

Determination of Pesticide Residues in Wheat Flour and Cucumber After Extraction With QuEChERS and Clean-up With Oasis™ PRiME HLB SPE

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Abstract

In this application note we describe the transfer of an established UPLC™ method onto a CORTECS™ T3 Column, assessing overall method performance by validating this approach using high-water and high-starch content food commodities, namely cucumber and wheat flour. The use of the CORTECS™ T3 Column generates significantly lower back pressure, allowing for a LC system with decreased performance specifications to be used. Using the Xevo™ TQ-S cronos for detection, we demonstrate that similar method performance can be achieved for a selection of representative pesticides at 0.01 mg/kg, the typical EU default pesticide MRL in these food commodities. A pass-through SPE with Oasis™ PRiME HLB was utilized for this approach, which offered a quick and effective alternative to dispersive SPE. The method was successfully validated using the SANTE guidelines. Results from analysis of spikes at both 0.01 and 0.1 mg/kg showed that for cucumber and wheat flour, 94% and 99% of analytes passed the validation criteria, with overall %RSDs for cucumber of 3.6%, and 4.6% for wheat flour respectively.

Benefits

Oasis PRiME HLB technology provides a quick and simple alternative to a dispersive SPE workflow, whilst maintaining excellent recoveries for the pesticides of interest.

The ACQUITY Arc™, in combination with the CORTECS T3 Column, offers a UHPLC method that gives comparable run times, and method performance to UPLC multi-residue methods.

The Xevo TQ-S cronos provides a sensitive and robust analysis for over 150 pesticides in one analytical method, at the typical EU default MRL of 0.01 mg/kg.

Introduction

Pesticides are an essential safeguard to the modern food ecosystem, helping to stabilize food supply by suppressing pests, weeds, and disease. However, excessive or unlawful use of pesticides on agricultural crops and in produce destined for human consumption can result in inadmissibly high levels of these compounds, which can pose adverse risk to human health.¹ As a result, Maximum Residue Levels (MRLs) have been established within the EU to oversee pesticides in raw agricultural commodities, where approved pesticides are applied in accordance with Good Agricultural Practice.² For many pesticides a default MRL of 0.01 mg/kg is used within the EU when the product is not authorized for use on a food commodity, and this is generally the target method limit of quantification used when establishing an analytical pesticide method.

Here we describe the performance of a robust, quantitative method for the routine determination of a range of LC-amenable pesticides in food commodities belonging to commodity groups 1 and 5 defined under SANTE/11312/2021.³ Following QuEChERS sample preparation and clean-up with an Oasis PRiME HLB SPE Cartridge (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>>), analysis was performed with an ACQUITY Arc System coupled to the Xevo TQ-S cronos Tandem Quadrupole Mass Spectrometer.⁴ Pass-through clean-up with the Oasis PRiME HLB SPE Cartridge can be a quick and effective alternative to traditional dispersive SPE, following extraction using the QuEChERS CEN approach. The sensitivity and robustness of the Xevo TQ-S cronos is shown, presenting suitability for the simultaneous quantitative determination of many pesticides in compliance with the default EU MRL of 0.01 mg/kg. The unique reverse cone design of the TQ-S cronos helps to reduce matrix aggregation, increasing the up-time of the instrument.

The objective of this work was to demonstrate ease of method transfer of a UPLC to UHPLC chromatographic

method, while maintaining expected performance and sensitivity required for pesticide analysis. The CORTECS T3 Column generates significantly lower back pressure, allowing for a LC system with decreased performance specifications to be used. The column's superficially porous silica particle morphology offers high efficiency and provides excellent peak shape, giving longer column lifetime ideal for supporting high throughput multi-residue methods.

Experimental

Samples of wheat flour and cucumber were purchased at a local retail outlet and screened to ensure an absence of target pesticide residues. These matrices were selected as representative samples of high-starch and high-water content commodity groups respectively. Samples were homogenized, with the cucumber sample stored at -20 °C and the wheat flour stored at room temperature.

Samples were extracted using a modification of the QuEChERS CEN Method 15662 where the dSPE step was replaced by pass-through SPE, using the Oasis PRiME HLB Short Plus Cartridge (see Figure 1).⁵ Following routine QuEChERS extraction, approximately 4 mL of supernatant was passed through an Oasis PRiME HLB Plus Short Cartridge. Discarding the first 1 mL, the following 2 mL aliquot was collected in a marked vial. 250 µL of this sample was then diluted to 1 mL with 750 µL of H₂O in an LC-MS vial ready for injection.

Samples were spiked at 0.01 mg/kg and 0.1 mg/kg, with 5 replicates at each level for both sample types of cucumber and wheat flour for the method validation study. Linearity was assessed using solvent and matrix matched standards (MMS), with calibration curves created – ranging from 0.005 mg/kg to 0.5 mg/kg (equating to in-vial concentrations of 0.00125–0.125 µg/mL for cucumber, and 0.000625–0.0625 µg/mL in wheat flour).

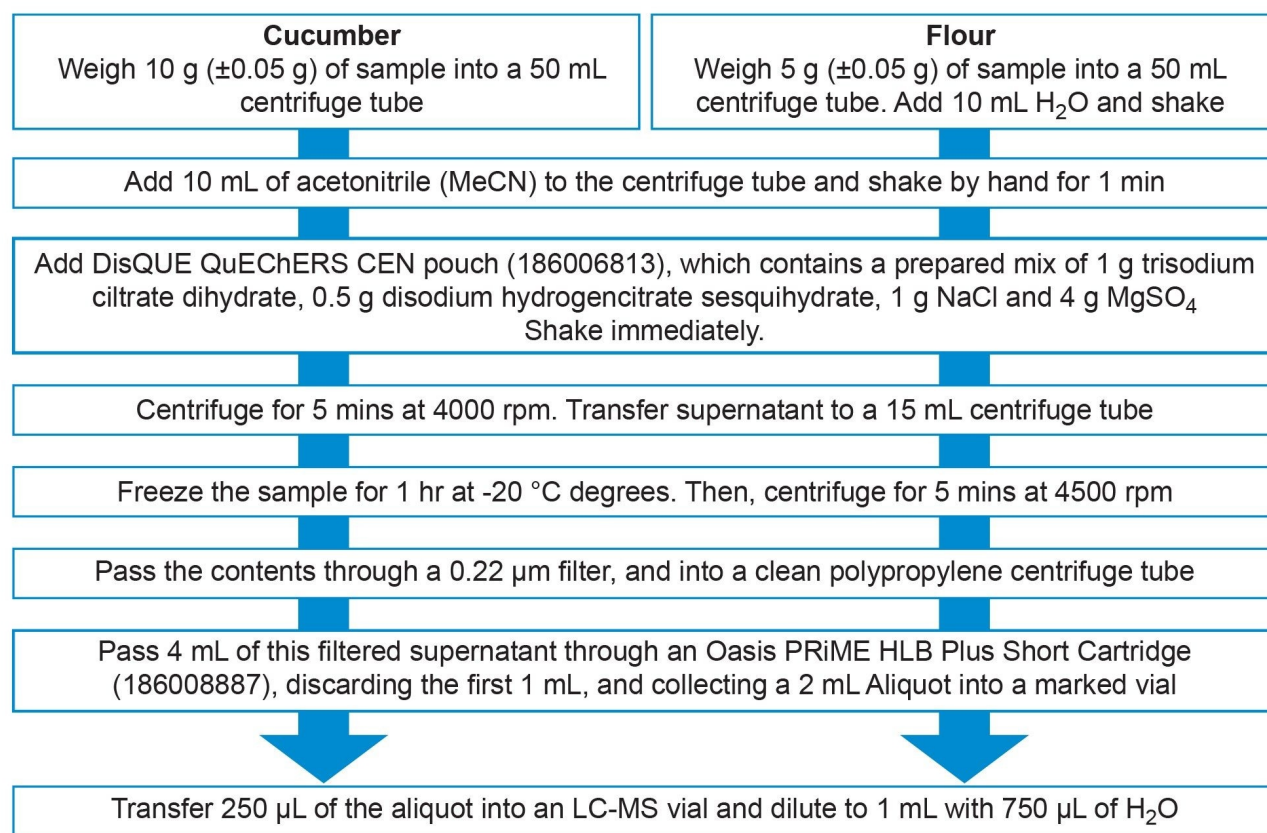


Figure 1. A workflow to show sample preparation method for cucumber and wheat Flour.

