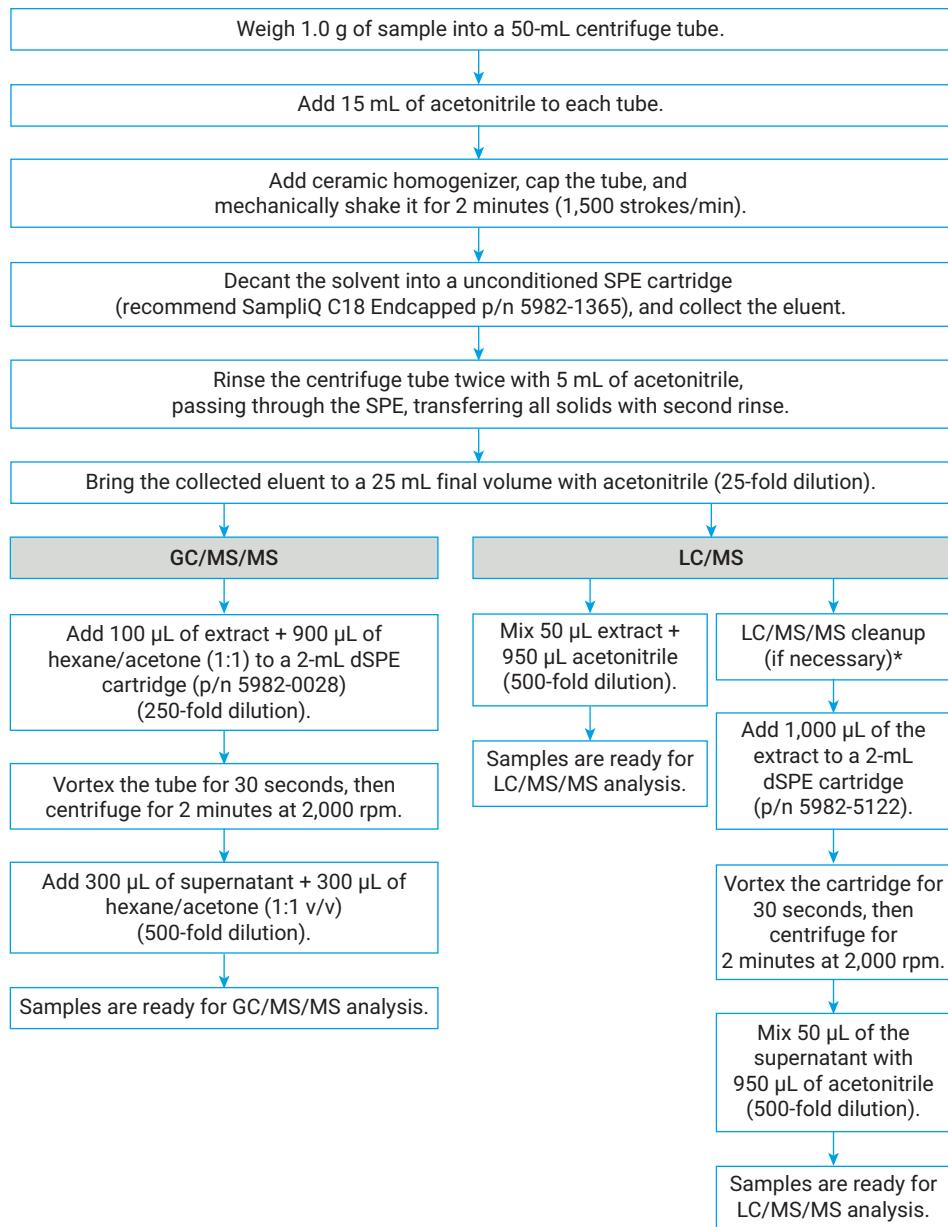
Source. This efficiency permits high sample dilution factors that minimize matrix effects while still achieving the required limits of quantitation (LOQs), determined to be 0.1 mg/kg for 94 % of the comprehensive target list.

To achieve the necessary separations, the LC/MS/MS system used an Agilent InfinityLab Poroshell 120 Phenyl-Hexyl column (2.1 × 100 mm, 2.7 µm, p/n 695775-912). An online multisampler pretreatment procedure was used to improve the peak shape of early eluting compounds. This procedure entails sandwiching the sample between HPLC grade water using the sandwiched injection features of the autosampler. The sensitivity of the Agilent 6470 MS/MS system allows for high dilution factors that minimize matrix effects, and achieve the required LOQs. For the comprehensive target list, 89 % of the compounds achieved an LOQ of 0.1 mg/kg.

Experimental

Sample preparation

Figure 1 shows the sample preparation workflow for GC/MS/MS and LC/MS/MS. First, 1.0 g of homogenized cannabis flower was accurately weighed into 50-mL centrifuge tubes. Then, 15 mL of high-purity, pesticide-grade acetonitrile was added to each tube. The tubes were sealed and mechanically shaken for 2 minutes at 1,500 strokes/minute. The extract was passed through a unconditioned polymeric solid phase extraction (SPE) cartridge and eluted by gravity. The centrifuge tube was rinsed twice with 5 mL of acetonitrile, and passed through the SPE cartridge. During the final rinse, the sample solids were transferred to the SPE cartridge. All fractions were collected and brought to a final volume of 25 mL with acetonitrile.



* An optional cleanup step can be used for LC/MS/MS compounds if necessary (that is, the observation of matrix interference, poor chromatography, and so forth, when using the simpler dilution approach) using the Agilent dispersive-SPE kit (p/n 5982-5122). In this procedure, 1 mL of collected sample extract is added to a 2-mL d-SPE cartridge, vortexed/centrifuged, and diluted 20-fold (50 μL treated extract + 950 μL acetonitrile) prior to analysis. Please note, PSA is a powerful cleanup sorbent for cannabis analysis, and Daminozide, Spinosad, Spirotetramat, and Spiroxamine will bind to the PSA in the d-SPE, and thus are not recoverable. These compounds will need to be measured and quantified prior to this cleanup step.

Figure 1. Sample preparation flowchart.

Results and discussion

Sample preparation

Using a simple extraction and SPE cleanup of the cannabis flower matrix substantially reduces the issues associated with QuEChERS extraction, which include spiking pH and exothermic conditions that can degrade sensitive pesticides. In our approach, we eliminate the hydration step necessary with QuEChERS, thus increasing the recovery of the more polar pesticides.

For GC/MS/MS analysis, a cleanup step was performed for each sample extract before analysis using the Agilent Universal dispersive-SPE kit (p/n 5982-0028). In this procedure, 100 µL of the collected extract was added to 900 µL of hexane:acetone (1:1, v/v) in the 2-mL cleanup tube. The slurry was vortexed and centrifuged, and 300 µL of the supernatant was added to 300 µL of hexane:acetone (1:1, v/v) resulting in an overall 500-fold dilution before analysis.

For LC/MS/MS analysis, 50 µL of the collected extract was transferred to an autosampler vial containing 950 µL of acetonitrile, resulting in a 500-fold dilution.

If additional sample cleanup is necessary due to the complexity of the coextracted matrix (see note in Figure 1), a third cleanup procedure can be followed using the Agilent dispersive-SPE kit (p/n 5982-5122). In this tertiary procedure, 1 mL of collected sample extract is added to a 2-mL dSPE cartridge, vortexed, centrifuged, and diluted to a final factor of 500-fold before analysis. Please note, under these conditions, Daminozide, Spinosad, Spirotetramat, and Spiroxamine bind to the PSA in the dSPE, and are not recoverable. Therefore, these compounds need to be measured and quantified before the cleanup procedure.

Table 1. GC/MS/MS Conditions.

GC Conditions	
GC	Agilent 7890B
Column 1	Agilent HP-35MS, 15 m × 0.25 mm, 0.25 µm (p/n 122-3812)
Column 2	Agilent HP-5, 15 m × 0.25 mm, 0.25 µm (p/n 19091J-431)
Inlet	Multimode (MMI)
Inlet liner	Splitless, 4 mm single taper w/ deactivated fused silica wool Recommend: Agilent Ultra Inert (p/n 5190-2293)
Column 1 flow	1.2 mL/min
Column 2 flow	1.25 mL/min
Inlet temperature	180 °C
Inlet temperature program	180 to 280 °C at 400 °C/min
Injection volume	2 µL
Oven temperature program	70 °C (1 minute) 35 °C/min to 180 °C (0 minutes) 10 °C/min to 200 °C (0 minutes) 8 °C/min to 300 (4.5 minutes)
Run time	23.14 minutes
Column backflush	Agilent Purged Ultimate Union (p/n G3186) Intra-run, when deltamethrin elution passes the Ultimate Union Post run, 2.4 minutes at 4.0 mL/min
MS Conditions	
Spectrometer	Agilent 7010 triple quadrupole GC/MS with high efficiency source (HES)
Mode	Electron ionization (EI)
Transfer line temperature	300 °C
Source temperature	230 °C (280 °C recommended)
Quadrupole temperature	150 °C

Table 2. LC/MS/MS Conditions.

