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Comprehensive LC-MS/MS Multi-Residue Pesticide Method for Food and Feed-Standardization With Quality Control Materials

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Abstract

To comply with strict regulations, hundreds of pesticide residues must be monitored using reliable analytical methods for their detection, quantification, and identification in many different food and feed commodities. One cost effective approach is to add more residues to a single method, but this requires generic sample preparation and the injection and analysis of more complex extracts. More residues can also mean that users spend more time data processing and reviewing results.

Herein we describe a LC-MS/MS multi-residue pesticide method for more than 200 residues using the ACQUITY™ Premier System, Xevo™ TQ-S micro Triple Quadrupole Mass Spectrometer and waters_connect™ for Quantitation Software to check compliance against commercially available quality control materials of strawberry purée, baby food, and animal feed.

The food and feed commodities were extracted using QuEChERS with the baby food and feed samples cleaned up using a simple and effective Oasis™ PRiME HLB Plus pass through protocol. The 1 µL acetonitrile extract was injected without dilution using a post injector extension loop which significantly improved the peak shape of

early eluting compounds and removed the need to manually reintegrate peaks.

Exception focused review in waters_connect for Quantitation Software highlighted results that fell outside SANTE tolerances defined by a ruleset, increasing the efficiency of data review.

Method performance was assessed using quality control (QC) materials with all measured values within the assigned range of the analytes, trueness was in the range of 100–130% and all calculated %RSDs less than 20%. The robustness of the method was demonstrated for animal feed extracts with peak response %RSDs all less than 9% for 250 injections.

Benefits

- Cost effective approach to monitoring multi-residue pesticides in food and feed using LC-MS/MS which met SANTE guidelines for method performance
- No dilution of final extract required as 1 µL injection of acetonitrile extract using a post injector extension loop improved early eluting peak shapes, removing the need for manual peak re-integrations
- Exception focused review in waters_connect for Quantitation Software increased the efficiency of data review

Introduction

Plant protection products, commonly known as pesticides, are essential to the food and agricultural industries, helping to stabilize food supplies by controlling pests, weeds, and disease. Pesticide residues resulting from the use of plant protection products during planting, storage or production may pose a risk to public health or hinder trade due to rejections of imported consignments and product recalls.

Hundreds of pesticides are approved and routinely used for crop protection and regulations are in place for maximum residue levels (MRL), that are legally tolerated in food and feed when pesticides are applied correctly in accordance with Good Agricultural Practice. Excessive use of pesticides may result in residues that exceed the MRLs if the pre-harvest intervals and correct dosages are not adhered to by growers or illegal pesticides are used. Compliance with these MRLs is checked by competent authorities by monitoring for residues in food and feed samples. All food business operators must ensure compliance with the same requirements but also must consider brand protection too. Pesticides that have not been authorized for use on crops generally have a default MRL value of 0.01 mg/kg.

Hence, reliable analytical methods are needed for detection, quantification, and identification of hundreds of pesticide residues in many different commodities. A primary goal for all laboratories involved in pesticide residue testing is to accurately determine as many compounds as possible in the most cost-effective manner. By implementing multi-residue methods using Ultra Performance Liquid Chromatography Tandem Quadrupole Mass Spectrometry (UPLC™-MS/MS) systems, many labs have significantly extended their scope of analysis to meet the demand for testing. Nevertheless, multi-residue methods often employ QuEChERS for sample preparation and the injection of more complex extracts so the ability for systems to provide accurate and precise results over extended time periods is another important goal for these labs.

Herein we describe a QuEChERS extraction followed by determination with LC-MS/MS for multi-residue pesticide analysis using commercially available quality control materials of strawberry purée, baby food, and animal feed as proof of performance for this method approach.

Experimental

Sample Description

Quality Control Materials (T19380QC strawberry purée, T09160QC cereal-based baby food, and T09151QC cereal-based animal feed) were obtained from FAPAS.

Pesticide standards (31971 LC Multiresidue Pesticide Standards Kit) were purchased from Restek and serial diluted with extracted blank matrix into the sample concentration calibration range 0.005–0.2 mg/kg.

Samples were extracted using a previously published QuEChERS CEN method with DisQue (p/n: 186006813 < https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/186006813-disque-1-g-trisodium-citrate-dihydrate-05-g-disodium-hydrogencit.html>).[1] In short, 10 g strawberry purée, 5 g baby food, and 2 g animal feed were taken through the method. Baby food and animal feed were subjected to a pass-through clean-up using Oasis PRiME HLB Plus Short (p/n: 186008887 < https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/186008887-oasis-prime-hlb-plus-short-cartridge-335-mg-sorbent-per-cartridg.html>) cartridges. The pure acetonitrile extracts were injected without dilution using the bracketed calibration approach. Five replicates of each QC material were analyzed using this method. <https://www.waters.com/nextgen/global/shop/sample-preparation--filtration/186008887-oasis-prime-hlb-plus-short-cartridge-335-mg-sorbent-per-cartridg.html>

LC Conditions

LC system:	ACQUITY Premier System including Sample Organizer
Vials:	LC-MS Certified Clear Glass 12 x 32 mm Screw Neck Max Recovery Vial (p/n: 600000670CV)
Column(s):	ACQUITY Premier HSS T3, 2.1 x 100 mm, 1.8 µm (p/n: 186009468)
Column temperature:	40 °C
Sample temperature:	6 °C
Injection volume:	1 µL using 50 µL extension loop fitted post injector (p/n: 430002012)
Mobile phase A:	Water+0.1% formic acid and 5 mM ammonium formate
Mobile phase B:	Methanol and acetonitrile (1:1)+0.1% formic acid and 5 mM ammonium formate

Gradient Table

Time (min)	Flow (mL/min)	%A	%B	Curve
0	0.5	99	1	Initial
0.5	0.5	99	1	6
3.5	0.5	60	40	6
12.5	0.5	15	85	6
12.6	0.5	1	99	6
15	0.5	1	99	6
15.1	0.5	99	1	6
19	0.5	99	1	6

MS Conditions

MS system:	Xevo TQ-S micro Triple Quadrupole Mass Spectrometer
Ionization mode:	ElectroSpray
Acquisition mode:	Multiple Reaction Monitoring (MRM)
Polarity:	Positive and negative
Capillary voltage:	1.5 kV/-1.5 kV
Desolvation temperature:	650 °C
Desolvation gas flow rate:	1000 L/hr
Cone gas flow rate:	150 L/hr
Source temperature:	150 °C

Collision energy:	See Appendix
Cone voltage:	See Appendix

Data Management

Software:	waters_connect for Quantitation Software

Results and Discussion

The injection solvent and volume used in the method were both considered as the QuEChERs extraction methodology utilizes acetonitrile as the extraction solvent. With the expanding scope of the method to cover hundreds of pesticides, it has often been advised to dilute the final extract in an aqueous solution to improve chromatographic performance for the early eluting pesticides, such as methamidophos.

