LABORATORY INFORMATION BULLETIN

Pesticide matrix extension validation for grasshoppers/chapulines using QuEChERS extraction with GC-MS/MS and LC-MS/MS detection
CARTS Project IR01672

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ABSTRACT

A method for quantifying 304 pesticides and industrial chemicals has been validated to include two new matrices: grasshoppers and chapulines. The method was based on existing SOPs. These methods use QuEChERS extraction, followed by GC-MS/MS analysis (for 144 analytes) and LC-MS/MS separation and detection (for 161 analytes). There were 33 analytes that met quality control criteria for both GC-MS/MS and LC-MS/MS. There were 71 analytes that did not meet QC criteria for either GC-MS/MS or LC-MS/MS. One source was a specific type of grasshoppers referred to as chapulines. The accuracies were evaluated by measuring spike recoveries on three different brands of grasshoppers corresponding to the normal action level and minimum reporting level of 10 ng/g; half this level at 5 ng/g; and twice this level at 20 ng/g. The average recovery for all analytes on the GC-MS/MS was 89%. The percent relative standard deviations (RSDs) ranged from 3-29%. The average %STD was 11.0%. The average recovery for all analytes on the LC-MS/MS was 84%. The average recovery for all analytes on the LC-MS/MS was 84%. The percent relative standard deviations (RSDs) ranged from 6-29%. The average %STD was 15.1%. In both GC-MS/MS and LC-MS/MS methods, there were some compounds showing low level interferences, either matrix or instrument related. The average relative percent difference (RPD) for all analytes on the GC-MS/MS was 16%. The average RPD for all analytes on the LC-MS/MS was 17%. To establish the ruggedness and/or robustness of the method, analyses of a single matrix were done by three different analysts over four days.

1. INTRODUCTION

The FDA Kansas City Lab (KCL) has been analyzing hundreds of different foods and beverages for pesticides and industrial chemicals as part of the Total Diet Study and regulatory samples (1) and regulatory samples. The analytes that are either inherently volatile or can be volatilized at elevated temperatures have been separated by gas chromatography (GC) and detected by tandem mass spectrometry (MS/MS). The analytes that are neither volatile nor stable at elevated temperatures were separated by liquid chromatography (LC) and detected by MS/MS. In addition, some analytes presented special problems (2-7).

The analyses performed in the current study were done using validated methods that have been written as SOPs (8, 9, 10, 14). This is an extension of these methods into a new matrix – grasshoppers, also known by some as chapulines. The KCL was asked to perform analysis of imported grasshoppers for human consumption, for both metals and pesticides. An historical case of lead contamination of this food type prompted this survey, as noted in a December 2017 Import Bulletin. A matrix extension validation was deemed necessary for pesticides analysis due to the unusual nature of this food matrix. This validation was performed January – February 2018. There is also 2017 Import Bulletin.

The QuEChERS extraction for non-fat, low-fat, and fatty matrices for multi-residues has been validated previously using both the LC-MS/MS and GC-MS/MS detectors. These validations covered multiple matrices to cover different commodity groups as defined by the "Guidelines for the Validation of Chemical Methods for the FDA FVM Program," version 2.0, April 2015. This method matrix extension was designed specifically for grasshoppers using the Level Two criteria validation defined under Appendix 3, part C for a single matrix. Quality control (QC) criteria, as defined in the study plan, were for accuracy, precision, uncertainty, selectivity, LOD/LOQ, i.e., reporting level in this case; and ruggedness/robustness.

Up to 194 analytes in other matrices have been quantified by GC-MS/MS, while 229 other analytes have been quantified by LC-MS/MS. Normally, some of the compounds are not reported due to several concerns, included stability, matrix interferences, or other factors. Several potential LC-MS/MS target compounds were excluded from this matrix extension validation Study. Compounds such as phosmet and thiophanate-methyl have limited stability in the standard solution and were not evaluated in this matrix extension. Avermectin, butachlor, dodine, emamectin-benzoate, fuberidazole, halofenozide, isocarbophos, ivermectin, methfuroxam, metsulfuron-methyl and trichlorfon provided such poor recoveries they were not evaluated in this matrix extension. Azinphos-methyl and cyromazine were not evaluated for this study due to chromatographic problems. No GC-MS/MS compounds were eliminated as targets although some had low recoveries.