HPLC Conditions	
HPLC	Agilent 1260 Infinity multisampler
Column	Agilent InfinityLab Poroshell 120 Phenyl-Hexyl, 2.1 × 100 mm, 2.7 µm (p/n 695775-912)
Column temperature	45 °C
Injection volume	2 µL
Mobile phase A	5 mM Ammonium formate + 0.1 % formic acid in 95:5 (v/v) water:methanol
Mobile phase B	5 mM Ammonium formate + 0.1 % formic acid in 95:5 (v/v) methanol:water
Flow rate	0.4 mL/min
MS Conditions	
Gas temperature	250 °C
Gas flow	9 L/min
Nebulizer	35 psi
Sheath gas heater	300 °C (200 °C recommended)
Sheath gas flow	12 L/min
Capillary	4,000 (+) 3,500 (-)

Table 3. Mobile phase gradient.

	Time (min)	A	B
1	0.50	95.0 %	5.0 %
2	5.00	50.0 %	50.0 %
3	10.00	5.0 %	95.0 %
4	16.00	5.0 %	95.0 %
5	16.10	95.0 %	5.0 %

GC/MS/MS

Matrix-matched calibrators were prepared over a range of 0.2 ng/mL to 20 ng/mL. Regression curves were constructed with a minimum of five levels for linear models and a minimum of six levels for quadratic models. Using a 1/X weighting factor, and excluding the origin, all correlation coefficients were >0.990. All quantitation was

performed using the external standard technique (absolute response versus concentration).

The HES of the 7010 GC/MS/MS system makes high sample dilution possible for injection of analytes at sub-ppb levels as part of a routine workflow. By diluting the cannabis flower samples as much as 500-fold, overall robustness of the analyses

was improved. This procedure greatly reduced matrix effects/interferences, and allowed for improved instrument up-time by preventing frequent inlet maintenance and column replacement. Using the defined sample preparation techniques and dilution factors, LOQs of 0.1 mg/kg for 94 % of the target list of over 70 compounds were determined.

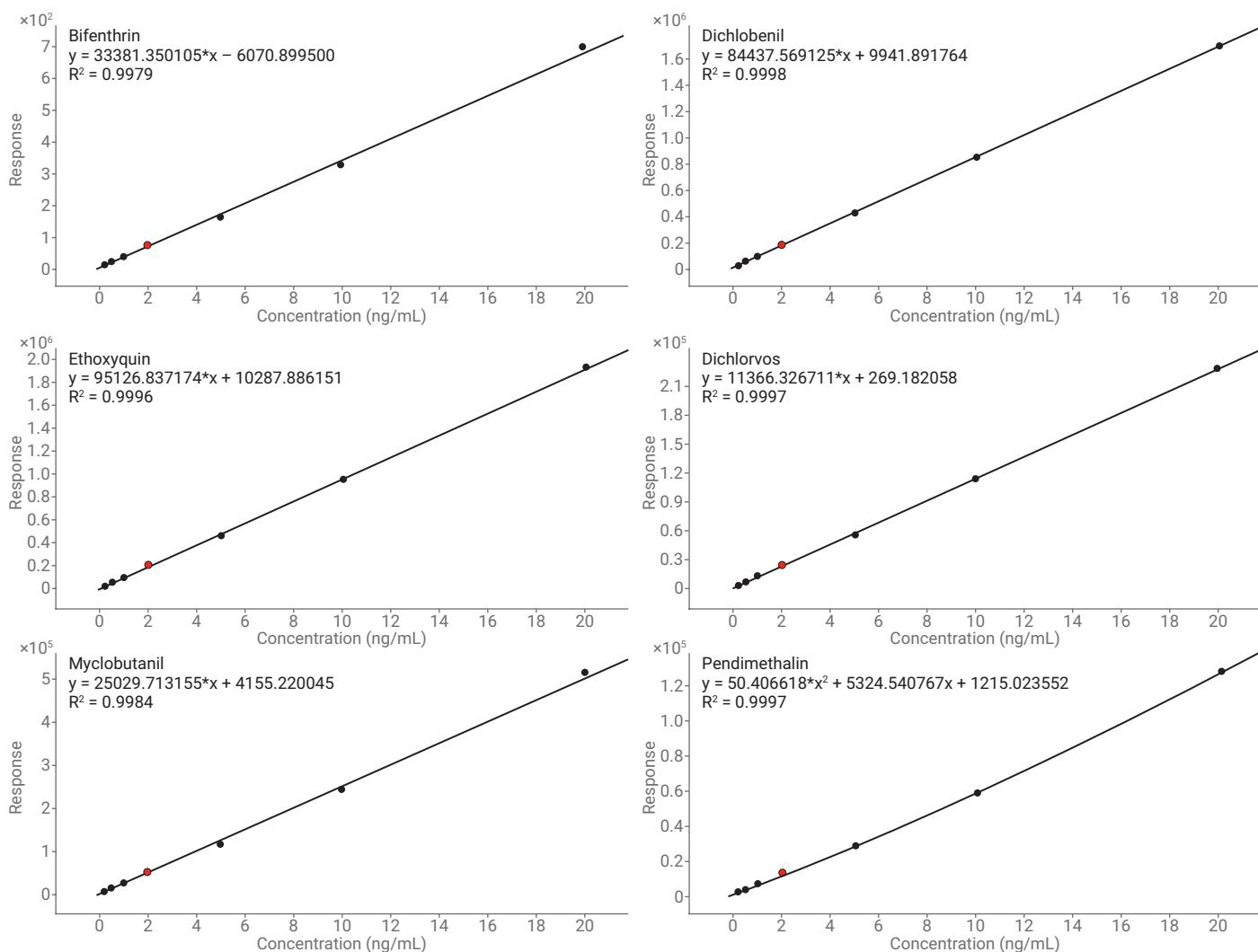


Figure 2. Select GC/MS/MS calibration curves (red triangles represent injected QCs).