This UHPLC method was generated from an existing UPLC method, using the Columns Calculator 2.0 Software shown in Figure 2 ([667005222 <](https://www.waters.com/waters/support.htm?lid=134891632&lcid=134891631&type=DWNL)

<https://www.waters.com/waters/support.htm?lid=134891632&lcid=134891631&type=DWNL>).⁶ Specifications for the CORTECS T3 Column, 120 Å, 2.7 μ m, 2.1 mm x 100 mm, were used to translate the existing LC method. The software allowed for verification that an exact translation of this method was within the maximum pressure limit of the system configuration, thus allowing for flow rate and run times to mirror UPLC conditions.

MRM transitions and compound specific MS parameters such as cone voltage and collision energy were downloaded from the relevant Quanpedia™ database (see Appendix 1). The database houses a compendium of compound data, and automatically creates LC and MS acquisition methods, in addition to processing methods. The MS method used in this work was created by Quanpedia and contains at least 2 MRM transitions per pesticide. Source conditions were optimized in favor of poorer performing compounds in both ESI+ and ESI-.

Auto-dwell functionality allowed for sufficient points across the peak throughout the method (more than 12 data points per peak).

The screenshot shows the Columns Calculator software interface with two main panels: "From..." and "To...".

From... (Original Method):

- Column:** Diameter (D): 2.100 mm, Length (L): 100 mm, Particle Size (dp): 1.8 μ m, L/dp: 55,556
- System:** Dwell volume: 0.000 mL
- Method:** Injection volume: 5.0 μ L, Temperature: 40 $^{\circ}$ C, Run time: 19.00 min

To... (Target Method):

- Column:** Diameter (D): 2.100 mm, Length (L): 100 mm, Particle Size (dp): 2.7 μ m, L/dp: 37,037
- System:** Dwell volume: 0.000 mL, High pressure limit: 9,000 psi
- Method:** Flow rate: Scaled (0.333 mL/min) or Custom (0.500 mL/min)

Both panels include a table with the following columns: Time (min), Flow Rate (mL/min), %A Water, %B Acetonitrile, and Column Volumes.

From... Table:

	Time (min)	Flow Rate (mL/min)	%A Water	%B Acetonitrile	Column Volumes
1	0.00	0.500	99.0	1.0	0.00
2	0.50	0.500	99.0	1.0	1.09
3	3.50	0.500	60.0	40.0	6.56
4	12.50	0.500	15.0	85.0	19.69
5	12.60	0.500	1.0	99.0	0.22
6	15.00	0.500	1.0	99.0	5.25
7	15.10	0.500	99.0	1.0	0.22

To... Table:

	Time (min)	Flow Rate (mL/min)	%A Water	%B Acetonitrile	Column Volumes
1	0.00	0.500	99.0	1.0	0.00
2	0.50	0.500	99.0	1.0	1.09
3	3.50	0.500	60.0	40.0	6.56
4	12.50	0.500	15.0	85.0	19.69
5	12.60	0.500	1.0	99.0	0.22
6	15.00	0.500	1.0	99.0	5.25
7	15.10	0.500	99.0	1.0	0.22

From... Summary: 8,724 psi (Maximum pressure)

To... Summary: 3,877 psi (Maximum pressure), 5.0 μ L (Injection volume), 19.00 min (Run time)

Figure 2. Transferring an existing pesticide LC Method from the ACQUITY UPLC HSS T3 Column, 1.8 μ m, 2.1 x 100 mm (p/n: 186003539) to the CORTECS T3 Column, 120 Å , 2.7 μ m, 2.1 mm x 100 mm (p/n: 186008484) using the Columns Calculator 2.0 Software (667005222).

UHPLC-MS/MS Conditions

LC system:	ACQUITY Arc with FTN-R Sample Manager
Detection:	Xevo TQ-S cronos
Post injector mixing kit:	50 µL Extension Loop (p/n: 430002012)
Column(s):	CORTECS T3 Column, 120 Å, 2.7 µm, 2.1 mm x 100 mm (p/n: 186008484)
Column temperature:	40 °C
Sample temperature:	10 °C
Injection volume:	5 µL
Flow rate:	0.5 mL/min
Run time:	19 mins
Mobile phase A:	5 mM Ammonium Formate in H ₂ O +0.1% Formic Acid
Mobile phase B:	5 mM Ammonium Formate in 50:50 MeCN:MeOH +0.1% Formic Acid
Vials:	Clear Glass 12 x 32 mm Screw Neck Vial, 100/pk (p/n: 186000273)

Gradient

Time (min)	Flow (mL/min)	%A	%B	Curve
Initial	0.5	99	1	6
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

Software

Chromatography software:

TargetLynx™ XS

MS software:

MassLynx™ Version 4.2

Source Conditions

MS system: Xevo TQ-S cronos

Ionization: Electrospray

Ionization mode: +/-

Capillary voltage: +0.4 kV / - 0.50 kV

Desolvation temperature: 600 °C

Desolvation gas flow: 1000 L/Hr

Source temperature: 150 °C

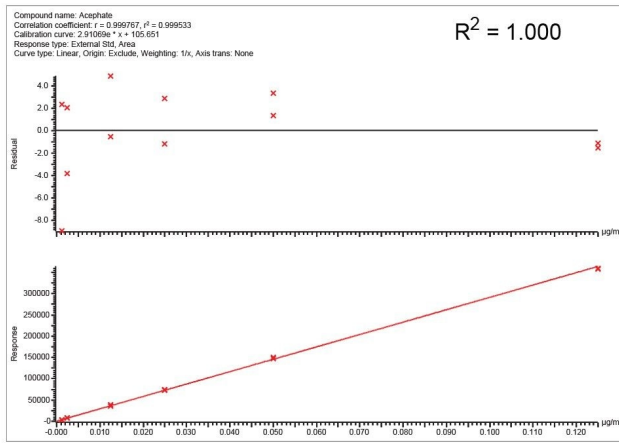
Cone gas flow: 0 L/Hr

Results and Discussion

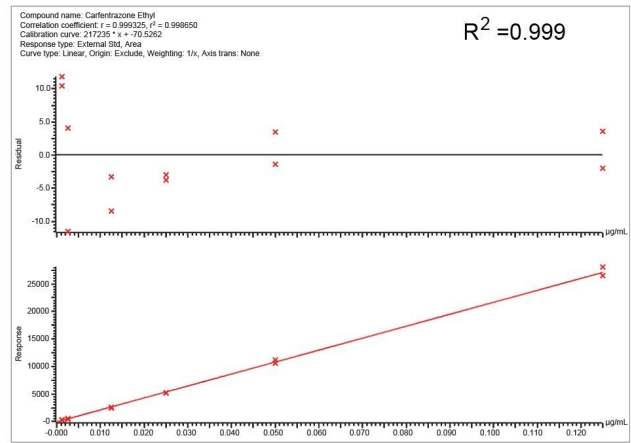
The sensitivity of the method was evaluated by assessment of matrix-matched calibration standards, with concentrations ranging from an equivalent of 0.005 mg/kg in the lowest concentration standard (0.00125 µg/mL and 0.000625 µg/mL in-vial, for cucumber and wheat flour respectively). Blank response for the respective matrices was below the criteria of ≤30% of the required reporting limit for the more than 180 analytes in the method. Response in matrix blanks was negligible, and no significant interferences in the chromatograms for either the quantifier or qualifier transitions were observed at the respective retention times.

The deviation of the back-calculated concentrations of the calibration standards from the true concentrations (residuals) did not exceed ±20%. All analytes exhibited residuals within this tolerance, set out by the SANTE guidelines. Some examples of this can be seen in Figure 3 and Figure 4. Calibration plots for almost all analytes (98% in cucumber, 96% in wheat flour) gave values of $r^2 \geq 0.99$, with only a few exceptions. Clothianidin and thiophanate methyl in cucumber matrix, and aldicarb, mesotrione, monolinuron, sulfentrazone, thiofanox, and zoxamide in wheat flour matrix, all gave of $r^2 > 0.97$ or higher. Ethofumesate ($r^2 > 0.94$) was the only analyte to fall below this threshold in both matrices. A majority of calibration plots across both matrices were linear. A weighting factor of (1/x) was used to construct the matrix-matched calibration curves.