2. EXPERIMENTAL

Equipment

A. Equipment and Consumables

Equivalents may be substituted, as necessary

1. Homogenizer (Spex Sample Prep GenoGrinder 2000 or 2010)
2. Benchtop centrifuge (Thermo Scientific Heraeus Multifuge X3R)
3. Vortex mixer
4. Evaporator, e.g. Turbovap, N-Evap, VacEvap
5. SPE Positive Pressure Manifold (UCT # MFPPM16)
6. Volumetric conical glass tube, 15 mL, calibrated
7. Pipettors
8. Disposable pipette tips
9. Glass centrifuge tubes, 15 mL
10. Polypropylene centrifuge tubes, 50 mL
11. Glass transfer pipettes
12. Disposable syringes
13. Syringeless PTFE filter device, 0.2-0.45 µm
14. Glass homogenizing beads

B. Reagents

All reagents are reagent grade or higher, unless otherwise noted. Equivalents may be substituted, as necessary.

1. Acetonitrile
2. Toluene
3. Acetone
4. $MgSO_4$ (6g) and NaCl (1.5g) packet (UCT ECMSSCFS-MP)
5. Ammonium formate, HPLC grade
6. Formic Acid, LC/MS spec grade
7. Water, HPLC grade
8. Methanol, LCMS grade
9. LCMS dilution solvent: 4 mM ammonium formate in water
10. SPE eluent: 25% Toluene in acetone

11. LCMS dispersive SPE cleanup centrifuge tube: 2 mL tube containing 100-200 mg anhydrous MgSO4 and 50-150 mg PSA (UCT #CUMPS2CT)

12. GC SPE cleanup column: 6 mL SPE column containing 200-500 mg each of PSA, GCB and lipophilic, e.g. C18 or polymer, sorbents (Agela # TPT0006);

13. Z-Sep tube: 12 mL centrifuge tube containing Supelco Supel QuE Z-Sep+ 500 mg (Supelco #55296-U)

14. Internal standard (GC): 0.500 µg/ml of acenaphthene-d_{10} (Aldrich 99.8%), chrysene-d_{12} (Supelco 98.6%), and pyridaben-d_{13} (CDN Isotopes 99.5%) in acetone.

C. Samples

The sources for the grasshoppers are shown in Table 1.

Table 1. Sources of Grasshoppers for Matrix Extension Validation

Source #	**Company**	**Description**
Source 1	Newport Jerky Company	Edible Grasshoppers
Source 2	Edibleinsects.com	Chapulines Sazonados
Source 3	Edibleinsects.com	Edible Grasshoppers

The only ingredients listed for sources 1 and 3 were grasshoppers and salt. The ingredients listed for source 2 include lemon, salt, garlic, and chili, in addition to chapulines. Reportable levels of pesticides were found in only one of the three sources. Triazophos and rotenone were present in source 2 at about 10 ppb. This source had several other residues below 10 ppb.

D. Extraction

1. Into a 50 mL centrifuge tube, weigh 5.0 g of sample. To low moisture, high sugar, and/or high fat items add 10 mL deionized water. After adding water to samples, place in GenoGrinder at 1000 strokes/min for 1 minute and allow sample to sit for ~15 min.

2. To a separate tube, add 10 mL of deionized water to serve as a blank.

3. Add 25 mL acetonitrile.

4. Add two glass beads.

5. Cap tube, ensuring there are no leaks, and shake in the GenoGrinder at 1000 strokes/min for fifteen minutes.

6. Add 6 g anhydrous $MgSO_4$ + 1.5 g NaCl (Reagent 4) and shake in the GenoGrinder at 1000 strokes/min for the length of time specified above.

7. Centrifuge to separate phases, e.g. about 10-20 min @ 4500 rpm. Sample is ready for LC or GC cleanup steps, below.

E. Cleanup

1. Cleanup

1.1 LC dilution for injection –

No cleanup:

A. Dilute 200 µL extract + 200 µL LCMS dilution solvent (Reagent 9).

B. Vortex for approx. 30 seconds.

C. Filter through 0.2-0.45 µm PTFE filter device into an autosampler vial.

D. Final concentration: 0.1 g/mL

1.2. Optional LC cleanup:

A. Add ~1 mL extract to 2 mL dispersive SPE centrifuge tube (Reagent 8).

B. Vortex for 30 seconds.

C. Centrifuge to separate phases, about 5 min @ 4500 rpm.

D. Dilute 200 µL extract + 200 µL LCMS dilution solvent (Reagent 6).

E. Vortex for approx. 30 seconds.

F. Filter through 0.2-0.45 µm PTFE filter device into an autosampler vial.

G. Final concentration: 0.1 g/mL

2. GC Cleanup

2.1. For samples containing less than 6% fat:

A. In a glass centrifuge tube, concentrate 5.0 mL extract to less than 2 mL using an evaporator at 40-50 °C. Do not take sample to dryness

B. Precondition 6 mL SPE column (Reagent 12) with 2 mL SPE eluent (Reagent 10); discard the eluent.

C. Transfer concentrated extract to the preconditioned column and use manifold pressure to elute the sample extract until it reaches the top of the SPE sorbent.