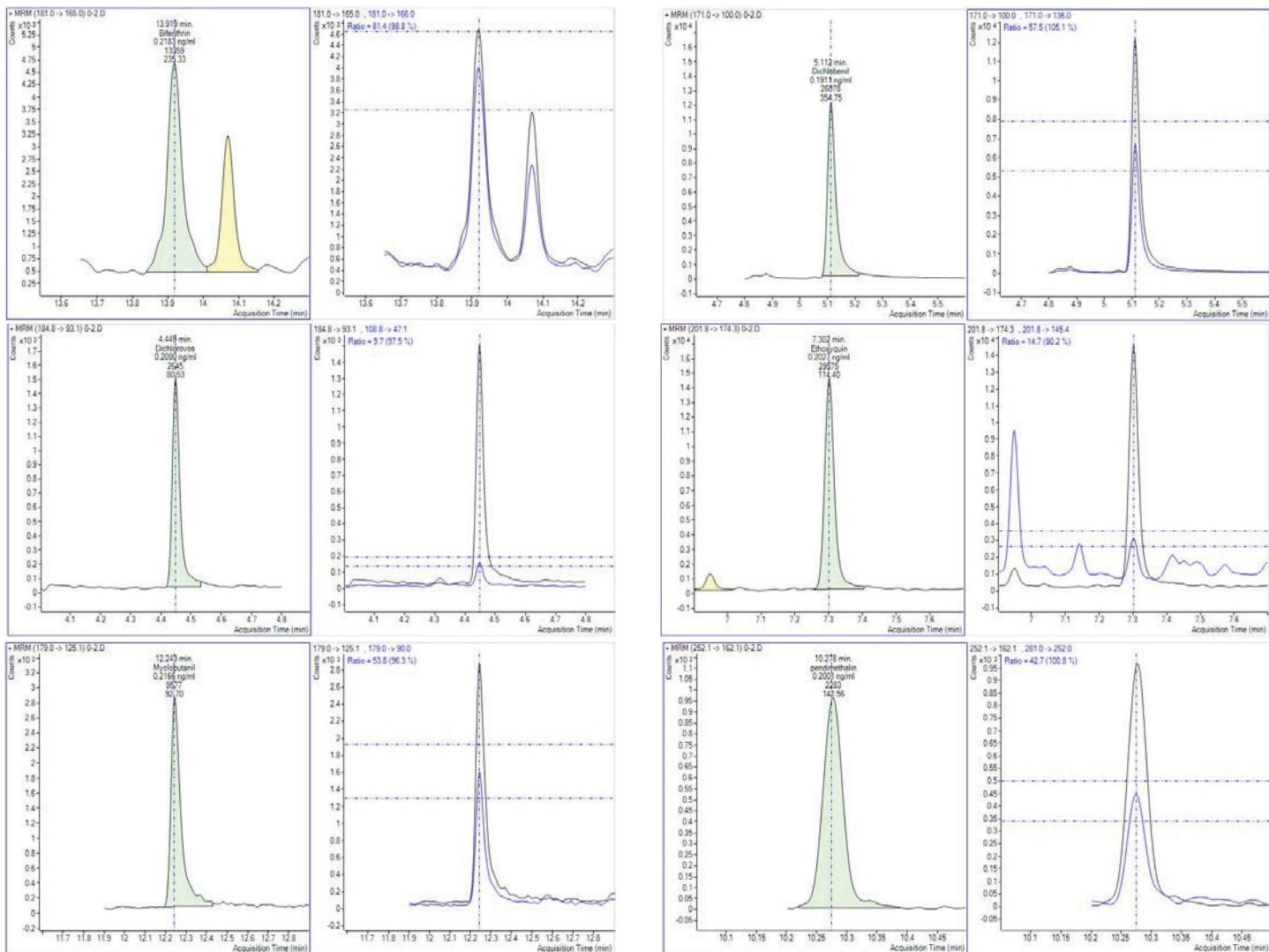


Figure 3. Select GC/MS/MS MRM chromatograms.

Table 4. In-vial and in-sample concentrations for compounds analyzed.

Compound	In-vial conc. (ng/mL)	In-sample conc. (mg/kg)
Bifenthrin	0.2	0.1
Dichlobenil	0.2	0.1
Dichlorvos	0.2	0.1
Ethoxyquin	0.2	0.1
Myclobutanil	0.2	0.1
Pendimethalin	0.2	0.1

Table 5. GC/MS/MS recoveries and % RSD (n = 5 replicates).

Compound	% Recovery	%RSD
α-BHC	93	2.27
Aldrin	100	3.14
β-BHC	93	2.14
Benfluralin	85	4.11
Bifenthrin	108	6.96
Bolstar	112	5.53
Bromopropylate	105	5.67
Captan	133	5.79
Chlopyrifos	122	3.87
Chlordane <i>cis/trans</i>	100	2.12
Chloroneb	89	1.37
Chloropropham	90	6.71
Chlorothalonil	86	8.58
<i>cis</i> Nonachlor	95	3.91
Cyfluthrin	106	5.45
Cyhalothrin	94	3.44
Cypermethrin	97	12.82
δ-BHC	93	2.08
Dacthal	95	3.71
Deltamethrin	112	9.08
Diazinon	106	4.47
Dichlobenil	86	1.62
Dichlofenthion	101	3.48
Dichlorovos	95	3.83
Diclofop methyl	115	6.69
Dicloran	90	5.04
Dicofol	88	3.50
Diphenamid	94	5.29
Dithiopyr	98	4.85
Esfenvalerate	109	8.96
Ethalfluralin	87	3.20
Ethofumesate	113	4.12
Ethoprop	99	1.84
Ethoxyquin	84	4.91
Etoxazole	115	9.41
Etridiazole	100	3.55

All compounds listed in Table 4 had LOQs of 0.1 mg/kg, except for those given in Table 5.

Table 6. Compounds with LOQs different from those in Table 4.

GC-MS/MS	
Compound	LOQ (mg/kg)
Captan	0.5
Deltamethrin	0.5
Dicloran	0.5
Pirimicarb	0.2

LC/MS/MS

Matrix-matched calibrators were prepared over a range of 0.2 ng/mL to 10 or 20 ng/mL. Regression curves were constructed with a minimum of five levels for linear models and a minimum of six levels for quadratic models. Using a 1/X weighting factor and excluding the origin, all correlation coefficients were >0.990. All quantitation was performed using the external standard technique.

The sensitivity of the Agilent 6470 MS/MS system allows for dilution factors up to 500-fold, which helps minimize matrix effects and reduces MS/MS source maintenance. The use of Agilent InfinityLab Poroshell column technology allows for UHPLC separation and run times with a standard HPLC system. The Agilent Infinity 1260

Multisampler Pretreatment option that sandwiches the sample between LC/MS/MS grade water, allows for improvement of peak shape for early eluting compounds. This option is helpful when analyzing a large list of compounds with a wide range of polarities. Using these techniques, an LOQ of 0.1 mg/kg was achieved for 89 % of the target list of over 140 compounds.

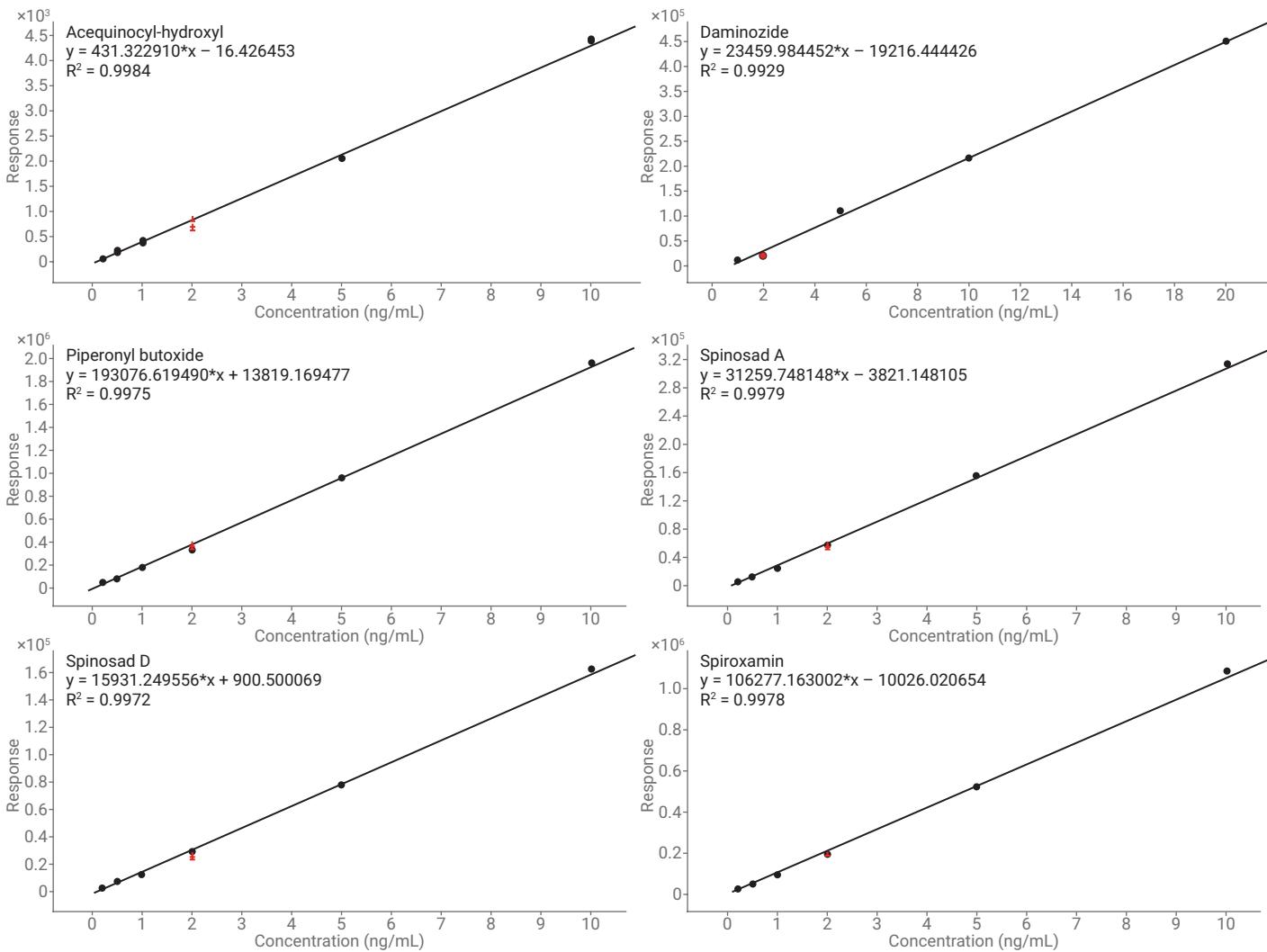


Figure 4. Select LC/MS/MS calibration curves (red triangles represent injected QCs).