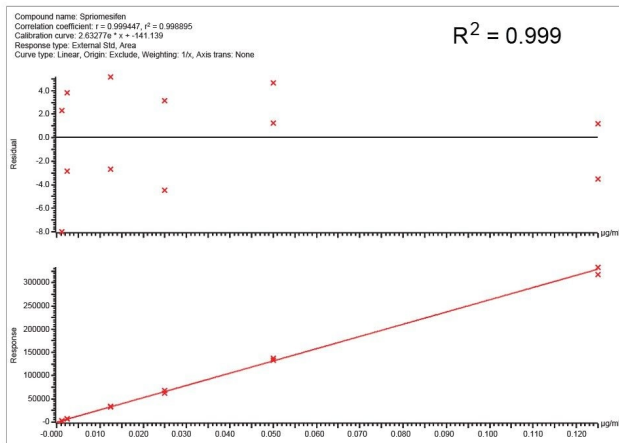
Matrix-matched calibration plots – cucumber



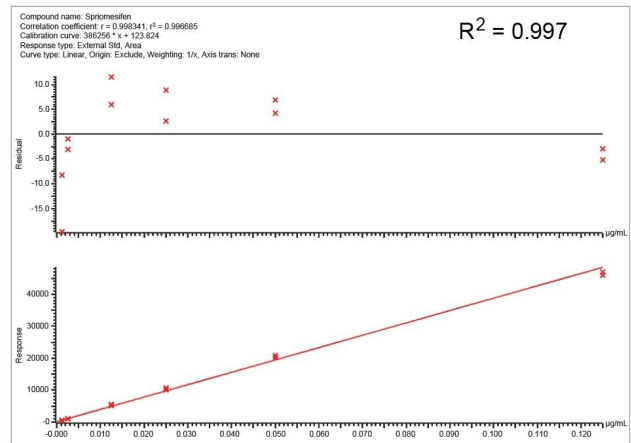
Acephate



Carfentrazone Ethyl



Propiconazole



Spiromesifen

Figure 3. Calibration graphs from the analysis of a selection of pesticides in cucumber matrix.

Matrix-matched calibration plots – wheat flour

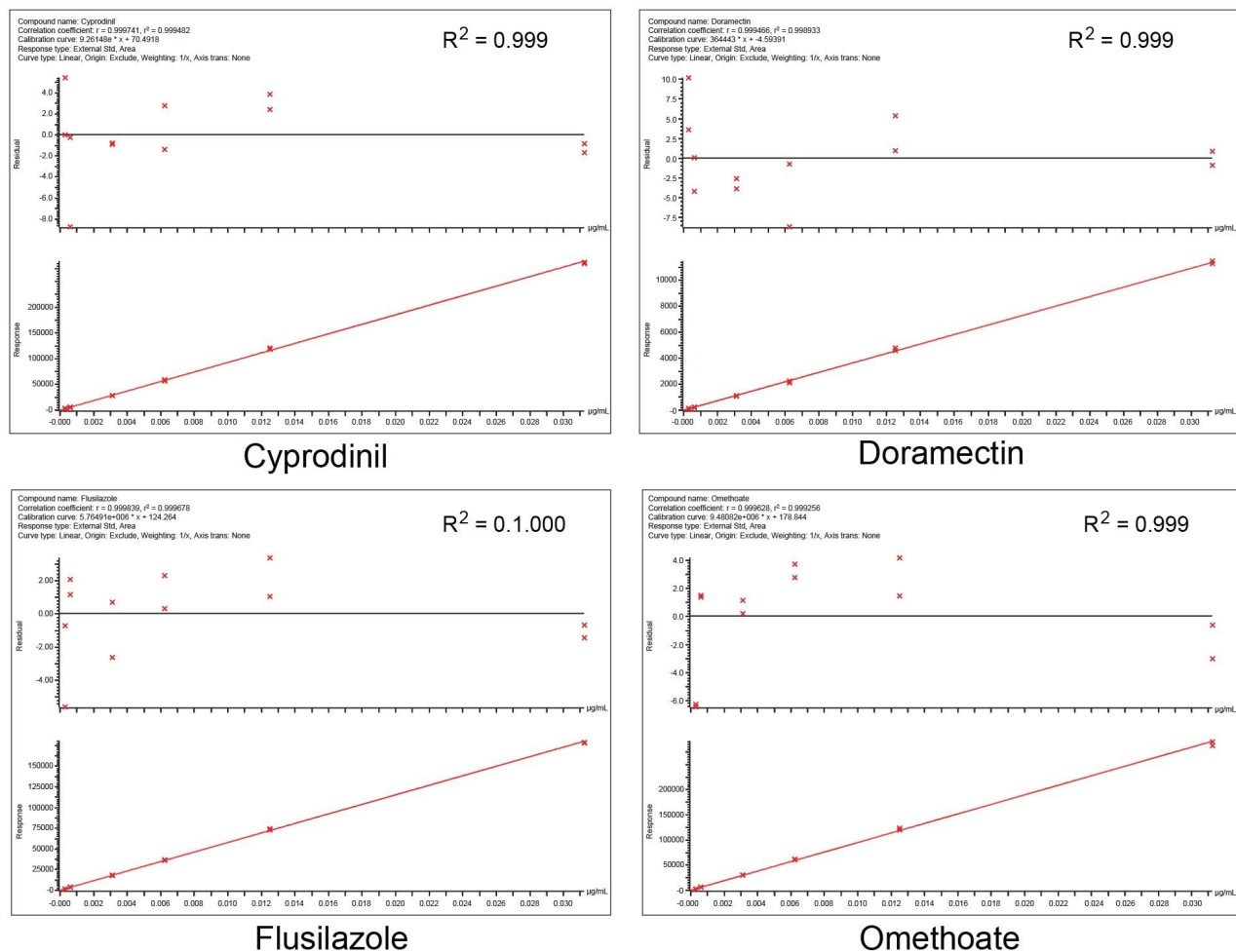


Figure 4. Calibration graphs from the analysis of a selection of pesticides in wheat flour matrix.

Matrix effects are often observed in routine pesticide analysis by LC-MS/MS and are primarily due to the presence of matrix components co-eluting with the analyte, thus competing for ionization efficiency. We have calculated matrix effect using the following percentage calculation:

$$\%ME = \left[\frac{b_M}{b_S} - 1 \right] \times 100$$

Where b_M and b_S are the slope of the matrix-matched and solvent calibration curves respectively.

Compounds displayed in Figure 5 give a representation of the matrix effect across the method, with the first (methamidophos) and last (ivermectin) eluting compounds displayed amongst other compounds, as well as one of two negative compounds in the method (fluazinam).