D. Add ~1 mL acetone to the centrifuge tube, vortex and add to the column. Use manifold pressure to elute the solvent until it reaches the top of the SPE sorbent.

E. Add ~1 mL SPE eluent to the centrifuge tube, vortex and add to the column. Use manifold pressure to elute the eluent until it reaches the top of the SPE sorbent.

F. Elute using SPE eluent at 2 mL/min to a total volume of ~15 mL collecting in a 15 mL calibrated, conical volumetric tube.

G. Concentrate the eluent to ~100 µL using an evaporator at 40-50 °C. Do not take sample to dryness.

H. Dilute sample concentration to 1.0 g/mL by bringing to 1.0 mL with acetone.

I. Transfer sample to autosampler vial.

2. Samples greater than 6% fat: Although we didn't determine the fat content we did combine the contents from 2 tubes.

A. Add approximately 7 mL extract to 500 mg Z-Sep tube (Reagent 13).

B. Cap tube, ensuring there are no leaks, and vortex ~1 min.

C. Centrifuge approximately 10 min @ 4500 rpm.

D. Continue from GC cleanup step 2.1.A.

F. Residue Determination

Residues are analyzed using GC-MS/MS and LC-MS/MS. See attachment A for GC instrument parameters and attachment B for LC instrument parameters. Refer to attachment C for GC instrument settings and attachment D for LC instrument settings.

1. GC Equipment

A. Agilent Model 7000 GC/MS Triple Quad

B. Agilent 7890 gas chromatograph

C. Agilent 7693 Autosampler

D. Agilent 7693A Autoinjector

E. Agilent MassHunter software

2. LC Equipment

A. AB Sciex 5500 QTrap or 6500 QTrap LC- MS/MS detector

B. Shimadzu Prominence XR HPLC System

1) Pump: LC-20AD XR

2) Autosampler: SIL-20AC XR

3) Column Oven: CTO-20AC

4) Controller: CBM-20A

5) Degasser: DGU-20A3 6.2.2.6

5. Quantitation

1. GC-MS/MS

Quantitation is determined by using the ratio of response of the analyte to an internal standard compared to the ratio of the response of the analyte and internal standard from an external single level standard. Alternate methods may be used when needed. Manual calculation may be required in some cases, for example, to determine a multi-peak residue or use of a specific standard not used in the instrument calibration.

The relative response factor (RRF) of the target analyte to an internal standard is used to determine the concentration in a sample:

$$RRF = \left(\frac{R(Std)}{R(IS)}\right) * \left(\frac{Conc(IS)}{Conc(Std)}\right)$$

The concentration of the analyte in a sample is calculated using the sample responses with the corresponding RRF:

$$Conc(Spl)\left(\frac{ug}{g}\right) = \left(\frac{R(Spl)}{R(IS)}\right) * \left(\frac{Conc(IS)}{Conc(Spl)}\right) * \left(\frac{1}{RRF}\right)$$

Where:

RRF = Relative response factor for the analyte in the standard

R(Std)= Area response of the analyte in the standard

R(IS)= Area response of the internal standard

R(Spl)= Area response of the analyte in the sample

Conc(IS)= IS concentration (µg/mL) in the injection vial

Conc(Std)= Standard analyte concentration (µg/mL) in the injection vial

Conc(Spl)= Concentration of sample extract in g/mL

2. LC-MS/MS

Quantitation is done using the response of the analyte compared to the response from the average responses from the initial calibration and continuing calibration external standards.

$$Conc.(ppb) = \frac{ConcStd\ \left(\frac{ng}{ml}\right) X\ SplVolume\ (mL) X\ SplArea}{SplWt(g) XStdArea} X\ Dilution\ Factor$$

3. RESULTS AND DISCUSSION

3.1 Accuracy and Precision

Table 2 summarizes the percent of compounds meeting accuracy and precision criteria by each platform and combined. If a compound failed to meet criteria on one platform but was also included on the other platform, then the combined results include the results from the platform which met criteria.