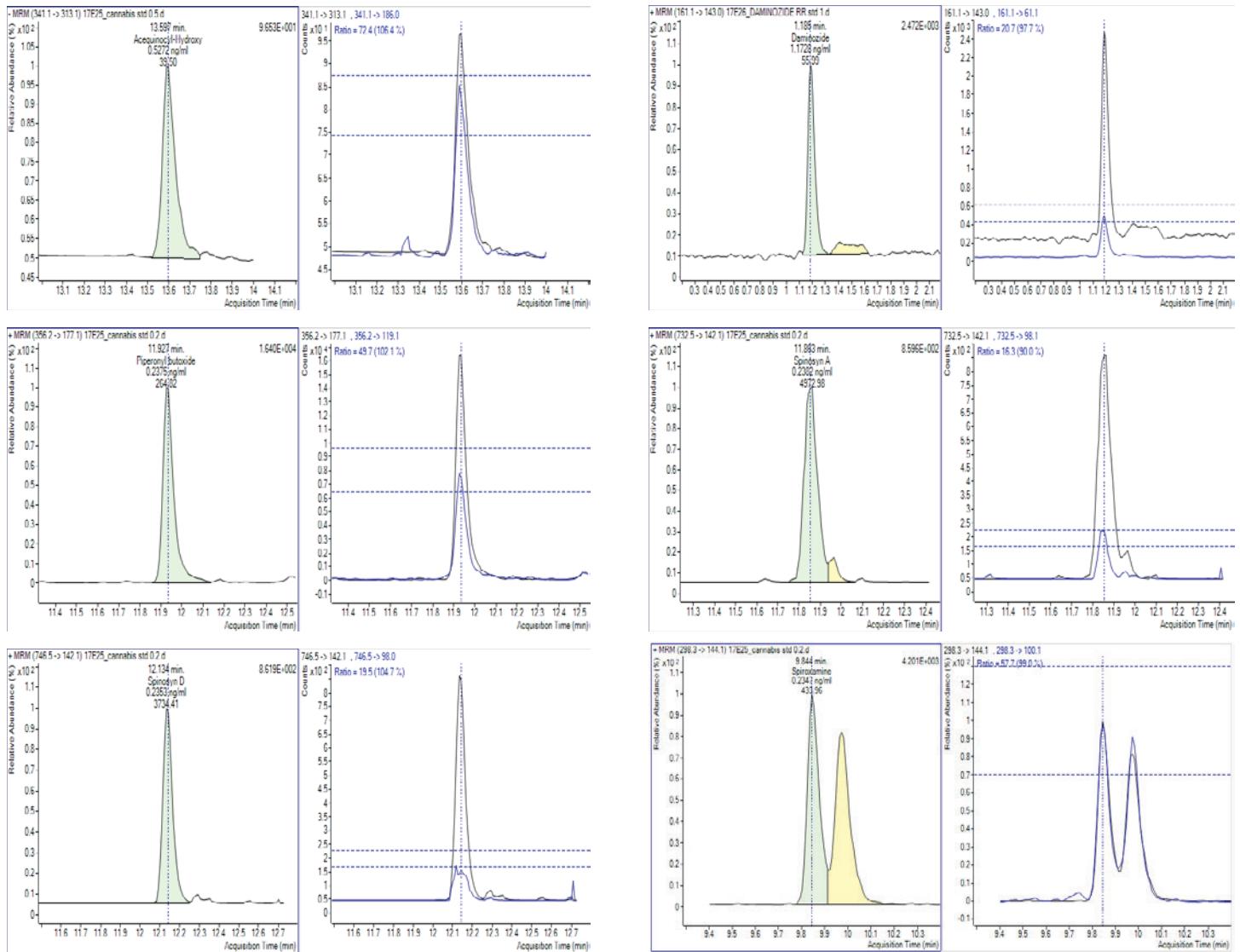


Figure 5. Select LC/MS/MS MRM chromatograms.

Table 7. Concentrations for illustrated LC/MS/MS chromatograms.

Compound	In-vial conc. (ng/mL)	In-sample conc. (mg/kg)
Acequinocyl hydroxide	0.5	0.25
Daminozide	1.0	0.5
Piperonyl butoxide	0.2	0.1
Spinosyn A	0.2	0.1
Spinosyn D	0.2	0.1
Spiroxamine	0.2	0.1

Table 8. LC/MS/MS recoveries and %RSD (n = 5 replicates). All compound listed in Table 7 had LOQs of 0.1 mg/kg, except for those given in Table 8.

Compound	% Recovery	%RSD
3-Hydroxy carbofuran	88	7.13
Abamectin	97	17.97
Acephate	86	8.75
Acequinocyl-hydroxy	87	9.31
Acetamiprid	84	5.36
Aldicarb	85	4.42
Aldicarb-sulfoxide	88	4.01
Allethrin	92	12.76
Ametoctradin	80	5.11
Atrazine	81	3.78
Azinphos-methyl	82	9.18
Azoxystrobin	88	6.27
Bendiocarb	82	9.04
Bensulide	88	9.33
Bifenazate	87	7.38
Famphur (Famophos)	107	7.06
Bromacil	85	8.18
Carbaryl	83	5.38
Carbendazim	82	5.04
Carbofuran	88	2.77
Carfentrazone-ethyl	88	5.58
Chlorantraniliprole	87	10.11
Clethodim	79	5.89
Clofentezine	82	5.92
Clothianidin	88	6.41
Cyanazine (Fortrol)	85	7.61
Cyantraniliprole	76	7.24
Cyazofamid	91	3.05
Cycloate	79	5.76
Cyflufenamid	80	12.18
Cyflumetofen	85	6.05
Cymoxanil (Curzate)	93	5.57
Cyprodinil	99	10.23
Cyromazine	73	11.48
Daminozide	95	6.72
DCPMU	83	3.64
Diazoxon	90	7.84
Diflubenzuron	78	8.52
Dimethoate	87	5.12
Dimethomorph(E)	86	8.33
Dimethomorph(Z)	83	9.83
Dinotefuran	87	5.70
Disulfoton-sulfon	87	8.46
Diuron	90	2.86
Etofenprox	83	4.59
Famphur (Famophos)	89	6.76
Fenamidone	87	10.77
Fenamiphos - sulfone	84	6.01
Compound	% Recovery	%RSD
Fenamiphos - sulfoxide	84	5.39
Fenazaquin	86	5.93
Fenbuconazole	85	5.78
Fenoxy carb	89	2.43
Fenpropothrin	80	12.53
Fenpyroximate	82	4.19
Flonicamid	86	4.94
Fluometuron	90	2.86
Fluopicolide	91	2.95
Fluopyram	87	5.43
Fluoxastrobin	83	8.64
Flupyradifurone	86	6.88
Fluridone	92	6.55
Flutriafol	84	4.13
Fluvalinate	91	5.54
Fluxapyroxad	83	9.33
Formentanate HCl	75	3.66
Hexazinone	87	4.64
Hexythiazox	78	4.18
Imazalil	83	8.80
Imidacloprid	90	5.84
Indaziflam	86	4.75
Indoxacarb	74	2.53
Isoxaben	92	5.35
Linuron	88	5.14
Malaxon	94	3.32
Mandipropamid	91	5.47
Metconazole	85	7.55
Methamidophos	89	3.39
Methidathion	81	3.97
Methiocarb	87	2.97
Methomyl	84	2.59
Methoxyfenozide	87	4.83
Metrafenone	81	9.32
Mevinphos	87	1.79
Norflurazon	89	2.56
Novaluron	83	5.51
Omethoate	87	4.52
Oxadixyl	91	5.30
Oxamyl	86	3.90
Oxydemeton-methyl	85	2.38
Penthiopyrad	87	7.73
Phorate sulfone	79	11.32
Phorate sulfoxide	89	2.21
Phosalone	105	19.24
Phosmet	104	12.38
Phosphamidon	83	2.90
Piperonyl butoxide	89	6.32

Compound	% Recovery	%RSD
Pirimicarb	89	3.08
Pirimiphos-methyl	88	6.56
Prallethrin	92	4.99
Prometon	84	3.27
Prometryn	83	3.89
Propamocarb	91	4.56
Propargite	88	3.90
Propazine	82	2.07
Propiconazole	84	12.93
Propoxur	82	8.26
Pymetrozine	80	4.26
Pyraclostrobin	84	6.35
Pyraflufen-ethyl	85	11.06
Pyrethrin I	88	4.83
Pyrethrin II	91	9.56
Pyridaben	84	4.43
Pyrimethanil	77	6.34
Rotenone	98	15.92
Sethoxydim	82	5.78
Siduron	86	4.87
Simazine	89	9.04
Simetryn	82	5.69
Spinetoram J	81	5.54
Spinetoram L	86	6.96
Spinosyn A	82	7.16
Spinosyn D	74	10.02
Spiromesifen	85	7.13
Spirotetramat	82	8.77
Spiroxamine	94	5.55
Sulfoxaflor	73	7.56
Tebuconazole	84	5.41
Tebufenoxide	82	6.92
Tebufenozide	82	6.92
Tebuthiuron	88	2.40
Terbutylazine	86	4.94
Terbutryn	82	3.47
Thiabendazole	76	3.81
Thiacloprid	86	3.03
Thiamethoxam	85	5.12
Thiobencarb	90	7.85
Thiodicarb	96	3.51
Tolfenpyrade	82	7.03
Triadimefon	103	20.12
Triadimenol	86	3.66
Trifloxystrobin	88	2.89
Triflumizole	85	3.25

Table 9. Compounds with LOQs different from those in Table 7.