Peak shapes are compared in Figure 1 for the first four early eluting compounds at 1 µL with and without an extension loop installed and at 2 µL with it fitted. The pre-column extension loop provides sufficient mobile phase mixing to allow focusing of the early eluting pesticides with significant improvement in peak shape compared to the 1 µL injection without the loop fitted. This not only significantly improves the chromatographic peak shape but also reduces the time required to review data for these pesticides as no manual re-integration was required. In fact, the positive effect of the extension loop on peak shape could be observed up to omethoate, the sixth eluting compound at R_t=2.52 minutes. Although 2 µL injections provided similar peak shape improvements for acephate and beyond, the sensitivity of the Xevo TQ-S micro allowed for a 1 µL injection volume to be used for the method performance requirements to be met.

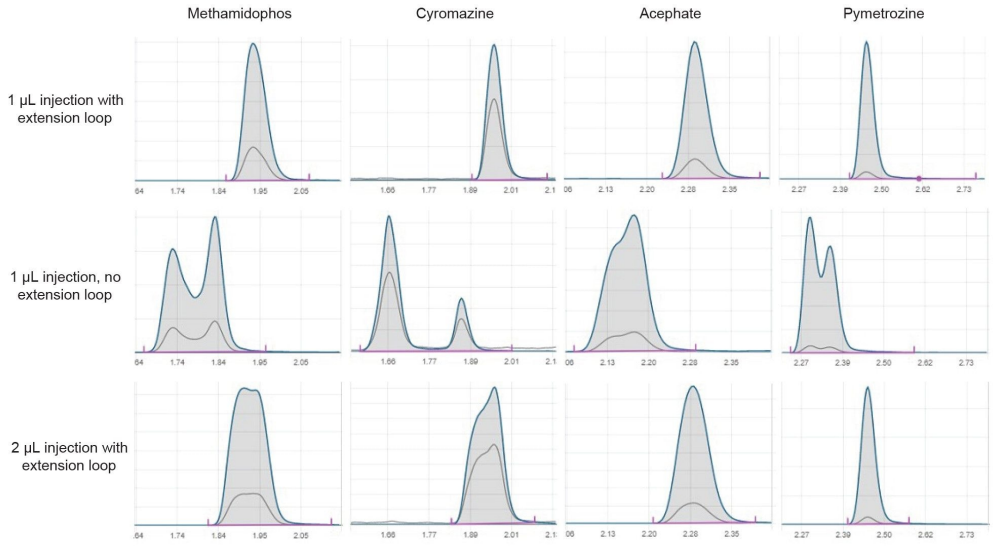

Figure 1. Impact of 50 µL extension loop fitted post injector, pre-column on early eluting pesticides of QuEChERs (acetonitrile extracts) without dilution in aqueous mobile phase.

The described multi-residue method was evaluated in strawberry purée, cereal-based baby food, and cereal-based animal feed using FAPAS Quality Control Materials. Concentrations of all residues were calculated using waters_connect for Quantitation Software using exception focused review (XFR). With XFR, the method automatically flags out of specification QC, calibration, and ion ratios based on the ruleset that was loaded into the processing method allowing users to focus on the injections and analytes that need the most attention. In this instance, these criteria were based on the SANTE guidelines listed in Table 1.[2]

Performance requirement	Tolerance
Retention times	± 0.1 min
Calibration curves	R^2>0.99, deviation ±20%
Blank response	<20% LLOQ
Signal to noise	S/N>3
Ion ratios	±30%
Calibration residual	±20%

Table 1. List of performance requirements used to drive exception focused review in waters_connect for Quantitation Software.

The method performance is shown in Figure 2 for each of the FAPAS QC materials against the assigned value with all observed values being within the assigned range of the analytes. The trueness of the detected pesticides was in the range of 100–130% compared to the assigned value with all calculated %RSDs under 20%.

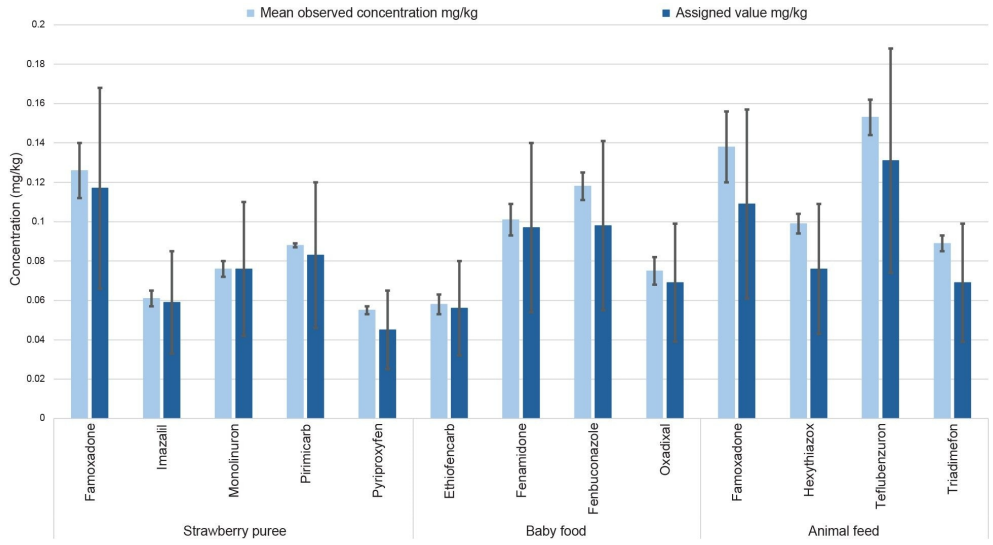

Figure 2. Results of FAPAS QC materials (n=5) with %RSD displayed as error bars in strawberry purée, cereal-based baby food, and animal feed.