Table 2. Percent of Compounds Meeting Accuracy and Precision Criteria

Platform	Number of target compounds	Number meeting recovery criterion	Number meeting precision (RSD) criterion	Overall (number meeting both criteria)
GC-MS/MS	194	79%	93%	77%
LC-MS/MS	229	90%	88%	83%
Combined	325	82%	91%	77%

RSD is relative standard deviation

The number of target compounds in the combined platform is less than the totals from GC-MS/MS plus LC-MS/MS because some compounds (analytes) were quantified by both methods.

An accuracy objective of 60 to 130% was specified in the study proposal. Accuracy was evaluated versus this objective by using spike recoveries at three levels on three different brands of grasshoppers corresponding to the normal action level and minimum reporting level of 10 ng/g; half this level at 5 ng/g; and twice this level at 20 ng/g.

Based on previous validations, the recovery range for most compounds should be within an acceptance range of 60 – 130%. The mid-level spiking concentration of 10 ng/g was selected since this represents the currently accepted regulatory action level for residues that don't have an established tolerance. Additionally, there are Food Handling Establishment tolerances for some residues above 10 ng/g. Many compounds are covered by both GC and LC-based platforms and results can be compared from both systems to select the better result.

A precision objective of no greater than 30% relative percent difference (RPD) was specified in the study proposal for precision. Precision was evaluated versus this objective via the use of duplicate spikes.

In the following sections, the QC results are compared against the criteria for each platform.

3.2 QC Results by Platform

3.2.1 GC-MS/MS accuracy results

The results for each compound are shown in Table 3. The average recovery for all analytes on the GC-MS/MS was 89%. There were some compounds showing low level interferences that were either matrix or instrument related. Triazophos was present above the action level of 10 ng/g in one of the three sources. Recoveries were determined by taking the contribution of the interferences and incurred residues into account. Forty (40) of the 194 compounds were outside the acceptance range of 60 -130%. Twenty-three (23) of the 40 compounds are also analyzed by LC/MS/MS.

In this study and in previous validations for QuEChERS, captan, chlorothalonil, dichlofluanid, and tolylfluanid had no recovery. Amitraz and tolylfluanid are base-sensitive compounds. The use of an acidified acetonitrile solvent is recommended for such compounds to help stability in solution and improve recoveries per LIB 4594, "A QuEChERS Procedure Focused on Pesticides and Chemical Contaminants that Require Determination by Gas Chromatography," by Greg Mercer, *et al* (15). However, the extraction solvent used in this study was not acidified because the initial studies, validations, and analyses in the Kansas Laboratory did not use acidified solvent. We do not plan to explore the effect of changing the solvent due to the possible impact on recoveries of other compounds.

Ethoxyquin was not recovered at the levels used in this study. Previous studies have shown poor recoveries below 50 ppb.

Carboxin was not recovered in several spiked samples. Also, the 50 ng/ml standard used as the initial calibration standard had very low responses, so the 500 ng/ml standard was used to calculate the amount of carboxin in the samples.

Planar compounds have had low recoveries in previous studies, and similar results were also seen for this matrix. These compounds include hexachlorobenzene, pentachloroaniline, pentachloroanisole, pentachlorobenzene, pentachlorobenzonitrile, and pentachlorothioanisole. The clean-up step for the GC based analysis uses solid phase extraction (SPE) cartridges containing graphitized carbon black or similar material that is known to reduce recovery of these planar compounds. Recoveries may be improved by use of toluene or ethyl acetate as a solvent for the final extract instead of acetone. However, no changes were made in this study.

The dried grasshopper extracts had significant fatty acid esters present based on a full scan analysis so an additional cleanup step was used to remove the fat. Even with this step enough interfering matrix

remained to shift the retention times for several compounds outside the MS/MS instrument method acquisition window, especially for two of the three sources. The GC-MS/MS instrument parameters were not changed to account for this shift. The peak shapes for some other compounds were also affected by the presence of the matrix.

Some compounds could not be determined in source 1 and/or source 3 but had acceptable recoveries in source 2. Chapulines were used as source 2, which also included more flavor additives (spices). Both source 1 and 3 were other types of grasshoppers. These compounds include bupirimate, flusilazole, methoprene, myclobutanil, and triazophos. Both sources 1 and 3 had higher fatty acid ester backgrounds than source 2, so the difference in results is possibly due to matrix differences between the grasshopper and chapulines.