% Matrix effect in cucumber and wheat flour matrix

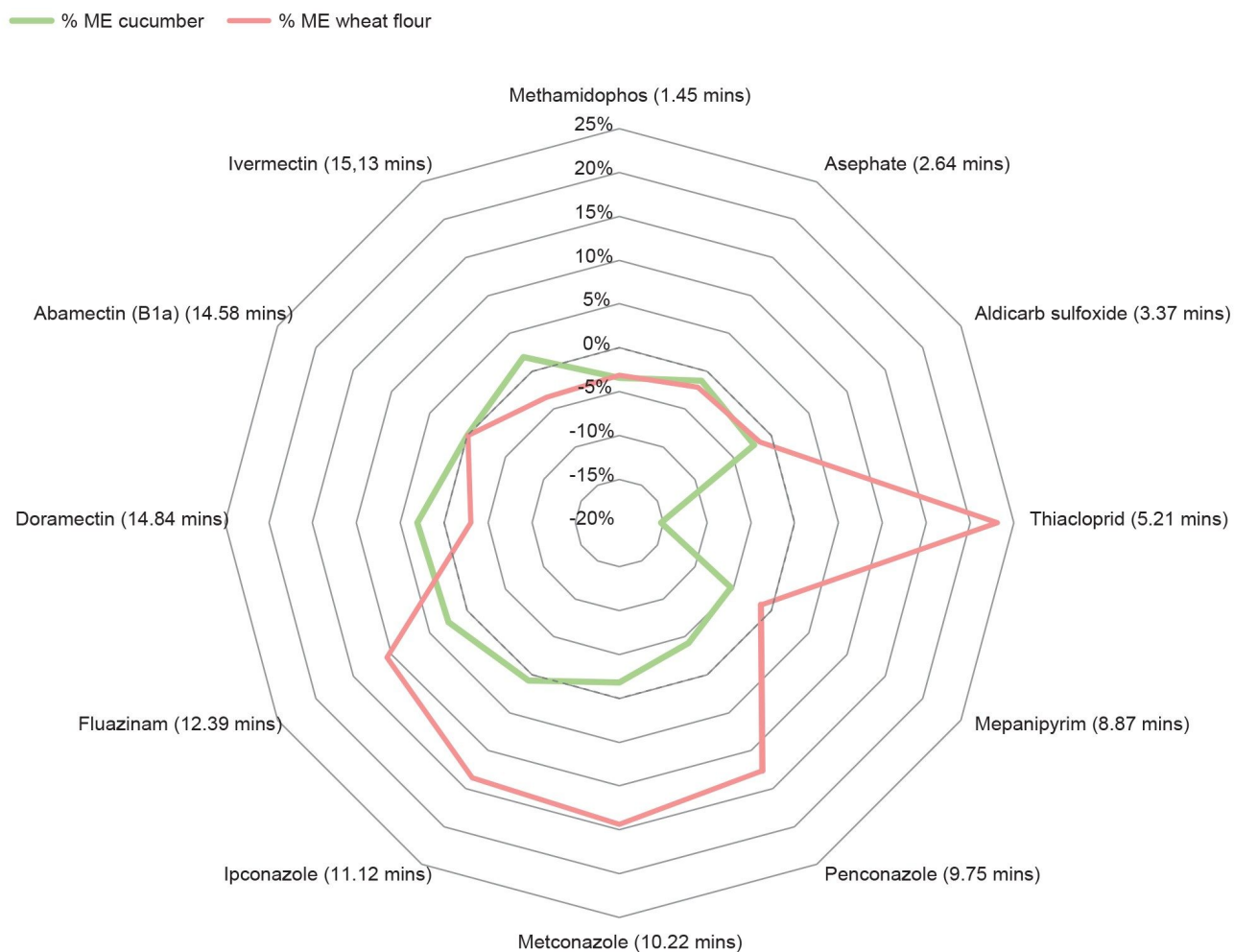
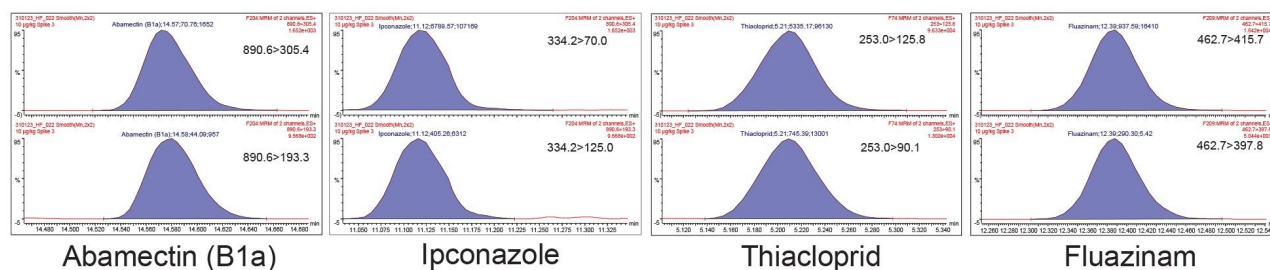


Figure 5. % Matrix effect observed throughout the LC-MS/MS run for cucumber and wheat flour matrices.

The retention times (RT) of all the analytes were found to be within the tolerance of ± 0.1 minute. In general, where two transitions were detected for a pesticide, ion ratios were within the SANTE specified $\pm 30\%$ of the average of calibration standards for the same sequence. Example chromatograms can be seen in Figure 6.

Cucumber 0.01 mg/kg spike level



Wheat Flour 0.01 mg/kg spike level

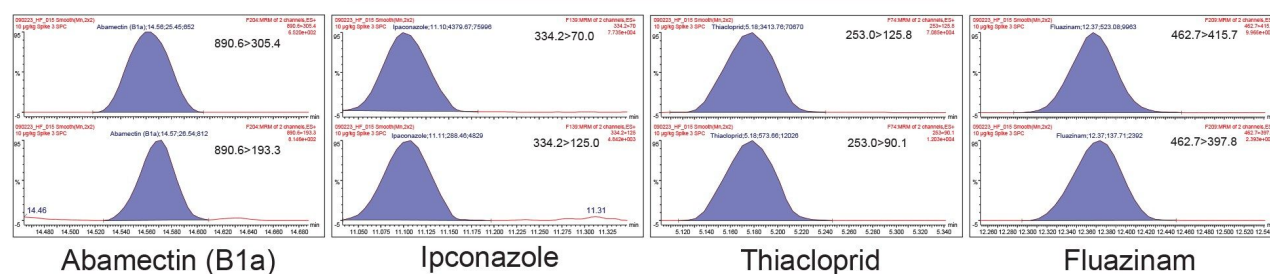


Figure 6. Chromatographic comparison of peak shape at 0.1 mg/kg spike level in cucumber and wheat flour, with quantification transition displayed as the top trace.

The recovery was evaluated using the data from the analysis of the five replicate spikes, at two spike levels, 0.01 mg/kg and 0.1 mg/kg. SANTE guidelines specify an average recovery for each spike level tested to be between 70 and 120%. Demonstrated in Figure 7 and Figure 8, almost all recoveries plotted in both cucumber (94%) and in wheat flour (99%) are within the range of 70% to 120%, following the QuEChERS extraction and Oasis PRiME HLB Plus Short Cartridge clean-up.

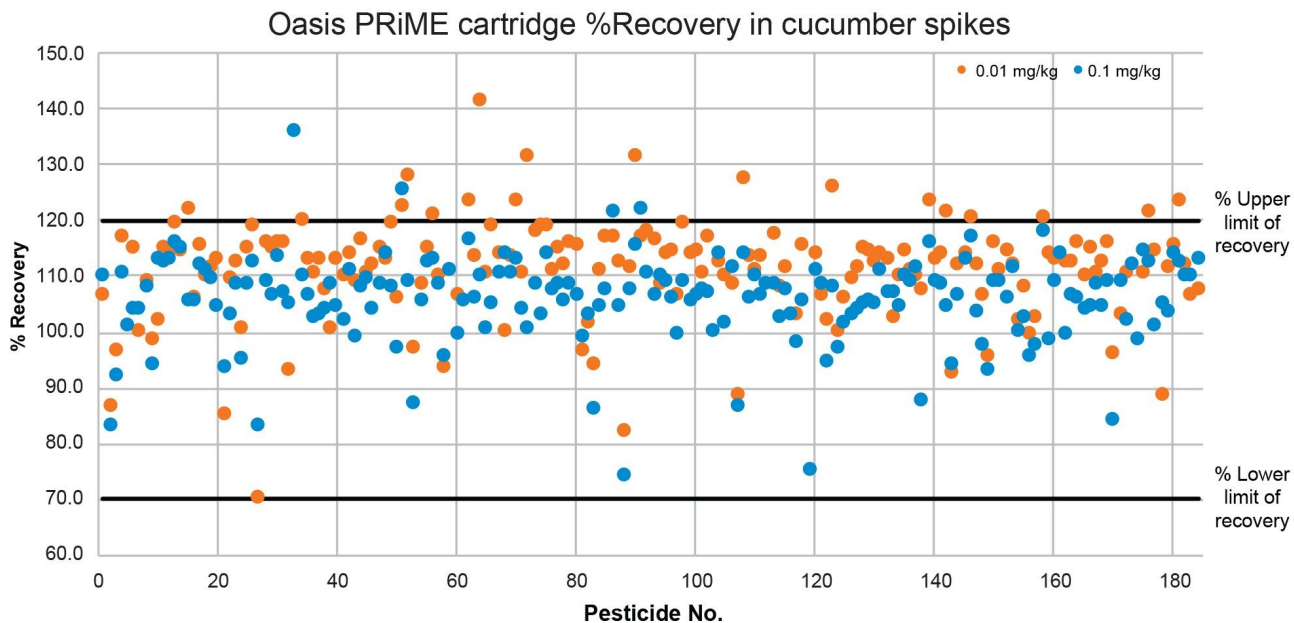


Figure 7. A summary of recoveries in cucumber spikes at 0.01 mg/kg and 0.1 mg/kg for method analytes (each datapoint is a mean of 5 replicates).