Data was reviewed using waters_connect for Quantitation Software with the processing method used, directly generated from the acquisition method. Figure 3 shows the dashboard view for unknowns where the data assessment for the QC samples was completed. Positive results are shown at the top of the list, allowing users to focus their investigations on these residues efficiently. XFR was used to flag any problems with calibrations, QC samples, blanks, and ions ratios which significantly reduced the time on task reviewing the data set.

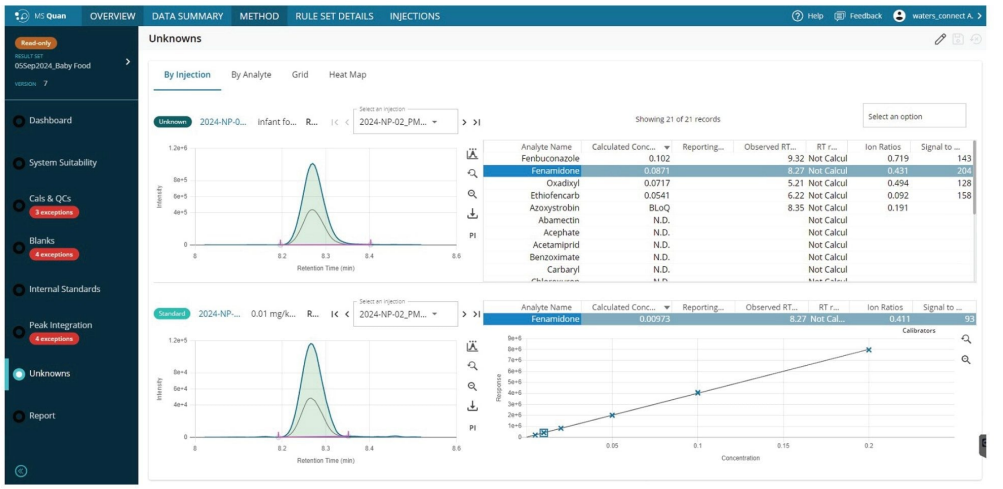

Figure 3. waters_connect for Quantitation Unknowns dashboard for the baby food QC material with the method exceptions automatically highlighted in red.

The last critical method parameter addressed in this work was the “up time” of the instrument to continue to analyze samples without excessive operator intervention. Figure 4 displays the peak area for Triadimefon in animal feed (with additional information on the other analytes in the legend) where the sample was repeatably injected into the LC-MS/MS system and the peak area plotted.

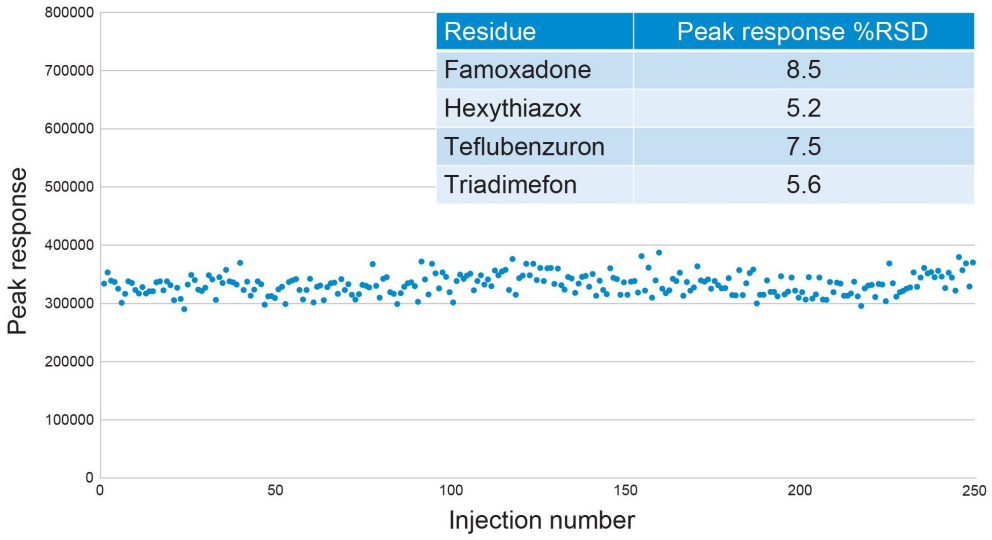

Figure 4. Robustness of Triadimefon with %RSD of the externally standardised peak responses for all residues in the animal feed QC material.

Conclusion

An LC-MS/MS multi-residue pesticide method for more than 200 residues using the ACQUITY Premier System, Xevo TQ-S micro Triple Quadrupole Mass Spectrometer, and waters_connect for Quantitation Software was used to assess method performance against commercially available quality control materials of strawberry purée, baby food, and animal feed.

Complex food and feed commodities were extracted using QuEChERS with baby food, and animal feed samples cleaned up using a simple and effective Oasis PRiME HLB Plus pass through protocol. The 1 µL acetonitrile extract was injected without dilution by using a post injector extension loop. The loop increased mobile phase mixing and significantly improved the peak shapes of early eluting compounds, such as methamidophos, which aided data processing and review of results, removing the need for any manual peak re-integrations.

Exception focused review in waters_connect for Quantitation Software was used to highlight results that fell outside SANTE tolerances, defined by a ruleset, increasing the efficiency of data review. The quality control materials were assessed using the Unknowns tab in this software, which significantly reduced the time taken for

data review. The processing method was directly generated from the acquisition method reducing the possibility of transcription errors.