Triazophos was found in source 2. It is also a target analyte in the LC-MS/MS analysis and was found with that method also, as mentioned in the next section.

3.2.2 LC-MS/MS accuracy results

The average recovery for all analytes on the LC-MS/MS was 84%. There were some compounds showing low level interferences, either matrix or instrument related. Triazophos and rotenone were present above the action level of 10 ng/g in one of the three sources. There were eight other compounds present in the same source at levels below 5.0 ng/g used for the study: azoxystrobin, buprofezin, chlorantraniliprole, difenoconazole, ethion, imazalil, tebuconazole, and thiabendazole. The recoveries were calculated taking the contribution of the interferences and incurred residues into account.

Twenty-one (21) of the 209 target pesticide residues evaluated for this study were outside the acceptance range of 60 – 130%.

Carboxin, chlorfluazuron, chlorimuron-ethyl, clethodim, chlothiadin, cycloxydime, ethirimol, fenhexamid, flonicamid, fomesafen, imazasulfuron, lufenuron, metaflumizone, moxidectin, propamocarb, pymetrozine, teflubenzuron, thiamethoxam, tolfenpyrad, and triflusulfuron methyl ester had recoveries below 60%. Metoxuron had a recovery above 130%.

It was observed for many compounds that the recoveries for sources 1 and 3 tended to be lower than the recoveries for source 2. Sources 1 and 3 had a higher fatty acid ester content than source 2 which caused

matrix effects in the chromatography. A higher than typical fatty acid ester content was observed in a GC-MS full scan analysis of the matrix.

Table 3. QC Results. Red numbers indicate compounds (analytes) that did not meet the QC criteria. The notations in parentheses are FDA codes.

	GC			LC			
Compound	**Recovery**	**RSD**	**Uncertainty**	**Recovery**	**RSD**	**Uncertainty**	**Notes**
2,3,5,6-Tetrachloroaniline (508)	57	6	7				
2,6-Dichlorobenzamide(272)				63	18	35	Meets criteria
2,6-DIPN (B89)	42	99	128				
3-Hydroxycarbofuran (512)				81	13	25	Meets criteria
Acephate (204)				63	12	23	Meets criteria
Acetamiprid (B80)				80	17	35	Meets criteria
Acetochlor (807)				99	11	23	Meets criteria
Acibenzolar-S-methyl (B51)				107	21	43	Meets criteria
Alachlor (277)				99	10	20	Meets criteria
Aldicarb (167)				82	35	69	
Aldicarb Sulfoxide (169)				99	13	25	Meets criteria
Aldoxycarb (168)				88	12	24	Meets criteria
Ametoctradin (D27)	25	61	23	82	12	23	Use LC
Ametryn (156)	80	19	30	101	11	23	Meets criteria
Amicarbazone (D05)				96	14	28	Meets criteria
Amitraz (233)	10		12	95	78	155	Good recovery by LC
Atrazine (305)	90	9	16	85	12	23	Meets criteria
Avermectin B1a (948)							
Azinphos-methyl (042)							
Azoxystrobin (B48)	90	18	19	109	7	14	Meets criteria
Benalaxyl (B45)	87	7	12	101	7	14	Meets criteria
Bendiocarb (658)				91	12	24	Meets criteria
Benfluralin (191)	90	11	16				Meets criteria
BHC-alpha (903)	81	11	15				Meets criteria
BHC-beta (904)	94	14	21				Meets criteria
BHC-delta (905)	95	3	14				Meets criteria
BHC-gamma (050)	83	9	15				Meets criteria
Bifenazate (B82)	132	35	60	64	37	73	RSD high on both platforms. Recovery by LC within limits.
Bifenthrin (930)	79	4	7				Meets criteria
Bitertanol (850)	51	21	22	71	29	59	Use
Boscalid (B75)	80	6	13	79	20	39	Meets criteria
Bromopropylate (523)	56	19	40				
Bromuconazole (B20)				83	17	34	Meets criteria
Bupirimate (872)	89	21	40	99	9	18	Meets criteria
Buprofezin (B52)	91	14	32	82	18	36	Meets criteria
Butachlor (806)							
Butocarboxim (857)				86	40	79	
Butralin (960)	116	14	39				Meets criteria
Cadusafos (953)	75	9	16				Meets criteria
Captan (011)							
Carbaryl (102)				92	12	24	Meets criteria
Carbendazim (666)				75	14	27	Meets criteria
Carbetamide (875)				86	16	32	Meets criteria
Carbofuran (180)				99	11	22	Meets criteria
Carboxin (210)	46		17	34	87	174	Not good on either platform
Chlorantraniliprole (C86)				77	11	22	Meets criteria
Chlordane-cis (173)	69	22	28				Meets criteria