LC-MS/MS	
Compound	LOQ (mg/kg)
Clethodim	0.2
Cycloate	0.2
Fenbuconazole	0.2
Flubendiamide	0.2
Methidathion	0.2
Propargite	0.2
Pyrethrin (I,II)	0.5
Sethoxydim	0.2
Spiromesifen	0.2
Tebuconazole	0.2
Triadimefon	0.2
Triadimenol	0.2
Trifloxystrobin	0.2

Conclusion

Though cannabis flower provides a challenging sample matrix, a comprehensive approach to residue analysis is possible. Using a simplified extraction technique with a pass-through SPE step can reduce the amount of co-extracted materials. Using dispersive cleanup techniques optimized for each analytical system reduce matrix effects, and increases the robustness of the analysis. Using high sensitivity mass spectrometers capable of low calibration levels (0.2–0.4 ng/mL) provides large dilution factors (up to 500-fold), again reducing matrix effects, instrument maintenance, and downtime. The success of this approach was confirmed by evaluating the % recovery and %RSDs for a large compound list.

Acknowledgements

The authors would like to thank Joan Stevens for her contributions to this work.

Reference

1. Hengel, M. J. Expanded Method Development for the Determination of Pesticides in Dried Hops by Liquid Chromatography with Tandem Mass Spectrometry. *J. Am. Soc. Brewing Chemists* **2011**, 69(3), 121–126.

Disclaimer

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law.

Appendix

Table A1. GC/MS/MS MRM Transitions.

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Dwell time (ms)	Collision energy
Dichlorovos	185	Unit	93.1	Unit	80.00	10
Dichlorovos	109	Unit	47.1	Unit	80.00	51
Etridiazole	213	Unit	185	Unit	40.00	10
Etridiazole	211	Unit	182.9	Unit	40.00	5
Dichlobenil	171	Unit	136	Unit	40.00	15
Dichlobenil	171	Unit	100	Unit	40.00	25
Trifluralin	306	Unit	264	Unit	13.00	5
Trifluralin	306	Unit	160	Unit	13.00	25
Benfluralin	292	Unit	264	Unit	13.00	4
Benfluralin	292	Unit	160.1	Unit	13.00	18
Ethalfluralin	276	Unit	202	Unit	13.00	12
Ethalfluralin	276	Unit	105	Unit	13.00	32
α-BHC	219	Unit	183	Unit	18.00	10
Chloropropham	213	Unit	171	Unit	13.00	5
Chloropropham	213	Unit	127	Unit	13.00	5
Chloroneb	191	Unit	141	Unit	13.00	10
Chloroneb	191	Unit	113	Unit	13.00	15
α-BHC	181	Unit	145	Unit	18.00	15
OPP	170	Unit	141.1	Unit	13.00	30
OPP	169	Unit	115.1	Unit	13.00	30
Ethoprop	158	Unit	114	Unit	13.00	5
Ethoprop	158	Unit	80.9	Unit	13.00	15
Hexachlorobenzene	284	Unit	248.8	Unit	18.00	25
Hexachlorobenzene	284	Unit	213.9	Unit	18.00	35
α-BHC	219	Unit	183	Unit	18.00	10
Chloropropham	213	Unit	171	Unit	18.00	5
Chloropropham	213	Unit	127	Unit	18.00	5
Ethoxyquin	202	Unit	174.3	Unit	18.00	15
Ethoxyquin	202	Unit	145.4	Unit	18.00	30
α-BHC	181	Unit	145	Unit	18.00	15
Ethoprop	158	Unit	114	Unit	18.00	5
Ethoprop	158	Unit	80.9	Unit	18.00	15
Diazinon	304	Unit	179	Unit	12.00	15
Diazinon	304	Unit	137	Unit	12.00	40
Hexachlorobenzene	284	Unit	248.8	Unit	12.00	25
Hexachlorobenzene	284	Unit	213.9	Unit	12.00	35
Dichlofenthion	279	Unit	205	Unit	12.00	32
Dichlofenthion	279	Unit	222.9	Unit	12.00	15
Heptachlor	272	Unit	236.9	Unit	12.00	15
Heptachlor	272	Unit	234.9	Unit	12.00	15
Pronamide	254	Unit	225.7	Unit	12.00	18
β-BHC	219	Unit	183	Unit	12.00	10
Dicloran	208	Unit	178	Unit	12.00	8
Dicloran	206	Unit	175.9	Unit	12.00	5

Table A1. GC/MS/MS MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Dwell time (ms)	Collision energy
Ethoxyquin	202	Unit	174.3	Unit	12.00	15
Ethoxyquin	202	Unit	145.4	Unit	12.00	30
2,6-Dichlorobenzamide	189	Unit	109	Unit	12.00	36
2,6-Dichlorobenzamide	189	Unit	74	Unit	12.00	36
β -BHC	181	Unit	145	Unit	12.00	15
Dithiopyr	354	Unit	306	Unit	12.00	6
Dithiopyr	354	Unit	286	Unit	12.00	12
Prodiamine	321	Unit	279	Unit	12.00	5
Prodiamine	321	Unit	215.9	Unit	12.00	15
Dichlofenthion	279	Unit	205	Unit	12.00	32
Dichlofenthion	279	Unit	222.9	Unit	12.00	15
Heptachlor	272	Unit	236.9	Unit	12.00	15
Heptachlor	272	Unit	234.9	Unit	12.00	15
Chlorothalonil	266	Unit	231	Unit	12.00	15
Chlorothalonil	264	Unit	229	Unit	12.00	15
Pirimacarb	238	Unit	166	Unit	12.00	10
γ -BHC	219	Unit	183	Unit	12.00	10
γ -BHC	181	Unit	145	Unit	12.00	15
Pirimacarb	166	Unit	96	Unit	12.00	15
Dithiopyr	354	Unit	306	Unit	8.00	6
Dithiopyr	354	Unit	286	Unit	8.00	12
Prodiamine	321	Unit	279	Unit	8.00	5
Prodiamine	321	Unit	215.9	Unit	8.00	15
Pentachlorothioanisole	296	Unit	262.9	Unit	8.00	15
Pentachlorothioanisole	296	Unit	245.8	Unit	8.00	40
Methyl chlopyrifos	286	Unit	207.9	Unit	8.00	18
Methyl chlopyrifos	286	Unit	93	Unit	8.00	18
Heptachlor	272	Unit	236.9	Unit	8.00	15
Heptachlor	272	Unit	234.9	Unit	8.00	15
Chlorothalonil	266	Unit	231	Unit	8.00	15
Chlorothalonil	264	Unit	229	Unit	8.00	15
Parathion methyl	263	Unit	109	Unit	8.00	14
Mefenoxam	249	Unit	190	Unit	8.00	5
Mefenoxam	249	Unit	146	Unit	8.00	20
Pirimacarb	238	Unit	166	Unit	8.00	10
Parathion methyl	233	Unit	124	Unit	8.00	10
δ -BHC	181	Unit	145	Unit	8.00	15
Pirimacarb	166	Unit	96	Unit	8.00	15
Metolachlor	162	Unit	133.1	Unit	8.00	15
Metolachlor	162	Unit	132.1	Unit	8.00	25
Dacthal	332	Unit	300.9	Unit	9.00	10
Dacthal	301	Unit	222.9	Unit	9.00	20
Aldrin	298	Unit	263	Unit	9.00	8
Pentachlorothioanisole	296	Unit	262.9	Unit	9.00	15
Pentachlorothioanisole	296	Unit	245.8	Unit	9.00	40
Ethofumesate	286	Unit	207	Unit	9.00	5
Ethofumesate	286	Unit	178.9	Unit	9.00	15