Method performance was assessed using FAPAS QC materials with all measured values being within the assigned range of the analytes, the trueness was in the range of 100–130% and with all calculated %RSDs less than 20%. The robustness of the method was established, with animal feed as the most complex matrix, with externally standardised peak responses %RSDs all less than 9% for 250 injections.

References

1. Shah, D, ***et al***. Multi-residue Method for the Quantification of Pesticides in Fruits, Vegetables, Cereal, and Black Tea Using UPLC-MS/MS. Waters Application Note. 720006886. Revised February. 2021.
2. Document No. SANTE/11312/2021 V2. <https://food.ec.europa.eu/system/files/2023-11/pesticides_mrl_guidelines_wrkdoc_2021-11312.pdf> Guidance Document on Analytical Quality, Control, and Method Validation Procedures for Pesticides Residues Analysis in Food and Feed.

Appendix

Analyte	Precursor	Product	Polarity	Cone voltage (V)	Collision energy (V)	Retention time (min)
Abamectin	890.6	305.3	Positive	30	25	13.5
Abamectin	890.6	567.4	Positive	30	15	13.5
Acephate	183.93	94.93	Positive	5	21	2.29
Acephate	183.93	142.92	Positive	5	8	2.29
Acetamiprid	223	56.1	Positive	30	15	4.16
Acetamiprid	223	126	Positive	30	20	4.16
Acibenzolar-s-methyl	210.9	69	Positive	25	35	7.6
Acibenzolar-s-methyl	210.9	91	Positive	25	20	7.6
Acibenzolar-s-methyl	210.9	135.9	Positive	25	30	7.6
Alanycarb	400.1	90.9	Positive	5	25	13.3
Alanycarb	400.1	238.1	Positive	5	8	13.3
Aldicarb	213.1	47	Positive	35	25	4.8
Aldicarb	213.1	89	Positive	35	20	4.8
Aldicarb	213.1	116	Positive	35	11	4.8
Aldicarb sulfone	223	86	Positive	35	14	2.99
Aldicarb sulfone	223	148	Positive	35	10	2.99
Aldicarb sulfoxide	207	69	Positive	20	14	2.66
Aldicarb sulfoxide	207	89	Positive	20	15	2.66
Ametryn	228.1	68.1	Positive	25	35	6.66
Ametryn	228.1	186.1	Positive	25	20	6.66
Aminocarb	209	137	Positive	25	25	2.59
Aminocarb	209	152	Positive	25	15	2.59
Azoxystrobin	404.02	328.9	Positive	10	29	8.34
Azoxystrobin	404.02	372	Positive	10	11	8.34
Benalaxyl	326.1	91	Positive	25	30	10.24
Benalaxyl	326.1	148	Positive	25	20	10.24
Bendiocarb	224.11	109	Positive	15	15	5.53
Bendiocarb	224.11	167	Positive	15	10	5.53
Benfuracarb	411.1	190	Positive	35	10	7
Benfuracarb	411.1	195	Positive	35	20	7
Benzoximate	364	105	Positive	5	25	10.96
Benzoximate	364	199.1	Positive	5	10	10.96
Bifenazate	301.1	170	Positive	30	20	8.91
Bifenazate	301.1	198	Positive	30	5	8.91
Bitertanol	338.2	69.97	Positive	6	6	10
Bitertanol	338.2	99.1	Positive	6	12	10
Bitertanol	338.2	269.3	Positive	6	8	10
Boscalid	342.9	139.9	Positive	25	20	8.47
Boscalid	342.9	307	Positive	25	45	8.47
Bromuconazole I	376	70.1	Positive	15	20	8.95
Bromuconazole I	376	158.9	Positive	15	30	8.95
Bupirimate	317.11	108.09	Positive	35	27	9.4
Bupirimate	317.11	166.03	Positive	35	24	9.4
Buprofezin	306.1	115.9	Positive	20	16	12
Buprofezin	306.1	201	Positive	20	12	12
Butafenacil	492	180	Positive	25	35	9.58
Butafenacil	492	331	Positive	25	25	9.58
Butocarboxim	213	75	Positive	30	15	4.59

Butocarboxim	213	156	Positive	30	10	4.59
Butoxycarboxim	223	106	Positive	30	10	2.88
Butoxycarboxim	223	166	Positive	30	5	2.88
Carbaryl	202	117	Positive	25	25	5.9
Carbaryl	202	145	Positive	25	10	5.9
Carbendazim	192.1	132.1	Positive	10	30	3.14
Carbendazim	192.1	160.1	Positive	10	15	3.14
Carbetamide	237	118	Positive	5	15	4.94
Carbetamide	237	192	Positive	5	10	4.94
Carbofuran	222.11	123	Positive	5	20	5.65
Carbofuran	222.11	165.1	Positive	5	10	5.65
Carbofuran-3-hydroxyl	238	163	Positive	34	16	3.98
Carbofuran-3-hydroxyl	238	181	Positive	34	10	3.98
Carboxin	236	87	Positive	5	25	6.06
Carboxin	236	143	Positive	5	15	6.06
Carfentrazone-ethyl	412	346	Positive	55	24	9.88
Carfentrazone-ethyl	412	366	Positive	55	18	9.88
Chlorantraniliprole	481.6	283.9	Positive	15	23	7.65
Chlorantraniliprole	481.6	450.9	Positive	15	25	7.65
Chlorfluazuron	539.8	158	Positive	35	15	12.95
Chlorfluazuron	539.8	382.9	Positive	35	20	12.95
Chloroxuron	291.11	72.02	Positive	25	20	8.35
Chloroxuron	291.11	164.1	Positive	25	15	8.35
Chlorpyrifos-methyl	321.9	125	Positive	30	20	10.65
Chlorpyrifos-methyl	321.9	289.9	Positive	30	15	10.65
Clethodim	360.1	164	Positive	38	24	11.45
Clethodim	360.1	268.1	Positive	38	16	11.45
Clofentezine	303	102	Positive	20	35	10.45
Clofentezine	303	138	Positive	20	15	10.45
Clothianidin	250	132	Positive	25	15	3.81
Clothianidin	250	169	Positive	25	10	3.81
Cyazofamid	325	107.9	Positive	25	15	9.68
Cyazofamid	325	261	Positive	25	10	9.68
Cycluron	199	69.2	Positive	15	20	6.66
Cycluron	199	89.1	Positive	15	15	6.66
Cymoxanil	199.032	110.896	Positive	15	14	4.4
Cymoxanil	199.032	127.875	Positive	15	10	4.4
Cyproconazole I	292.2	70.2	Positive	5	20	8.25
Cyproconazole I	292.2	125.1	Positive	5	30	8.25
Cyprodinil	226.06	93	Positive	20	29	9.05
Cyprodinil	226.06	108	Positive	20	24	9.05
Cyromazine	167	60.2	Positive	40	20	2
Cyromazine	167	84.896	Positive	40	12	2
Demeton-S-methyl-sulfon	263	121	Positive	20	15	3.39
Demeton-S-methyl-sulfon	263	169	Positive	20	15	3.39
Desmedipham	301.01	136	Positive	45	25	7.55
Desmedipham	301.01	182	Positive	45	10	7.55
Diclobutrazol	328	70	Positive	15	20	9.36
Diclobutrazol	328	158.9	Positive	15	38	9.36
Dicrotophos	238	112	Positive	30	10	3.5