Chlordane-trans (172)	75	10	19				Meets criteria
Chlorfenapyr (B13)	103	10	22				Meets criteria
Chlorfenvinphos (B74)				97	11	21	Meets criteria
Chlorfluazuron (C72)				51	17	34	
Chlorimuron ethyl				42	66	131	

	GC			LC			
Compound	Recovery	RSD	Uncertainty	Recovery	RSD	Uncertainty	Notes
Chlorofenvinphos (617)	100	12	21				Meets criteria
Chlorothalonil (164)							
Chlorotoluron (862)				93	8	16	Meets criteria
Chlorpropham (114)	82	9	17				Meets criteria
Chlorpyrifos (160)	88	11	25	92	13	25	Meets criteria
Chlorpyrifos methyl (235)	95	9	23	108	37	73	Use GC
Clethodim (B93)				40	56	111	
Clofentezine (699)				92	14	27	Meets criteria
Clothianidin (B95)				54	21	42	
Coumaphos (124)	90	14	26	88	12	23	Meets criteria
Cumyluron (D14)				95	9	18	Meets criteria
Cyantraniliprole (D46)				68	20	39	Meets criteria
Cyazofamid (C48)				98	10	21	Meets criteria
Cycloate (232)	66	17	22				Meets criteria
Cycloxydime (D28)				27	88	176	
Cyfluthrin (781)	97	7	13				Meets criteria
Cyhalothrin-lambda (A39)	85	10	18				Meets criteria
Cymoxanil (877)				71	23	46	Meets criteria
Cypermethrin (597)	99	3	19				Meets criteria
Cyproconazole (A22)	22	72	27	87	14	27	Use LC
Cyprodinil (B22)	71	18	24	98	12	24	Use LC
Cyromazine (255)							
Dacthal (134)	77	7	20				Meets criteria
Daimuron (D06)				105	7	14	Meets criteria
DDE-o,p' (911)	66	7	19				Meets criteria
DDE-p,p' (910)	65	9	24				Meets criteria
DDT-o,p' (907)	80	3	22				Meets criteria
DDT-p,p' (906)	94	8	28				Meets criteria
DEF (217)	78	11	25				Meets criteria
Deltamethrin Total (612)	96	11	21				Meets criteria
Desmedipham (786)				77	11	23	Meets criteria
Diafenthiuron (D10)							
Diazinon (024)	86	10	17	98	7	15	Meets criteria
Dichlobenil (324)	56	12	12				
Dichlofluanid (588)							
Dichlorvos (338)	54	22	27	96	20	41	Use LC
Dicloran (144)	91	9	13				Meets criteria
Dicofol-p,p' IPfrag (254)	97	11	24				Meets criteria
Dicrotophos (209)				97	11	22	Meets criteria
Dieldrin (028)	101	19	78				Meets criteria
Diethofencarb (B62)	109	10	21	89	18	36	Meets criteria
Difenoconazole (B58)	53	29	20	85	18	35	Use LC
Diflubenzuron (651)				78	18	36	Meets criteria
Dimethachlor (A28)	93	32	59				
Dimethoate (171)				73	15	30	Meets criteria
Dimethomorph (B77)				92	15	30	Meets criteria
Dimethomorph Total (B77)	70	12	13				Meets criteria
Diniconazole (D01)	36	34	27	65	26	52	Use LC
Dinitramine (775)	107	8	24				Meets criteria
Dinotefuran (C75)				68	17	34	Meets criteria
Dioxacarb (656)				88	13	27	Meets criteria
Diphenylamine (125)	224	22	93				
Dithianon (XAL)				112	15	31	Meets criteria