Table A1. GC/MS/MS MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Dwell time (ms)	Collision energy
Aldrin	263	Unit	193	Unit	9.00	30
Parathion methyl	263	Unit	109	Unit	8.00	14
Mefenoxam	249	Unit	190	Unit	9.00	5
Mefenoxam	249	Unit	146	Unit	9.00	20
Parathion methyl	233	Unit	124	Unit	8.00	10
Chlopyrifos	197	Unit	168.9	Unit	9.00	15
Chlopyrifos	197	Unit	107	Unit	9.00	40
Malathion	173	Unit	127	Unit	9.00	4
Malathion	173	Unit	99	Unit	9.00	15
Metolachlor	162	Unit	133.1	Unit	9.00	15
Metolachlor	162	Unit	132.1	Unit	9.00	25
Fipronil	367	Unit	228	Unit	10.00	30
Fipronil	367	Unit	213	Unit	10.00	30
Tetraconazole	336	Unit	218	Unit	10.00	20
Tetraconazole	336	Unit	156	Unit	10.00	34
Dacthal	332	Unit	300.9	Unit	10.00	10
Dacthal	301	Unit	222.9	Unit	10.00	20
Pentachlorothioanisole	296	Unit	262.9	Unit	10.00	15
Pentachlorothioanisole	296	Unit	245.8	Unit	10.00	40
Ethofumesate	286	Unit	207	Unit	10.00	5
Ethofumesate	286	Unit	178.9	Unit	10.00	15
Pendimethalin	281	Unit	252	Unit	10.00	2
Parathion methyl	263	Unit	109	Unit	10.00	14
Pendimethalin	252	Unit	162.1	Unit	10.00	10
Dicofol	250	Unit	139	Unit	10.00	16
Dicofol	250	Unit	111	Unit	10.00	42
Parathion methyl	233	Unit	124	Unit	10.00	10
Chlopyrifos	197	Unit	168.9	Unit	10.00	15
Chlopyrifos	197	Unit	107	Unit	10.00	40
MGK 264	164	Unit	98.1	Unit	10.00	6
MGK 264	164	Unit	80.1	Unit	10.00	30
Heptachlor epoxide	353	Unit	316.9	Unit	14.00	15
Heptachlor epoxide	353	Unit	262.9	Unit	14.00	15
Tetraconazole	336	Unit	218	Unit	14.00	20
Tetraconazole	336	Unit	156	Unit	14.00	34
Procymidone	283	Unit	96	Unit	14.00	10
Procymidone	283	Unit	67.1	Unit	14.00	40
Pendimethalin	281	Unit	252	Unit	14.00	2
Pendimethalin	252	Unit	162.1	Unit	14.00	10
Dicofol	250	Unit	139	Unit	14.00	16
Dicofol	250	Unit	111	Unit	14.00	42
Diphenamid	239	Unit	167	Unit	14.00	0
Diphenamid	167	Unit	165	Unit	14.00	20
MGK 264	164	Unit	98.1	Unit	14.00	6
MGK 264	164	Unit	80.1	Unit	14.00	30
<i>trans</i> -Nonachlor	409	Unit	299.9	Unit	14.00	25

Table A1. GC/MS/MS MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Dwell time (ms)	Collision energy
Chlordane <i>cis/trans</i>	375	Unit	266	Unit	14.00	25
Oxadiazon	302	Unit	175	Unit	14.00	13
Procymidone	283	Unit	96	Unit	14.00	10
Procymidone	283	Unit	67.1	Unit	14.00	40
Diphenamid	239	Unit	167	Unit	14.00	0
Paclobutrazole	238	Unit	127	Unit	14.00	15
Paclobutrazole	236	Unit	125	Unit	14.00	15
Oxadiazon	175	Unit	112	Unit	14.00	15
Diphenamid	167	Unit	165	Unit	14.00	20
Captan	149	Unit	70	Unit	14.00	15
Chlordane <i>cis/trans</i>	375	Unit	266	Unit	18.00	25
Flutolanil	323	Unit	173	Unit	18.00	13
Oxadiazon	302	Unit	175	Unit	18.00	13
Oxyfluorfen	300	Unit	223	Unit	18.00	20
Oxyfluorfen	252	Unit	196	Unit	18.00	20
<i>p,p'</i> -DDE	246	Unit	176	Unit	18.00	30
Paclobutrazole	238	Unit	127	Unit	18.00	15
Paclobutrazole	236	Unit	125	Unit	18.00	15
Oxadiazon	175	Unit	112	Unit	18.00	15
Flutolanil	173	Unit	145	Unit	18.00	15
Captan	149	Unit	70	Unit	18.00	15
<i>cis</i> -Nonachlor	409	Unit	109	Unit	20.00	20
Oxyfluorfen	300	Unit	223	Unit	20.00	20
Oxyfluorfen	252	Unit	196	Unit	20.00	20
Chlorfenapyr	249	Unit	112	Unit	20.00	30
Fludioxonil	248	Unit	182	Unit	20.00	15
Fludioxonil	248	Unit	127	Unit	20.00	30
Chlorfenapyr	247	Unit	227	Unit	20.00	20
Kresoxim-methyl	206	Unit	131	Unit	20.00	10
Kresoxim-methyl	206	Unit	116	Unit	20.00	5
Myclobutanil	179	Unit	125.1	Unit	20.00	15
Myclobutanil	179	Unit	90	Unit	20.00	40
<i>cis</i> Nonachlor	409	Unit	109	Unit	40.00	20
Bolstar	322	Unit	198	Unit	40.00	5
Bolstar	322	Unit	156	Unit	40.00	5
<i>p,p'</i> -DDD	235	Unit	200	Unit	40.00	8
<i>p,p'</i> -DDT	235	Unit	165.1	Unit	40.00	25
<i>cis</i> -Nonachlor	409	Unit	109	Unit	30.00	20
Bolstar	322	Unit	198	Unit	30.00	5
Bolstar	322	Unit	156	Unit	30.00	5
Quinoxifen	307	Unit	272	Unit	30.00	5
Quinoxifen	237	Unit	208	Unit	30.00	32
<i>p,p'</i> -DDD	235	Unit	200	Unit	30.00	8
<i>p,p'</i> -DDT	235	Unit	165.1	Unit	30.00	25
Etoxazole	359	Unit	187	Unit	18.00	15
Bromopropylate	341	Unit	185	Unit	18.00	20

Table A1. GC/MS/MS MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Dwell time (ms)	Collision energy
Bromopropylate	341	Unit	183	Unit	18.00	20
d-TPP	341	Unit	180.1	Unit	18.00	22
Diclofop methyl	340	Unit	254	Unit	18.00	14
Diclofop methyl	340	Unit	253	Unit	18.00	14
Quinoxifen	307	Unit	272	Unit	18.00	5
Etoxazole	300	Unit	270	Unit	18.00	20
Quinoxifen	237	Unit	208	Unit	18.00	32
Bifenthrin	181	Unit	166.2	Unit	18.00	10
Bifenthrin	181	Unit	165.2	Unit	18.00	25
Etoxazole	359	Unit	187	Unit	30.00	15
Etoxazole	300	Unit	270	Unit	30.00	20
Pyriproxyfen	226	Unit	186.1	Unit	30.00	14
Cyhalothrin	208	Unit	181.1	Unit	30.00	4
Cyhalothrin	197	Unit	161.1	Unit	30.00	4
Pyriproxyfen	136	Unit	96	Unit	30.00	15
Fenarimol	330	Unit	139	Unit	25.00	15
Spirodiclofen	314	Unit	109	Unit	25.00	16
Spirodiclofen	312	Unit	259.1	Unit	25.00	10
Pyriproxyfen	226	Unit	186.1	Unit	25.00	14
Permethrin	165	Unit	129	Unit	25.00	5
Permethrin	163	Unit	127	Unit	25.00	5
Fenarimol	139	Unit	75	Unit	25.00	35
Pyriproxyfen	136	Unit	96	Unit	25.00	15
Cyfluthrin	163	Unit	127	Unit	200.00	5
Cypermethrin	163	Unit	127	Unit	200.00	5
Fenvalerate	419	Unit	225.1	Unit	60.00	5
Esfenvalerate	419	Unit	225.1	Unit	60.00	5
Esfenvalerate	419	Unit	167.1	Unit	60.00	5
Deltamethrin	253	Unit	172	Unit	100.00	10
Deltamethrin	253	Unit	93	Unit	100.00	20

Table A2. LC/MS/MS Dynamic MRM Transitions.