Dicrotophos	238	193	Positive	30	10	3.5
Diethofencarb	268	124	Positive	10	30	7.93
Diethofencarb	268	226	Positive	10	10	7.93
Difenoconazole	406.16	111.01	Positive	8	58	10.6
Difenoconazole	406.16	251.02	Positive	8	24	10.6
Diflubenzuron	311.03	141.1	Positive	25	30	9.12
Diflubenzuron	311.03	158.15	Positive	25	15	9.12
Dimethoate	230.07	125.02	Positive	15	20	4.07
Dimethoate	230.07	198.89	Positive	15	10	4.07
Dimethomorph I	388.1	165	Positive	15	30	8.1
Dimethomorph I	388.1	300.9	Positive	15	20	8.1
Dimoxystrobin	327.1	116.1	Positive	20	20	9.63
Dimoxystrobin	327.1	205.2	Positive	20	20	9.63
Diniconazole	326.1	70.2	Positive	10	25	10.14
Diniconazole	326.1	159	Positive	10	30	10.14
Dinotefuran	203	113	Positive	15	10	2.7
Dinotefuran	203	129	Positive	15	10	2.7
Dioxacarb	224.1	123.1	Positive	10	16	4.11
Dioxacarb	224.1	167.1	Positive	10	8	4.11
Disulfoton sulphoxide	291	97	Positive	25	30	6.3
Disulfoton sulphoxide	291	185	Positive	25	15	6.3
Diuron	233	72	Positive	25	18	6.65
Diuron	233	159.9	Positive	25	25	6.65
Doramectin	916.6	331.2	Positive	15	23	13.66
Doramectin	916.6	593.4	Positive	15	14	13.66
Emamectin benzoate	886.6	82	Positive	20	72	12.06
Emamectin benzoate	886.6	126	Positive	20	30	12.06
Emamectin benzoate	886.6	158	Positive	20	35	12.06
Epoxiconazole	330	101	Positive	40	40	9
Epoxiconazole	330	121.04	Positive	40	20	9
Eprinomectin	915.6	144	Positive	10	41	13.4
Eprinomectin	915.6	186	Positive	10	35	13.4
Etaconazole I	327.93	158.97	Positive	65	32	8.85
Etaconazole I	327.93	204.81	Positive	65	17	8.85
Ethiofencarb	226.1	107	Positive	10	15	6.24
Ethiofencarb	226.1	164	Positive	10	10	6.24
Ethiprole	414.1	350.9	Positive	10	25	8.1
Ethiprole	414.1	396.9	Positive	10	9	8.1
Ethirimol	210.1	98	Positive	5	25	4.24
Ethirimol	210.1	140	Positive	5	20	4.24
Ethofumesate	287.1	121.1	Positive	25	15	8.4
Ethofumesate	287.1	259.1	Positive	25	10	8.4
Etoxazole	360.2	57.2	Positive	60	25	12.7
Etoxazole	360.2	141.1	Positive	60	25	12.7
Famoxadone	392.2	238	Positive	5	15	10.34
Famoxadone	392.2	331.1	Positive	5	10	10.34
Fenamidone	312.1	92	Positive	5	25	8.27
Fenamidone	312.1	236.1	Positive	5	14	8.27
Fenarimol	331	81	Positive	40	30	8.6
Fenarimol	331	268	Positive	40	25	8.6