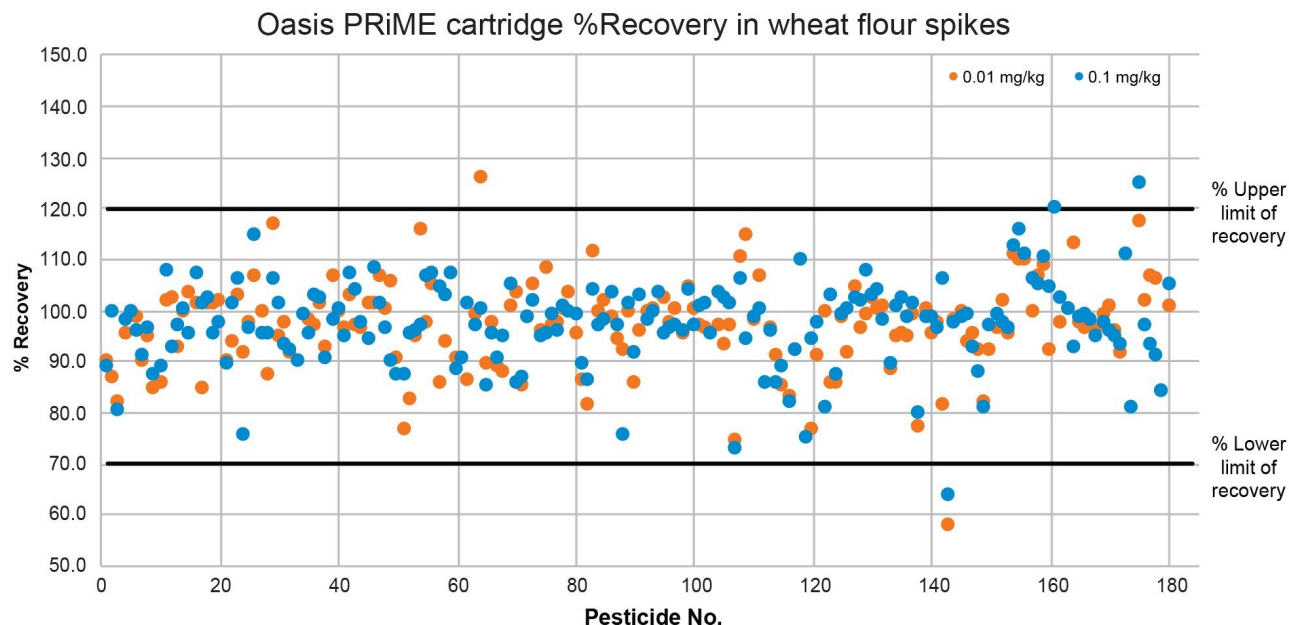


Figure 8. A summary of recoveries in wheat flour spikes at 0.01 mg/kg and 0.1 mg/kg for method analytes (each datapoint is a mean of 5 replicates).

95% of compounds analyzed were detected at the lowest spike level 0.01 mg/kg in cucumber matrix, with recoveries ranging from 70–142% across both spike levels. In wheat flour, 93% of compounds were detected at the corresponding spike level, with recoveries ranging from 58–126% across the two spike levels.

The repeatability (%RSD) of the method was satisfactory. This was assessed from the five replicates across both spike levels at 0.01 mg/kg and 0.1 mg/kg and was within the SANTE guidelines of $\leq 20\%$ deviation in all but three cases: in cucumber at the 0.1 mg/kg spike level for clothianidin, and in wheat flour at the 0.01 mg/kg spike level for fipronil and myclobutanil. As shown in Figure 9 and Figure 10, 99% of analytes were within the %RSD tolerance for cucumber and wheat flour respectively. Mean %RSD across both spike levels was 3.6% in cucumber, and 4.1% in wheat flour.

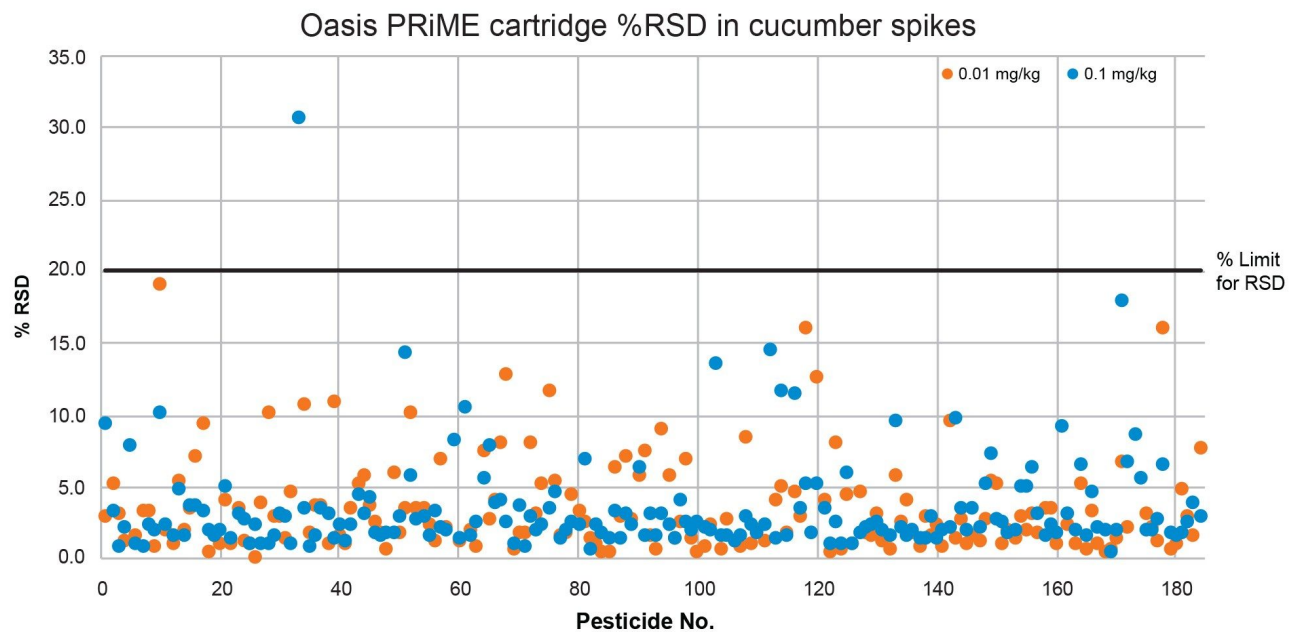


Figure 9. A summary of %RSD in cucumber spikes at 0.01 mg/kg and 0.1 mg/kg for method analytes (each datapoint is a mean of 5 replicates).

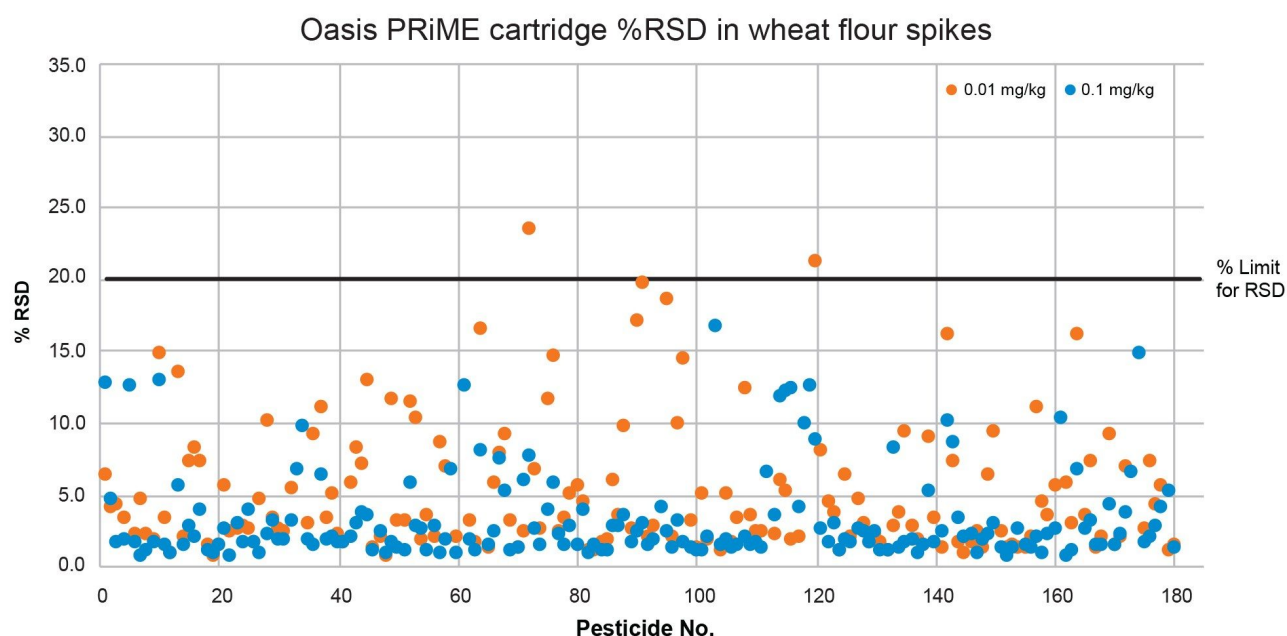


Figure 10. A summary of %RSD in wheat flour spikes at 0.01 mg/kg and 0.1 mg/kg for method analytes (each datapoint is a mean of 5 replicates).