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
3-Hydroxy carbofuran	238.11	Unit	163.08	Unit	80	10
Abamectin	895.5	Unit	751.4	Unit	170	42
Abamectin	895.5	Unit	449.2	Unit	170	46
Acephate	184	Unit	143	Unit	70	0
Acephate	184	Unit	125	Unit	70	15
Acequinocyl-hydroxy*	341.1	Unit	313.1	Unit	90	32
Acequinocyl-hydroxy*	341.1	Unit	186	Unit	90	30
Acetamiprid	223.1	Unit	99	Unit	80	40
Acetamiprid	223.1	Unit	56.1	Unit	80	12
Aldicarb	208.1	Unit	116.2	Unit	70	0
Aldicarb	208.1	Unit	89.1	Unit	70	12
Aldicarb-sulfone (Aldoxycarb)	223.1	Unit	166	Unit	80	0
Aldicarb-sulfone (Aldoxycarb)	223.1	Unit	86.1	Unit	80	8
Aldicarb-sulfoxide	207.1	Unit	131.9	Unit	65	0
Aldicarb-sulfoxide	207.1	Unit	105.2	Unit	65	4
Allethrin	303.2	Unit	135	Unit	60	10
Allethrin	303.2	Unit	123	Unit	60	20
Ametoctradin	276.2	Unit	176.1	Unit	175	48
Ametoctradin	276.2	Unit	149.1	Unit	175	44
Atrazine	216.1	Unit	104	Unit	125	28
Atrazine	216.1	Unit	68	Unit	125	40
Azinphos-methyl	318	Unit	261	Unit	60	0
Azinphos-methyl	318	Unit	125	Unit	60	15
Azoxystrobin	404.1	Unit	372.1	Unit	110	8
Azoxystrobin	404.1	Unit	156	Unit	110	56
Bendiocarb	224.1	Unit	109.1	Unit	80	12
Bensulide	398.07	Unit	158	Unit	80	20
Bifenazate	301.1	Unit	198.2	Unit	95	4
Bifenazate	301.1	Unit	170.1	Unit	95	16
Boscalid	343	Unit	307.1	Unit	145	16
Boscalid	343	Unit	271.2	Unit	145	32
Bromacil	261	Unit	205	Unit	70	20
Carbaryl	202.1	Unit	145.1	Unit	65	4
Carbaryl	202.1	Unit	127.1	Unit	65	28
Carbendazim	192.1	Unit	160.1	Unit	105	16
Carbendazim	192.1	Unit	132.1	Unit	105	32
Carbofuran	222.1	Unit	123.1	Unit	80	30
Carfentrazone-ethyl	412.1	Unit	365.9	Unit	150	12
Carfentrazone-ethyl	412.1	Unit	345.9	Unit	150	20
Chlorantraniliprole	483.9	Unit	452.9	Unit	105	16
Chlorantraniliprole	483.9	Unit	285.9	Unit	105	8
Cinerin I	317.2	Unit	149	Unit	120	10
Cinerin I	317.2	Unit	106.9	Unit	120	10
Clethodim	360.1	Unit	268.1	Unit	100	8
Clethodim	360.1	Unit	164.1	Unit	100	16
Clofentezine	303	Unit	138	Unit	110	12
Clofentezine	303	Unit	102	Unit	110	40

Table A2. LC/MS/MS Dynamic MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
Clothianidin	250.02	Unit	169	Unit	95	8
Clothianidin	250.02	Unit	131.9	Unit	95	8
Cyanazine (Fortrol)	241.1	Unit	214.1	Unit	120	18
Cyantraniliprole	473.1	Unit	441.9	Unit	117	10
Cyantraniliprole	473.1	Unit	283.9	Unit	117	6
Cyazofamid	325	Unit	108	Unit	90	8
Cycloate	216.1	Unit	134.1	Unit	90	10
Cyflufenamid	413.1	Unit	295	Unit	105	6
Cyflufenamid	413.1	Unit	241	Unit	105	18
Cyflumetofen	465.5	Unit	249.1	Unit	102	6
Cyflumetofen	465.5	Unit	173	Unit	102	18
Cymoxanil (Curzate)	199.1	Unit	128	Unit	65	5
Cymoxanil (Curzate)	199.1	Unit	83	Unit	50	20
Cyprodinil	226.1	Unit	133.1	Unit	140	24
Cyprodinil	226.1	Unit	92.9	Unit	140	40
Cyromazine	167.2	Unit	85.1	Unit	140	18
Daminozide	161.1	Unit	143	Unit	87	10
Daminozide	161.1	Unit	61.1	Unit	87	10
DCPMU	219	Unit	126.9	Unit	109	26
Diazoxon	289.2	Unit	233	Unit	115	14
Difenconazole	406.1	Unit	265	Unit	120	40
Difenconazole	406.1	Unit	251	Unit	120	20
Diflubenzuron	311	Unit	158	Unit	101	10
Diflubenzuron	311	Unit	141	Unit	80	32
Dimethoate	230	Unit	198.8	Unit	70	0
Dimethoate	230	Unit	125	Unit	70	16
Dimethomorph	388.1	Unit	301.1	Unit	145	20
Dimethomorph	388.1	Unit	165.1	Unit	145	32
Dinotefuran	203.1	Unit	129	Unit	75	6
Disulfoton-sulfon	307.1	Unit	96.9	Unit	130	30
Diuron	233.03	Unit	72.1	Unit	110	20
Diuron	233.03	Unit	46.1	Unit	110	16
d-Phenothrin	351.1	Unit	183	Unit	120	14
d-Phenothrin	351.1	Unit	128.6	Unit	120	46
Etofenprox	394.5	Unit	177	Unit	111	14
Etofenprox	394.5	Unit	107	Unit	111	50
Famphur (Famophos)	326	Unit	281	Unit	125	10
Fenamidone	312	Unit	236.1	Unit	100	8
Fenamidone	312	Unit	92.2	Unit	100	28
Fenamiphos-sulfone	336.1	Unit	266	Unit	115	16
Fenamiphos-sulfoxide	320.11	Unit	233	Unit	130	20
Fenazaquin	307.2	Unit	161.1	Unit	105	10
Fenbuconazole	337.1	Unit	125.1	Unit	145	40
Fenoxy carb	302	Unit	115.9	Unit	107	10
Fenoxy carb	302	Unit	88	Unit	107	22
Fenpropathrin	350.2	Unit	125.1	Unit	115	10

Table A2. LC/MS/MS Dynamic MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
Fenpropothrin	350.2	Unit	55.1	Unit	115	48
Fenpyroximate	422.21	Unit	366.2	Unit	135	12
Fenpyroximate	422.21	Unit	231.1	Unit	135	24
Flonicamid	230.1	Unit	203	Unit	110	15
Flonicamid	230.1	Unit	174	Unit	110	15
Flumioxazin	355.2	Unit	299	Unit	140	25
Flumioxazin	355.2	Unit	147.7	Unit	140	33
Fluometuron	233.1	Unit	72	Unit	105	16
Fluometuron	233.1	Unit	46.1	Unit	105	16
Fluopicolide	382.9	Unit	172.9	Unit	110	20
Fluopicolide	382.9	Unit	144.9	Unit	110	56
Fluopyram	397.1	Unit	207.9	Unit	135	18
Fluopyram	397.1	Unit	172.9	Unit	135	26
Fluoxastrobin	459	Unit	188.1	Unit	130	36
Fluoxastrobin	459	Unit	111	Unit	130	60
Flupyradifurone	289.2	Unit	126	Unit	100	18
Flupyradifurone	289.2	Unit	90.1	Unit	100	46
Fluridone	330.1	Unit	259	Unit	120	55
Flutriafol	302.2	Unit	94.9	Unit	110	50
Flutriafol	302.2	Unit	70	Unit	110	14
Fluvalinate	503.13	Unit	208	Unit	100	5
Fluxapyroxad	382.1	Unit	362.1	Unit	115	6
Fluxapyroxad	382.1	Unit	234.1	Unit	115	18
Formentanate HCL	167.1	Unit	110.1	Unit	57	6
Formentanate HCL	167.1	Unit	93	Unit	57	26
Hexazinone	253.2	Unit	171.1	Unit	120	20
Hexazinone	253.2	Unit	71.1	Unit	120	40
Hexythiazox	353.1	Unit	271	Unit	90	8
Hexythiazox	353.1	Unit	168.1	Unit	90	24
Imazalil	297.1	Unit	159	Unit	115	20
Imazalil	297.1	Unit	69	Unit	115	16
Imidacloprid	256	Unit	208.9	Unit	80	12
Imidacloprid	256	Unit	175	Unit	80	12
Indaziflam	302.3	Unit	158.1	Unit	103	13
Indaziflam	302.3	Unit	138	Unit	103	25
Indoxacarb	528.1	Unit	248.8	Unit	110	12
Indoxacarb	528.1	Unit	150	Unit	110	20
Iprodione	330.04	Unit	244.99	Unit	120	10
Iprodione	330	Unit	287.8	Unit	105	6
Isoxaben	333.2	Unit	165.1	Unit	100	16
Isoxaben	333.2	Unit	150	Unit	100	8
Jasmolin I	331.2	Unit	161.1	Unit	120	10
Jasmolin I	331.2	Unit	131.2	Unit	120	40
Jasmolin I	331.2	Unit	79.1	Unit	120	40
Linuron	249.02	Unit	182.3	Unit	100	8