Fenazaquin	307.2	57.2	Positive	5	20	12.85
Fenazaquin	307.2	161	Positive	5	15	12.85
Fenbuconazole	337	70.1	Positive	15	20	9.31
Fenbuconazole	337	125	Positive	15	30	9.31
Fenhexamid	301.986	55.181	Positive	35	35	9
Fenhexamid	301.986	97.117	Positive	35	25	9
Fenobucarb	207.89	95.07	Positive	15	15	7.65
Fenobucarb	207.89	152	Positive	30	15	7.65
Fenoxycarb	302.1	88	Positive	10	20	9.4
Fenoxycarb	302.1	116.1	Positive	10	11	9.4
Fenpropimorph	304.2	57.2	Positive	25	30	7.65
Fenpropimorph	304.2	147.1	Positive	25	30	7.65
Fenpyroximate	422.1	138.05	Positive	30	32	12.9
Fenpyroximate	422.1	366.1	Positive	30	15	12.9
Fenuron	165	45.9	Positive	15	15	3.93
Fenuron	165	71.9	Positive	15	15	3.93
Fipronil	435.1	249.81	Negative	50	27	9.9
Fipronil	435.1	329.75	Negative	50	15	9.9
Fipronil desulfinyl	386.9	282	Negative	35	30	9.87
Fipronil desulfinyl	386.9	351	Negative	35	15	9.87
Fipronil sulfide	418.9	262	Negative	10	25	10.5
Fipronil sulfide	418.9	383	Negative	10	20	10.5
Fipronil sulfone	451	282	Negative	30	27	10.58
Fipronil sulfone	451	415	Negative	30	16	10.58
Flonicamid	230.1	174	Positive	45	16	3.39
Flonicamid	230.1	203	Positive	45	16	3.39
Fluazinam	462.81	397.8	Negative	50	16	11.95
Fluazinam	462.81	415.7	Negative	50	19	11.95
Flubendiamide	680.84	253.974	Negative	28	30	10.25
Flubendiamide	680.84	273.996	Negative	28	18	10.25
Fludioxonil	246.7	126	Negative	40	32	7.85
Fludioxonil	246.7	179.9	Negative	40	32	7.85
Flufenacet	364	152.1	Positive	5	20	9.35
Flufenacet	364	194.1	Positive	5	11	9.35
Flufenoxuron	489	141	Positive	30	40	12.55
Flufenoxuron	489	158	Positive	30	20	12.55
Fluometuron	233	46.4	Positive	30	15	6.15
Fluometuron	233	72.2	Positive	30	15	6.15
Fluoxastrobin	459	188	Positive	40	35	9.45
Fluoxastrobin	459	427	Positive	40	15	9.45
Fluquinconazole	376	306.9	Positive	25	25	8.8
Fluquinconazole	376	348.8	Positive	25	20	8.8
Flusilazole	316	165	Positive	5	25	9.3
Flusilazole	316	247	Positive	5	20	9.3
Flutolanil	324.1	65	Positive	25	35	8.85
Flutolanil	324.1	262.1	Positive	25	20	8.85
Flutriafol	302.1	70	Positive	25	16	6.46
Flutriafol	302.1	70.1	Positive	25	30	6.46
Fonofos	247	109	Positive	20	20	10.3
Fonofos	247	137	Positive	20	10	10.3

Forchlorfenuron	248.1	93	Positive	25	35	6.45
Forchlorfenuron	248.1	129	Positive	25	15	6.45
Formetanate	222.01	46	Positive	30	26	2.54
Formetanate	222.01	165	Positive	30	15	2.54
Fuberidazole	185	156	Positive	10	26	3.6
Fuberidazole	185	157	Positive	10	21	3.6
Furalaxyl	302.1	95	Positive	10	25	8.05
Furalaxyl	302.1	242.1	Positive	10	15	8.05
Furathiocarb	383.2	194.9	Positive	20	15	12
Furathiocarb	383.2	252	Positive	20	10	12
Halofenozide	331.1	104.9	Positive	10	15	8.15
Halofenozide	331.1	275	Positive	10	5	8.15
Haloxyfop	360	288	Negative	15	15	9.33
Haloxyfop	362	290	Negative	15	15	9.33
Hexaconazole	314.1	70.1	Positive	30	20	9.8
Hexaconazole	314.1	158.8	Positive	30	40	9.8
Hexaflumuron	459.1	175	Negative	5	30	11.15
Hexaflumuron	459.1	276.1	Negative	5	15	11.15
Hexythiazox	353	168.1	Positive	10	25	12.2
Hexythiazox	353	228.1	Positive	10	15	12.2
Hydramethylnon	495.15	151.1	Positive	20	62	11.1
Hydramethylnon	495.15	323.9	Positive	20	40	11.1
Imazalil	297.01	69.08	Positive	23	18	6.2
Imazalil	297.01	158.88	Positive	23	22	6.2
Imidacloprid	256.1	174.9	Positive	25	20	3.9
Imidacloprid	256.1	209	Positive	25	12	3.9
Indoxacarb	528.1	202.9	Positive	30	40	11.3
Indoxacarb	528.1	217.9	Positive	30	20	11.3
Ipconazole	334.2	70	Positive	50	25	10.85
Ipconazole	334.2	125	Positive	50	25	10.85
Iprovalicarb	321.1	119.1	Positive	20	20	8.75
Iprovalicarb	321.1	203.1	Positive	20	10	8.75
Isocarbofos	291.2	121	Positive	16	30	8.8
Isocarbofos	291.2	215.1	Positive	16	8	8.8
Isocarbofos	291.2	231	Positive	16	12	8.8
Isoprocarb	193.99	95.09	Positive	15	13	6.6
Isoprocarb	193.99	136.91	Positive	15	8	6.6
Isoproturon	207	46	Positive	20	15	6.62
Isoproturon	207	72	Positive	20	20	6.62
Ivermectin	892.6	307.2	Positive	15	24	13.85
Ivermectin	892.6	551.4	Positive	15	25	13.85
Ivermectin	892.6	569.4	Positive	15	14	13.85
Kresoxim-methyl	314.2	115.9	Positive	30	12	9.9
Kresoxim-methyl	314.2	131	Positive	30	25	9.9
Linuron	249	159.9	Positive	20	20	7.75
Linuron	249	181.9	Positive	20	16	7.75
Lufenuron	509	325.89	Negative	2	20	12.15
Lufenuron	509	338.96	Negative	2	12	12.15
Mandipropamid	411.8	125	Positive	35	35	8.7
Mandipropamid	411.8	328.1	Positive	35	15	8.7