Conclusion

Here we described a sensitive and accurate multi-residue method for the determination of pesticide residues in common matrices of cucumber and wheat flour, using UHPLC-MS/MS with an ACQUITY Arc coupled to a Xevo TQ-S cronos Tandem Quadrupole Mass Spectrometer. The method allows for the reliable quantification at typical EU MRL concentrations of 0.01 mg/kg for over 150 pesticides - in accordance with SANTE guidelines for calibration, sensitivity, and within-lab reproducibility. The CORTECS T3 Column demonstrated comparable run times to traditional UPLC multi-residue methods, with similar method performance. Pass through SPE with Oasis PRiME HLB offers a fast and effective alternative to dSPE, with interferences shown to be of an acceptable level following clean-up.

References

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<https://standards.iteh.ai/catalog/standards/cen/167a30bc-edf9-4cf8-b96b-cabd932f2f02/en-15662-2018>>
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Appendix

Compound name	Ion mode	Precursor	Product	CV	CE	RT
3-Hydroxycarbofluran	ES+	238.1	181	25	10	4.6
3-Hydroxycarbofluran	ES+	238.1	163	25	15	
Abamectin (B1a)	ES+	890.6	567.4	15	11	14.58
Abamectin (B1a)	ES+	890.6	305.2	15	25	
Acephate	ES+	184.1	143	5	10	2.64
Acephate	ES+	184.1	49	5	20	
Acetamiprid	ES+	223	126	25	20	4.8
Acetamiprid	ES+	223	56.1	25	15	
Aldicarb	ES+	213.1	89.1	5	20	5.29
Aldicarb	ES+	213.1	47	5	25	
Aldicarb sulfone	ES+	223	86	35	15	3.64
Aldicarb sulfone	ES+	223	148	35	10	
Aldicarb sulfoxide	ES+	207	132	20	5	3.38
Aldicarb sulfoxide	ES+	207	89	20	15	
Ametryn	ES+	228.1	186.1	10	20	6.74
Ametryn	ES+	228.1	68.1	10	35	
Aminocarb	ES+	209	137	25	25	3.27
Aminocarb	ES+	209	152	25	15	
Amitraz	ES+	294.1	122	25	30	4.08
Amitraz	ES+	194.1	163	25	15	
Azoxystrobin	ES+	404.1	372	12	25	8.73
Azoxystrobin	ES+	404.1	329	12	30	
Benalaxyl	ES+	326.1	148	25	20	10.52
Benalaxyl	ES+	326.1	91	25	30	
Bendiocarb	ES+	224.1	167	15	10	6.02
Bendiocarb	ES+	224.1	109	15	15	
Benzoximate	ES+	364	199.1	20	10	11.26
Benzoximate	ES+	364	105	20	25	
Bifenazate	ES+	301.1	170	20	20	9.17
Bifenazate	ES+	301.1	198	20	5	
Bromuconazole I	ES+	376	158.9	30	30	9.19
Bromuconazole I	ES+	376	70.1	30	20	
Bromuconazole II	ES+	376	158.9	30	45	9.45
Bromuconazole II	ES+	376	70.1	30	40	
Bupirimate	ES+	317	166	15	25	9.44
Bupirimate	ES+	317	108	15	25	
Buprofezin	ES+	306.1	201	10	10	12.16
Buprofezin	ES+	306.1	115.9	10	20	
Butafenacil	ES+	492	331	15	25	10.02
Butafenacil	ES+	492	180	15	35	
Butocarboxim	ES+	213	75	25	15	5.15
Butocarboxim	ES+	213	116	25	10	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Butoxycarboxim	ES+	223	106	30	10	3.57
Butoxycarboxim	ES+	223	166	30	5	
Carbaryl	ES+	202	145	20	10	6.35
Carbaryl	ES+	202	117	20	25	
Carbendazim	ES+	192.1	160.1	35	15	3.79
Carbendazim	ES+	192.1	132.1	35	30	
Carbetamide	ES+	237	192	5	10	5.49
Carbetamide	ES+	237	118	5	15	
Carbofuran	ES+	222.1	165.1	15	10	6.08
Carbofuran	ES+	222.1	123	15	20	
Carboxin	ES+	236	143	15	15	6.43
Carboxin	ES+	236	87	15	25	
Carfentrazone Ethyl	ES+	412	346	45	24	10.27
Carfentrazone Ethyl	ES+	412	266	45	18	
Chlorantraniliprole	ES+	484	452.9	10	15	7.96
Chlorantraniliprole	ES+	484	285.9	10	15	
Chloroxuron	ES+	291.1	72	40	20	8.66
Chloroxuron	ES+	291.1	164.1	40	15	
Chlortoluron	ES+	213	72	15	15	6.51
Chlortoluron	ES+	213	46	15	15	
Clethodim I	ES+	360	164	25	20	11.8
Clethodim I	ES+	360	268.1	25	10	
Clothianidin	ES+	250	169	25	10	4.45
Clothianidin	ES+	250	132	25	15	
Cyazofamid	ES+	325	107.9	25	15	10.02
Cyazofamid	ES+	325	261	25	10	
Cycluron	ES+	199	89.1	10	15	6.85
Cycluron	ES+	199	69.2	10	20	
Cyproconazole I	ES+	292.1	70.2	20	20	8.51
Cyproconazole I	ES+	292.1	125.1	20	30	
Cyproconazole II	ES+	292.1	70.2	22	20	8.68
Cyproconazole II	ES+	292.1	125.1	22	30	
Cyprodinil	ES+	226	93	10	35	8.98
Cyprodinil	ES+	226	108	10	25	
Desmedipham	ES+	301	182	16	10	7.91
Desmedipham	ES+	301	136	16	25	
Diclobutrazol	ES+	328	70	30	20	9.61
Diclobutrazol	ES+	328	158.9	30	35	
Diclotophos	ES+	238	112	25	10	4.19
Diclotophos	ES+	238	193	25	10	
Diethofencarb	ES+	268.1	226	5	10	8.13
Diethofencarb	ES+	268.1	124	5	30	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Difenoconazole I	ES+	406.1	251.1	35	25	10.91
Difenoconazole I	ES+	406.1	111.1	35	35	
Difenoconazole II	ES+	406.1	251.1	35	25	10.97
Difenoconazole II	ES+	406.1	111.1	35	35	
Diflubenzuron	ES+	311	158	25	10	9.45
Diflubenzuron	ES+	311	141.1	25	15	
Dimethoate	ES+	230	124.8	15	10	4.67
Dimethoate	ES+	230	198.8	15	20	
Dimethomorph I	ES+	388.1	300.9	25	20	8.26
Dimethomorph I	ES+	388.1	165	25	30	
Dimethomorph II	ES+	388.1	300.9	10	20	8.54
Dimethomorph II	ES+	388.1	165	10	30	
Dimoxystrobin	ES+	327.1	116.1	15	20	9.87
Dimoxystrobin	ES+	327.1	205.2	15	10	
Diniconazole	ES+	326.1	70.2	20	25	10.36
Diniconazole	ES+	326.1	159	20	30	
Dinotefuran	ES+	203	129	15	10	3.41
Dinotefuran	ES+	203	113.1	15	10	
Dioxacarb	ES+	224.1	123.1	20	15	4.69
Dioxacarb	ES+	224.1	167.1	20	10	
Diuron	ES+	233	72.1	35	20	6.93
Diuron	ES+	233	46.3	35	15	
Doramectin	ES+	916.6	331.2	10	25	14.84
Doramectin	ES+	916.6	593.4	10	15	
Emamectin Benzoate B1a	ES+	886.6	158	50	35	12.61
Emamectin Benzoate B1a	ES+	886.6	126	50	30	
Epoxiconazole	ES+	330	121	30	20	9.25
Epoxiconazole	ES+	330	101	30	35	
Eprinomectin	ES+	914.6	186	10	35	14.29
Eprinomectin	ES+	914.6	144	10	40	
Etaconazole	ES+	328.1	159	20	25	9.02
Etaconazole	ES+	328.1	205	20	15	
Ethiofencarb	ES+	226.1	107	15	15	6.59
Ethiofencarb	ES+	226.1	164	15	10	
Ethiprole	ES+	414.1	350.9	15	25	8.5
Ethiprole	ES+	414.1	396.9	15	10	
Ethirimol	ES+	210.1	140	35	20	4.79
Ethirimol	ES+	210.1	98	35	25	
Ethofumesate	ES+	287.1	121.1	40	15	8.67
Ethofumesate	ES+	287.1	259.1	40	10	
Etoxazole	ES+	360.1	57.2	15	25	13.12
Etoxazole	ES+	360.1	141	15	25	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Fenamidone	ES+	312.1	92	15	25	8.56
Fenamidone	ES+	312.1	236.1	15	15	
Fenarimol	ES+	331	81	25	30	8.82
Fenarimol	ES+	331	268	25	25	
Fenazaquin	ES+	307.2	57.2	35	20	13.11
Fenazaquin	ES+	307.2	161	35	15	
Fenbuconazole	ES+	337	70.1	40	20	9.72
Fenbuconazole	ES+	337	125	40	30	
Fenhexamid	ES+	302.1	97.2	40	25	9.2
Fenhexamid	ES+	302.1	55.3	40	35	
Fenobucarb	ES+	208	94.9	20	15	7.86
Fenobucarb	ES+	208	152	20	10	
Fenoxycarb	ES+	302.1	88	25	20	9.69
Fenoxycarb	ES+	302.1	116.1	25	10	
Fenpropimorph	ES+	304.2	147.1	35	30	7.94
Fenpropimorph	ES+	304.2	57.2	35	30	
Fenpyroximate	ES+	422.2	366.1	20	20	13.3
Fenpyroximate	ES+	422.2	138.1	20	30	
Fenuron	ES+	165	71.9	18	15	4.48
Fenuron	ES+	165	45.9	18	15	
Fipronil	ES-	435.1	250	5	30	10.29
Fipronil	ES-	435.1	330	5	40	
Fluazinam	ES-	462.7	397.8	5	25	12.39
Fluazinam	ES-	462.7	415.7	5	25	
Flubendiamide	ES+	683	408	5	5	10.67
Flubendiamide	ES+	683	274	5	30	
Flufenacet	ES+	364	152.1	15	20	9.61