Table A2. LC/MS/MS Dynamic MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
Linuron	249.02	Unit	160.1	Unit	100	20
Malaxon	315.1	Unit	126.9	Unit	107	6
Mandipropamid	412.13	Unit	328.1	Unit	110	8
Mandipropamid	412.13	Unit	125	Unit	110	40
Metconazole	320.1	Unit	125	Unit	130	48
Metconazole	320.1	Unit	70.1	Unit	130	24
Methamidophos	142	Unit	125	Unit	85	10
Methamidophos	142	Unit	94	Unit	85	10
Methidathion	302.9	Unit	85.1	Unit	55	15
Methiocarb (Mercaptodimethylur)	226.1	Unit	169	Unit	70	4
Methiocarb (Mercaptodimethylur)	226.1	Unit	121.1	Unit	70	12
Methomyl	163.1	Unit	106	Unit	50	4
Methomyl	163.1	Unit	88	Unit	50	0
Methoxyfenozide	369.2	Unit	149.2	Unit	85	12
Methoxyfenozide	369.2	Unit	133	Unit	85	24
Metrafenone	409.1	Unit	226.9	Unit	110	16
Metrafenone	409.1	Unit	209.1	Unit	110	8
Mevinphos (Phosdrin)	225	Unit	127	Unit	65	12
Mevinphos (Phosdrin)	225	Unit	109	Unit	65	32
Norflurazon	304	Unit	284	Unit	120	20
Norflurazon	304	Unit	140	Unit	120	40
Novaluron	493	Unit	158.1	Unit	90	16
Novaluron	493	Unit	141.1	Unit	90	56
Omethoate	214	Unit	125	Unit	80	16
Oxadixyl	279.1	Unit	219.2	Unit	70	5
Oxadixyl	279.1	Unit	132.3	Unit	70	32
Oxamyl	237.1	Unit	90	Unit	60	0
Oxamyl	237.1	Unit	72	Unit	60	12
Oxydemeton-methyl	247.1	Unit	125	Unit	84	18
Oxydemeton-methyl	247.1	Unit	105.1	Unit	84	6
Pacllobutrazol	294.8	Unit	125.3	Unit	118	46
Pacllobutrazol	294.8	Unit	70	Unit	118	26
Penthiopyrad	360.1	Unit	275.9	Unit	113	6
Penthiopyrad	360.1	Unit	255.9	Unit	113	14
Phorate sulfone	293.01	Unit	171	Unit	60	5
Phorate sulfone	293.01	Unit	143	Unit	60	15
Phorate sulfoxide	277.02	Unit	97	Unit	80	35
Phosalone	368	Unit	182	Unit	70	8
Phosalone	368	Unit	110.9	Unit	70	44
Phosmet	317.99	Unit	160	Unit	70	8
Phosmet	317.99	Unit	133	Unit	70	36
Phosphamidon	300	Unit	174.1	Unit	110	8
Phosphamidon	300	Unit	127.1	Unit	110	16
Piperonyl butoxide	356.2	Unit	177.1	Unit	80	4
Piperonyl butoxide	356.2	Unit	119.1	Unit	80	40
Pirimicarb	239.15	Unit	72.1	Unit	100	20

Table A2. LC/MS/MS Dynamic MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
Pirimiphos-methyl	306.3	Unit	164	Unit	120	18
Pirimiphos-methyl	306.3	Unit	108	Unit	120	30
Prallethrin	301.18	Unit	169	Unit	80	5
Prallethrin	301.18	Unit	105	Unit	80	20
Prometon	226.2	Unit	142.1	Unit	120	24
Prometryn	242.1	Unit	200.1	Unit	120	20
Prometryn	242.1	Unit	158	Unit	120	28
Propamocarb	189.2	Unit	144	Unit	90	8
Propamocarb	189.2	Unit	102	Unit	90	12
Propargite	368.1	Unit	231.2	Unit	80	0
Propargite	368.1	Unit	175.2	Unit	80	8
Propazine	230.1	Unit	188.1	Unit	100	28
Propazine	230.1	Unit	146	Unit	100	22
Propiconazole	342.1	Unit	123	Unit	115	60
Propiconazole	342.1	Unit	69.1	Unit	115	16
Propoxur	210.11	Unit	168.1	Unit	55	0
Pymetrozine	218.11	Unit	105	Unit	110	20
Pyraclostrobin	388.11	Unit	193.8	Unit	95	8
Pyraclostrobin	388.11	Unit	163.1	Unit	95	20
Pyraflufen-ethyl	413	Unit	339	Unit	120	25
Pyrethrin I	329.21	Unit	161	Unit	100	5
Pyrethrin I	329.21	Unit	143	Unit	100	20
Pyrethrin II	373.1	Unit	161.1	Unit	102	2
Pyridaben	365.1	Unit	309.1	Unit	80	4
Pyridaben	365.1	Unit	147.2	Unit	80	20
Pyrimethanil	200.1	Unit	107	Unit	120	26
Pyrimethanil	200.1	Unit	82	Unit	120	30
Rotenone	395	Unit	192.1	Unit	145	20
Rotenone	395	Unit	139.1	Unit	145	28
Saflufenacil	501.2	Unit	459	Unit	165	6
Saflufenacil	501.2	Unit	348.9	Unit	165	22
Sethoxydim	328.2	Unit	178.1	Unit	120	16
Siduron	233.2	Unit	137.1	Unit	115	12
Siduron	233.2	Unit	94	Unit	115	20
Simazine	202.1	Unit	166.1	Unit	120	20
Simazine	202.1	Unit	96.1	Unit	120	20
Simetryn	214.11	Unit	124.09	Unit	120	20
Simetryn	214.11	Unit	96	Unit	120	20
Spinetoram J	748.5	Unit	142	Unit	165	26
Spinetoram J	748.5	Unit	98.1	Unit	165	50
Spinetoram L	760.5	Unit	142	Unit	165	26
Spinetoram L	760.5	Unit	98.1	Unit	165	50
Spinosyn A	732.5	Unit	142.1	Unit	155	28
Spinosyn A	732.5	Unit	98.1	Unit	155	60
Spinosyn D	746.5	Unit	142.1	Unit	145	35
Spinosyn D	746.5	Unit	98	Unit	145	55

Table A2. LC/MS/MS Dynamic MRM Transitions (continued).

Compound	Precursor ion	MS1 Resolution	Product ion	MS2 Resolution	Fragmentor	Collision energy
Spiromesifen	388	Unit	273	Unit	110	10
Spiromesifen	371.2	Unit	255.1	Unit	120	24
Spirotetramat	374.2	Unit	330.2	Unit	120	12
Spirotetramat	374.2	Unit	302.2	Unit	120	12
Spiroxamine	298.28	Unit	144.1	Unit	125	16
Spiroxamine	298.28	Unit	100.1	Unit	125	32
Sulfoxaflor	278.2	Unit	174.1	Unit	74	2
Sulfoxaflor	278.2	Unit	154.1	Unit	74	26
Tebuconazole	308.1	Unit	124.9	Unit	100	47
Tebuconazole	308.1	Unit	70	Unit	100	40
Tebufenoxide	353.2	Unit	297.1	Unit	95	0
Tebufenoxide	353.2	Unit	297.1	Unit	95	0
Tebufenoxide	353.2	Unit	102.9	Unit	95	60
Tebuthiuron	229.1	Unit	172.1	Unit	105	12
Terbutylazine	230.1	Unit	174.1	Unit	70	15
Terbutylazine	230.1	Unit	132	Unit	70	25
Terbutryn	242.1	Unit	186.1	Unit	110	16
Terbutryn	242.1	Unit	71.1	Unit	110	32
Thiabendazole	202	Unit	175	Unit	130	24
Thiabendazole	202	Unit	131	Unit	130	36
Thiacloprid	253	Unit	126	Unit	100	16
Thiacloprid	253	Unit	73	Unit	100	60
Thiamethoxam	292.03	Unit	211.1	Unit	85	8
Thiamethoxam	292.03	Unit	181.1	Unit	85	20
Thiobencarb	258.1	Unit	89.1	Unit	92	50
Thiobencarb	258.07	Unit	125.1	Unit	100	25
Thiodicarb	355.06	Unit	108.1	Unit	85	8
Tolfenpyrade	384.1	Unit	197	Unit	100	20
Tolfenpyrade	384.1	Unit	145	Unit	120	22
Triadimefon	294.1	Unit	225.1	Unit	90	20
Triadimefon	294.1	Unit	197.2	Unit	90	8
Triadimenol	296.1	Unit	70	Unit	70	8
Trifloxystrobin	409.1	Unit	186	Unit	110	12
Triflumizol	346.1	Unit	73.2	Unit	85	12
3-Hydroxy carbofuran	238.11	Unit	181.09	Unit	80	10

* Electrospray negative mode

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