Mefenacet	299	120	Positive	5	25	8.7
Mefenacet	299	148	Positive	5	15	8.7
Mepanipyrim	224.1	77	Positive	15	35	8.7
Mepanipyrim	224.1	106	Positive	15	25	8.7
Mepronil	270.1	91	Positive	15	35	8.75
Mepronil	270.1	119	Positive	15	25	8.75
Mesotrione	340.1	104	Positive	30	30	4.9
Mesotrione	340.1	228.1	Positive	30	15	4.9
Metaflumizone	507.13	178	Positive	40	28	12
Metaflumizone	507.13	287.1	Positive	45	22	12
Metalaxyl	280.1	192.1	Positive	30	15	6.94
Metalaxyl	280.1	220.1	Positive	30	15	6.94
Metconazole	320	70	Positive	10	20	10
Metconazole	320	125	Positive	10	35	10
Methabenzthiazuron	222	150	Positive	10	30	6.11
Methabenzthiazuron	222	165	Positive	10	15	6.11
Methamidophos	141.9	93.9	Positive	30	12	1.92
Methamidophos	141.9	124.8	Positive	30	14	1.92
Methiocarb	226	121	Positive	25	20	7.65
Methiocarb	226	169	Positive	25	10	7.65
Methiocarb sulfone	258.07	107.1	Positive	40	35	4.37
Methiocarb sulfone	258.07	122.1	Positive	40	20	4.37
Methomyl	162.9	88	Positive	15	15	3.17
Methomyl	162.9	105.9	Positive	15	15	3.17
Methoprotryne	272.07	197.98	Positive	13	22	6.85
Methoprotryne	272.07	240.05	Positive	13	18	6.85
Methoxyfenozide	369.2	149.1	Positive	15	15	9
Methoxyfenozide	369.2	313.23	Positive	5	10	9
Metobromuron	259.1	148.1	Positive	25	15	6.55
Metobromuron	259.1	170	Positive	25	20	6.55
Metribuzin	215	89	Positive	5	20	5.35
Metribuzin	215	131	Positive	5	20	5.35
Mevinphos I	225.1	127.1	Positive	15	15	4.37
Mevinphos I	225.1	193.1	Positive	15	10	4.37
Mexacarbate	223.2	151	Positive	40	25	4.25
Mexacarbate	223.2	166.1	Positive	40	15	4.25
Monocrotophos	224.1	98	Positive	20	10	3.3
Monocrotophos	224.1	109	Positive	20	30	3.3
Monocrotophos	224.1	127	Positive	20	15	3.3
Monolinuron	215.04	99	Positive	25	30	6.17
Monolinuron	215.04	126	Positive	25	15	6.17
Moxidectin	640.5	199	Positive	5	25	13.6
Moxidectin	640.5	498.3	Positive	5	14	13.6
Moxidectin	640.5	528.4	Positive	5	10	13.6
Myclobutanil	289.1	70.2	Positive	25	15	8.58
Myclobutanil	289.1	125.1	Positive	25	30	8.58
Neburon	275	57	Positive	15	20	9.6
Neburon	275	88	Positive	15	15	9.6
Nitenpyram	271.1	125.9	Positive	30	30	3.1
Nitenpyram	271.1	225	Positive	30	10	3.1

Novaluron	493.02	141	Positive	5	30	11.5
Novaluron	493.02	158.03	Positive	5	15	11.5
Nuarimol	315	81.1	Positive	25	15	7.5
Nuarimol	315	252	Positive	25	20	7.5
Omethoate	214	124.8	Positive	25	22	2.5
Omethoate	214	182.8	Positive	25	10	2.5
Oxadixyl	279.1	132.3	Positive	20	25	5.24
Oxadixyl	279.1	219	Positive	20	12	5.24
Oxamyl	237	72	Positive	15	10	4.95
Oxamyl	237	90	Positive	15	10	4.95
Paclobutrazol	294.1	70.2	Positive	10	20	8
Paclobutrazol	294.1	125.1	Positive	10	35	8
Penconazole	284	70.1	Positive	15	15	9.6
Penconazole	284	159	Positive	15	25	9.6
Pencycuron	329.1	124.9	Positive	30	30	10.85
Pencycuron	329.1	218	Positive	30	16	10.85
Phenmedipham	301	136	Positive	45	20	7.65
Phenmedipham	301	168	Positive	45	10	7.65
Picoxystrobin	368.01	145.07	Positive	13	24	10
Picoxystrobin	368.01	205.06	Positive	13	8	10
Piperonyl butoxide	356.3	119	Positive	20	35	12
Piperonyl butoxide	356.3	176.9	Positive	20	10	12
Pirimicarb	239.1	72	Positive	25	20	5.01
Pirimicarb	239.1	182.1	Positive	25	15	5.01
Pirimiphos Methyl	306.3	108.1	Positive	35	30	10.65
Pirimiphos Methyl	306.3	164.1	Positive	35	20	10.65
Prochloraz	376.03	70.1	Positive	25	25	10
Prochloraz	376.03	307.9	Positive	25	10	10
Promecarb	208.1	109	Positive	25	15	8.05
Promecarb	208.1	151	Positive	25	10	8.05
Prometon	226	86.3	Positive	15	30	5.8
Prometon	226	184.3	Positive	15	20	5.8
Prometryn	242	158	Positive	25	25	8
Prometryn	242	200.1	Positive	25	18	8
Propamocarb	189.1	102	Positive	15	15	2.58
Propamocarb	189.1	144	Positive	15	10	2.58
Propargite	368	57	Positive	15	15	12.75
Propargite	368	175	Positive	15	15	12.75
Propargite	368	231	Positive	15	10	12.75
Propetamphos	282.09	138	Positive	45	20	8.92
Propetamphos	282.09	156	Positive	45	15	8.92
Propham	180	120	Positive	10	15	6.6
Propham	180	138	Positive	10	10	6.6
Propiconazole	342.1	69.1	Positive	35	30	9.95
Propiconazole	342.1	158.9	Positive	35	20	9.95
Propoxur	210.1	92.9	Positive	15	25	5.6
Propoxur	210.1	110.9	Positive	15	12	5.6
Prothioconazole	344	189	Positive	15	20	9.85
Prothioconazole	344	326	Positive	15	10	9.85
Pymetrozine	218.1	79	Positive	25	25	2.47