Flufenacet	ES+	364	194.1	15	10	
Flufenoxuron	ES+	489.1	158	35	20	13.13
Flufenoxuron	ES+	489.1	141	35	45	
Fluometuron	ES+	233.1	72.2	35	20	6.49
Fluometuron	ES+	233.1	46.4	35	20	
Fluoxastrobin	ES+	459	427	22	15	9.8
Fluoxastrobin	ES+	459	188	22	35	
Flusilazole	ES+	316	247	25	20	9.62
Flusilazole	ES+	316	165	25	25	
Flutolanil	ES+	324.1	262.1	35	20	9.18
Flutolanil	ES+	324.1	65	35	35	
Flutriafol	ES+	302.1	70.2	25	15	6.84
Flutriafol	ES+	302.1	123.1	25	30	
Forchlorfenuron	ES+	248.1	129	35	15	6.85
Forchlorfenuron	ES+	248.1	93	35	35	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Formetanate-HCl	ES+	222	165	10	15	3.26
Formetanate-HCl	ES+	222	46	10	25	
Fuberidazole	ES+	185	157	25	20	4.2
Fuberidazole	ES+	185	156	25	25	
Furalaxyl	ES+	302.1	95	15	25	8.29
Furalaxyl	ES+	302.1	242.1	15	15	
Furathiocarb	ES+	383.2	194.9	30	15	12.33
Furathiocarb	ES+	383.2	252	30	10	
Halofenozide	ES+	331.1	104.9	15	15	8.41
Halofenozide	ES+	331.1	275	15	5	
Hexaconazole	ES+	314	70.1	35	20	10
Hexaconazole	ES+	314	159	35	25	
Hexythiazox	ES+	353	168.1	15	25	12.63
Hexythiazox	ES+	353	228.1	15	15	
Hydramethylnon	ES+	495.1	323.2	60	30	11.77
Hydramethylnon	ES+	495.1	151.1	60	35	
Imazalil	ES+	297	159	35	20	6.66
Imazalil	ES+	297	69	35	20	
Imidacloprid	ES+	256.1	175.1	20	20	4.53
Imidacloprid	ES+	256.1	209.1	20	15	
Indoxacarb	ES+	528	150	35	25	11.73
Indoxacarb	ES+	528	203	35	30	
Ipconazole	ES+	334.2	70	25	25	11.12
Ipconazole	ES+	334.2	125	25	25	
Iprovalicarb	ES+	321.1	119.1	15	20	8.94
Iprovalicarb	ES+	321.1	203.1	15	10	
Isocarbofos	ES+	291.1	121.1	15	30	7.64
Isocarbofos	ES+	291.1	231.1	15	15	
Isoprocarb	ES+	194.1	95.1	25	15	6.89
Isoprocarb	ES+	194.1	137.1	25	10	
Isoproturon	ES+	207	72	15	15	6.89
Isoproturon	ES+	207	46.1	15	15	
Ivermectin	ES+	892.6	551.4	5	25	15.13
Ivermectin	ES+	892.6	569.4	5	14	
Kresoxim-Methyl	ES+	314.1	116	5	15	10.13
Kresoxim-Methyl	ES+	314.1	206	5	5	
Mandipropamid	ES+	412	328	30	15	9.06
Mandipropamid	ES+	412	125	30	35	
Mefenacet	ES+	299	148	10	15	8.9
Mefenacet	ES+	299	120	10	25	
Mepanipyrim	ES+	224.1	106	45	25	8.87
Mepanipyrim	ES+	224.1	77	45	35	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Mepronil	ES+	270.1	119	25	25	9.01
Mepronil	ES+	270.1	91	25	35	
Mesotrione	ES+	340.1	228.1	40	15	5.55
Mesotrione	ES+	340.1	104	40	30	
Metalaxyl	ES+	280.1	220.1	5	15	7.19
Metalaxyl	ES+	280.1	192.1	5	20	
Metconazole	ES+	320.1	70	20	25	10.22
Metconazole	ES+	320.1	125	20	30	
Methabenzthiazuron	ES+	222	165	15	15	6.42
Methabenzthiazuron	ES+	222	150	15	30	
Methamidophos	ES+	142	93.9	20	15	1.46
Methamidophos	ES+	142	124.9	20	15	
Methiocarb	ES+	226	121	25	20	7.94
Methiocarb	ES+	226	169	25	10	
Methomyl	ES+	163	88	5	10	3.8
Methomyl	ES+	163	106	5	10	
Methoprotryne	ES+	272.2	170.2	25	30	6.91
Methoprotryne	ES+	272.2	198.2	25	25	
Methoxyfenozide	ES+	369.1	149.1	20	15	9.23
Methoxyfenozide	ES+	369.1	313.2	20	10	
Metobromuron	ES+	259.1	170	20	20	6.85
Metobromuron	ES+	259.1	148.1	20	15	
Metribuzin	ES+	215	89	30	20	5.76
Metribuzin	ES+	215	131	30	20	
Mevinphos I	ES+	225.1	127.1	15	15	4.6
Mevinphos I	ES+	225.1	193.1	15	10	
Mevinphos II	ES+	225.1	127.1	25	15	5.01
Mevinphos II	ES+	225.1	193.1	25	5	
Mexacarbate	ES+	223.2	166.1	5	15	4.41
Mexacarbate	ES+	223.2	151	5	25	
Monocrotophos	ES+	224.1	127.1	25	15	3.94
Monocrotophos	ES+	224.1	98.1	25	10	
Monolinuron	ES+	215	126	15	15	6.53
Monolinuron	ES+	215	99	15	30	
Moxidectin	ES+	640.5	528.4	15	10	14.78
Moxidectin	ES+	640.5	498.3	15	10	
Myclobutanil	ES+	289.1	124.9	25	30	8.86
Myclobutanil	ES+	289.1	150.9	25	25	
Neburon	ES+	275	88	40	15	9.81
Neburon	ES+	275	57	40	20	
Nitenpyram	ES+	271.1	125.9	10	30	3.79
Nitenpyram	ES+	271.1	224.9	10	10	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Nuarimol	ES+	315	81.1	30	15	7.79
Nuarimol	ES+	315	252	30	20	
Omethoate	ES+	214.1	125.1	10	20	3.12
Omethoate	ES+	214.1	183.1	10	10	
Oxadixyl	ES+	279.1	102	25	30	5.75
Oxadixyl	ES+	279.1	132.2	25	30	
Oxamyl	ES+	237.1	72	5	10	3.72
Oxamyl	ES+	237.1	90	5	10	
Paclobutrazol	ES+	294.1	125.1	35	35	8.24
Paclobutrazol	ES+	294.1	70.2	35	20	
Penconazole	ES+	284	70.1	22	15	9.75
Penconazole	ES+	284	159	22	25	
Pencycuron	ES+	329.1	218	40	15	11.14
Pencycuron	ES+	329.1	125	40	25	
Phenmedipham	ES+	301	168	35	10	8
Phenmedipham	ES+	301	136	35	20	
Picoxystrobin	ES+	368	145.1	5	25	10.33
Picoxystrobin	ES+	368	205.1	5	10	
Piperonyl butoxide	ES+	356.3	176.9	12	10	12.28
Piperonyl butoxide	ES+	356.3	119	12	35	
Pirimicarb	ES+	239.1	72	5	20	5.23
Pirimicarb	ES+	239.1	182.1	5	15	
Procloraz	ES+	376	307.1	15	15	10.16
Procloraz	ES+	376	70.1	15	25	
Promecarb	ES+	208.1	109	20	15	8.24
Promecarb	ES+	208.1	151	20	10	
Prometon	ES+	226.2	86.3	30	30	6
Prometon	ES+	226.2	184.3	30	20	
Prometryn	ES+	242	158	15	25	7.88
Prometryn	ES+	242	200.1	15	20	
Propamocarb	ES+	189.1	102	15	15	3.23
Propamocarb	ES+	189.1	74.1	15	20	
Propargite	ES+	368.2	231.1	5	10	13.19
Propargite	ES+	368.2	175.1	5	15	
Propiconazole	ES+	342	69	40	20	10.14
Propiconazole	ES+	342	159	40	25	
Propoxur	ES+	210	110.9	5	15	5.99
Propoxur	ES+	210	92.9	5	15	
Prothioconazole	ES+	344	326	20	10	10.14
Prothioconazole	ES+	344	189	20	20	
Pymetrozine	ES+	218	105	35	20	3.21
Pymetrozine	ES+	218	79	35	35	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Pyracarbolid	ES+	218.1	125.1	20	20	6.21
Pyracarbolid	ES+	218.1	97.1	20	30	
Pyraclostrobin	ES+	388.1	163	15	25	10.96
Pyraclostrobin	ES+	388.1	193.9	15	10	
Pyridaben	ES+	365.1	147.1	15	25	13.79
Pyridaben	ES+	365.1	309.1	15	10	
Pyrimethanil	ES+	200	107	35	25	7.09
Pyrimethanil	ES+	200	82	35	25	
Pyriproxifen	ES+	322.1	96	15	15	12.31
Pyriproxifen	ES+	322.1	227.1	15	10	
Quinoxifen	ES+	308	197	20	30	11.94
Quinoxifen	ES+	308	161.9	20	35	
Rotenone	ES+	395.1	213.1	35	25	9.83
Rotenone	ES+	395.1	192.1	35	20	
Secbumeton	ES+	226.2	170.2	10	20	6.15
Secbumeton	ES+	226.2	100.2	10	25	
Siduron	ES+	233.2	93.8	40	20	7.9
Siduron	ES+	233.2	137	40	15	
Simetryn	ES+	214	124	15	20	5.77
Simetryn	ES+	214	95.9	15	25	
Spinetoram	ES+	748.5	142.2	30	30	11.82
Spinetoram	ES+	748.5	98.1	30	35	
Spinosad A	ES+	732.6	142	15	30	10.92
Spinosad A	ES+	732.6	98.1	15	35	
Spinosad D	ES+	746.5	142	15	30	11.51
Spinosad D	ES+	746.5	98.1	15	35	
Spirodiclofen	ES+	411.1	71.2	35	15	13.86
Spirodiclofen	ES+	411.1	313	35	10	
Spiromesifen	ES+	371.1	273.1	30	5	13.59
Spiromesifen	ES+	371.1	255.1	30	25	
Spirotetramat	ES+	374	302	15	30	9.16
Spirotetramat	ES+	374	330	15	15	
Spiroxamine	ES+	298	144	5	20	8.08
Spiroxamine	ES+	298	100	5	30	
Sulfentrazone	ES+	387	145.8	50	35	6.58
Sulfentrazone	ES+	387	307	50	30	
Tebuconazole	ES+	308.1	70.1	15	20	9.76
Tebuconazole	ES+	308.1	125	15	35	
Tebufenozide	ES+	353.1	133	15	20	10.12
Tebufenozide	ES+	353.1	297.1	15	10	
Tebufenpyrad	ES+	334	117	25	25	11.9
Tebufenpyrad	ES+	334	145	25	25	

Compound name	Ion mode	Precursor	Product	CV	CE	RT
Tebuthiuron	ES+	229.1	172	20	15	5.66
Tebuthiuron	ES+	229.1	116	20	25	
Temephos	ES+	466.8	125	45	30	12.71
Temephos	ES+	466.8	418.9	45	20	
Terbumeton	ES+	226.1	170.1	22	15	6.15
Terbumeton	ES+	226.1	114.1	22	25	
Terbutryn	ES+	242.1	186.1	15	20	8.07
Terbutryn	ES+	242.1	91	15	25	
Tetraconazole	ES+	372	70.1	15	20	9.31
Tetraconazole	ES+	372	159	15	25	
Thiabendazole	ES+	202	174.9	20	25	4.04
Thiabendazole	ES+	202	130.9	20	30	
Thiacloprid	ES+	253	126	25	20	5.2
Thiacloprid	ES+	253	90.1	25	35	
Thiamethoxam	ES+	292	211.2	15	10	4.08
Thiamethoxam	ES+	292	132	15	20	
Thidiazuron	ES+	221	101.9	15	15	5.76
Thidiazuron	ES+	221	93.9	15	15	
Thiobencarb	ES+	258.1	125	15	15	10.7
Thiobencarb	ES+	258.1	89	15	35	
Thiofanox	ES+	219	57	30	5	6.64
Thiofanox	ES+	219	76	30	5	
Thiophanate methyl	ES+	343	151	30	20	6.06
Thiophanate methyl	ES+	343	93	30	35	
Triadimefon	ES+	294.1	69.3	30	20	8.85
Triadimefon	ES+	294.1	197.2	30	15	
Triadimenol	ES+	296.1	70.2	20	10	8.47
Triadimenol	ES+	296.1	227.1	20	10	
Trichlorfon	ES+	257	109	30	15	4.4
Trichlorfon	ES+	257	79	30	30	
Tricyclazole	ES+	190	163	35	20	5.03
Tricyclazole	ES+	190	136	35	25	
Trifloxystrobin	ES+	409	186	15	15	11.71
Trifloxystrobin	ES+	409	145	15	40	
Triflumizole	ES+	346	277.9	15	20	11.03
Triflumizole	ES+	346	73.1	15	15	
Triticonazole	ES+	318.1	70.1	25	20	8.88
Triticonazole	ES+	318.1	124.9	25	30	
Vamidothion	ES+	288	146	10	10	4.55
Vamidothion	ES+	288	118	10	25	
Zoxamide	ES+	336	187.1	25	20	10.57
Zoxamide	ES+	336	159	25	35	

Appendix 1. Summary of method transitions.

Featured Products

[ACQUITY Arc System <https://www.waters.com/134844390>](https://www.waters.com/134844390)

[Xevo TQ-S cronos Tandem Quadrupole Mass Spectrometry <https://www.waters.com/waters/en_US/Xevo-TQ-S-cronos-Triple-Quadrupole-Mass-Spectrometry/nav.htm?cid=135027354>](https://www.waters.com/waters/en_US/Xevo-TQ-S-cronos-Triple-Quadrupole-Mass-Spectrometry/nav.htm?cid=135027354)

[MassLynx MS Software <https://www.waters.com/513662>](https://www.waters.com/513662)

[TargetLynx <https://www.waters.com/513791>](https://www.waters.com/513791)

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