Pymetrozine	218.1	105	Positive	25	15	2.47
Pyracarbolid	218.1	97.1	Positive	10	30	5.85
Pyracarbolid	218.1	125.1	Positive	10	20	5.85
Pyraclostrobin	388.17	163.1	Positive	18	24	10.6
Pyraclostrobin	388.17	194.1	Positive	18	12	10.6
Pyridaben	365.1	147.1	Positive	20	25	13.3
Pyridaben	365.1	309.1	Positive	20	10	13.3
Pyrimethanil	199.99	82.05	Positive	45	26	7.1
Pyrimethanil	199.99	107.06	Positive	45	24	7.1
Pyriproxyfen	322.2	95.9	Positive	15	15	11.95
Pyriproxyfen	322.2	184.9	Positive	15	20	11.95
Quinoxyfen	308	161.9	Positive	35	45	11.75
Quinoxyfen	308	197	Positive	35	30	11.75
Rotenone	395	192.1	Positive	10	20	9.55
Rotenone	395	213.1	Positive	10	24	9.55
Secbumeton	226.2	100.2	Positive	5	25	6
Secbumeton	226.2	170.2	Positive	5	20	6
Siduron	233.01	94.06	Positive	23	20	7.7
Siduron	233.01	137.06	Positive	23	17	7.7
Simetryn	214	95.9	Positive	15	25	5.55
Simetryn	214	124	Positive	15	20	5.55
Spinetoram (J)	748.53	98.07	Positive	60	35	11.38
Spinetoram (J)	748.53	142.16	Positive	60	30	11.38
Spinetoram (L)	760.53	98.07	Positive	34	66	11.85
Spinetoram (L)	760.53	142.09	Positive	34	30	11.85
Spinosad A	732.6	98.1	Positive	35	50	10.5
Spinosad A	732.6	142	Positive	35	30	10.5
Spinosad D	746.52	98.1	Positive	40	35	11.08
Spinosad D	746.52	142	Positive	40	31	11.08
Spirodiclofen	411.14	71.6	Positive	35	15	13.35
Spirodiclofen	411.14	313.1	Positive	35	10	13.35
Spiromesifen	371.1	255.1	Positive	35	25	13.15
Spiromesifen	371.1	273.1	Positive	35	5	13.15
Spirotetramat	374	302	Positive	20	30	8.9
Spirotetramat	374	330	Positive	20	15	8.9
Sulfentrazone	387	145.8	Positive	60	35	6.15
Sulfentrazone	387	307	Positive	60	30	6.15
Tebuconazole	308.2	70.1	Positive	30	30	9.55
Tebuconazole	308.2	124.9	Positive	30	30	9.55
Tebufenozide	353.3	133.07	Positive	2	16	9.85
Tebufenozide	353.3	297.2	Positive	2	4	9.85
Tebufenpyrad	334	117	Positive	15	25	11.65
Tebufenpyrad	334	145	Positive	15	25	11.65
Tebuthiuron	229	116	Positive	5	25	5.2
Tebuthiuron	229	172	Positive	5	15	5.2
Teflubenzuron	381	113	Positive	25	60	11.18
Teflubenzuron	381	141	Positive	25	30	11.18
Teflubenzuron	381	158	Positive	25	15	11.18
Temephos	466.8	125	Positive	50	30	12.15
Temephos	466.8	418.9	Positive	50	20	12.15

Terbumeton	226.1	114.1	Positive	35	25	5.74
Terbumeton	226.1	170.1	Positive	35	15	5.74
Terbutryn	242.1	91	Positive	5	25	8.2
Terbutryn	242.1	186.1	Positive	5	20	8.2
Tetraconazole	372	70.1	Positive	15	20	9
Tetraconazole	372	159	Positive	15	25	9
Tetradifon	294	197.2	Positive	56	21	8.6
Tetradifon	294	225	Positive	56	19	8.6
Thiabendazole	202	130.9	Positive	45	30	3.44
Thiabendazole	202	174.9	Positive	45	25	3.44
Thiacloprid	253	90	Positive	35	40	4.6
Thiacloprid	253	125.8	Positive	35	20	4.6
Thiamethoxam	292	132	Positive	25	20	3.5
Thiamethoxam	292	211.2	Positive	25	10	3.5
Thidiazuron	221	102	Positive	10	15	5.2
Thidiazuron	221	128	Positive	10	15	5.2
Thiobencarb	258.1	89.1	Positive	25	45	10.5
Thiobencarb	258.1	125.1	Positive	25	15	10.5
Thiofanox	219	57	Positive	35	5	6.33
Thiofanox	219	76	Positive	35	5	6.33
Thiophanate methyl	343	151	Positive	25	20	5.55
Thiophanate methyl	343	311	Positive	25	15	5.55
Triadimefon	294.1	69.1	Positive	30	20	8.49
Triadimefon	294.1	196.9	Positive	30	16	8.49
Triadimenol	296.1	70	Positive	30	10	8.25
Triadimenol	296.1	98.9	Positive	30	15	8.25
Trichlorfon	256.9	79	Positive	25	30	3.88
Trichlorfon	256.9	108.8	Positive	25	20	3.88
Tricyclazole	190	136	Positive	10	25	4.41
Tricyclazole	190	163	Positive	10	20	4.41
Trifloxystrobin	409.2	145	Positive	25	40	11.3
Trifloxystrobin	409.2	185.9	Positive	25	14	11.3
Triflumizole	346.1	73.1	Positive	15	18	10.8
Triflumizole	346.1	278	Positive	15	10	10.8
Triflumuron	359	139.1	Positive	5	30	10.3
Triflumuron	359	156.1	Positive	5	20	10.3
Triticonazole	318.1	70.1	Positive	5	20	8.7
Triticonazole	318.1	124.9	Positive	5	30	8.7
Vamidothion	288	118	Positive	20	25	3.95
Vamidothion	288	146	Positive	20	20	3.95
Zoxamide	336.05	158.97	Positive	13	42	10.3
Zoxamide	336.05	186.91	Positive	13	20	10.3

Featured Products

ACQUITY Premier System <
https://www.waters.com/nextgen/global/products/chromatography/chromatography-systems/acquity-premier-system.html>

Xevo TQ-S micro Triple Quadrupole Mass Spectrometer <

https://www.waters.com/nextgen/global/products/mass-spectrometry/mass-spectrometry-systems/xevo-tq-s-micro.html>

waters_connect for Quantitation <https://www.waters.com/nextgen/global/products/informatics-and-software/waters_connect-for-quantitation.html>

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