	GC			LC			
Compound	**Recovery**	**RSD**	**Uncertainty**	**Recovery**	**RSD**	**Uncertainty**	**Notes**
Diuron (032)				71	13	25	Meets criteria
DMST (D30)				115	12	24	Meets criteria
Dodine (104)							
Doramectin (C62)				104	27	53	Meets criteria
Emamectin-Benzoate (C58)							
Endosulfan Sulfate (902)	91	10	26				Meets criteria
Endosulfan-1 (900)	71	12	19				Meets criteria
Endosulfan-II (901)	82	15	21				Meets criteria
Endrin (034)	93	12	26				Meets criteria
EPN (035)	98	7	16	79	15	31	Use GC
Epoxiconazole (B53)	70	8	11	90	16	32	Use LC
Eprinomectin (C59)				74	30	59	Meets criteria
Ethiofencarb sulfoxide (D31)				94	11	22	Meets criteria
Ethiolate (830)	48	12	14				
Ethion (107)	120	8	23	84	17	35	Use LC
Ethiprole (C64)				83	29	58	Meets criteria
Ethirimol (802)				33	70	139	
Ethofumesate (945)	97	18	29	98	21	42	Meets criteria
Ethoprop (175)	82	8	16	100	9	17	Use LC
Ethoxyquin (111)							
Etofenprox (B69)	78	4	37				Meets criteria
Etoxazole (B84)	73	10	19	93	8	17	Use LC
Etridiazole (722)	63	8	10				Meets criteria
Famoxadone (C01)	238	10	39	83	22	43	Use LC
Fenamidone (B64)	420	8	101	86	18	36	Use LC
Fenamiphos (236)	155	83	150				
Fenarimol (271)	61	19	25	76	24	47	Use LC
Fenazaquin (B73)				81	8	17	Meets criteria
Fenbuconazole (A30)	82	15	30	77	19	39	Chromatography on GC poor. Use LC
Fenbutatin oxide (639)							
Fenhexamid (B41)	414	45	497	53	27	55	Enhanced response on GC
Fenitrothion (391)	131	15	35				
Fenobucarb (C91)				97	13	26	Meets criteria
Fenoxycarb (811)				92	14	28	Meets criteria
Fenpropathrin (808)	90	14	27				Meets criteria
Fenpropimorph (886)				95	7	13	Meets criteria
FenpyroximateE (C73)				72	12	25	Meets criteria
Fenthion (177)	207	23	69				
Fenuron (840)				79	15	31	Meets criteria
Fenvalerate Total (546)	94	6	18				Meets criteria
Fipronil (A82)	99	17	23				Meets criteria
Flonicamid (C92)	115	11	20	55	22	44	Use GC
Flubendiamide (C87)				106	26	51	Meets criteria
Flucythrinate (229)	95	3	10				Meets criteria
Fludioxonil (B23)	95	11	17	74	28	57	GC preferred but chromatography often has significant tailing
Fluensulfone	95	11	40				Meets criteria
Flufenoxuron (C71)				61	26	52	Meets criteria
Fluopicolide (C74)	74	7	39				Meets criteria
Fluopyram (D32)	102	14	23	93	18	35	Meets criteria
Fluoxastrobin (C66)				105	7	13	Meets criteria

	GC			LC			
Compound	Recovery	RSD	Uncertainty	Recovery	RSD	Uncertainty	Notes
Fluquinconazole (B78)	74	8	14	88	16	32	Use LC
Fluridone (736)	74	14	25	99	9	19	Use LC
Flusilazole (950)	68	26	20	92	15	30	Use LC
Flutolanil (B63)	103	5	21	97	11	22	Meets criteria
Flutriafol (C53)	50	42	38	68	30	60	Use LC
Fluvalinate (297)	117	7	18				
Fluxapyroxad (D45)	83	8	12	94	17	33	Meets criteria
Folpet (126)	39		32				
Fomesafen (D19)				31	37	74	
Forchlorfenuron (B32)				68	20	41	Meets criteria
Formetanate (E19)				102	35	71	
Fuberidazole (887)							
Furalaxyl (D22)				90	13	27	Meets criteria
Halofenozide (C93)							
Heptachlor Epoxide (143)	77	17	28				Meets criteria
Hexachlorobenzene (321)	23	13	8				
Hexaconazole (954)	43			71	33	65	
Hexythiazox (B10)	96	45	85	69	18	35	Use LC. Chromatography and reproducibility on GC is poor.
Imazalil (604)				74	28	56	Meets criteria
Imazasulfuron (T07)				28	80	159	
Imidacloprid (967)				72	30	61	
Indoxacarb (C12)	80	10	14	84	18	37	Meets criteria
Iprobenfos (867)	96	8	21				Meets criteria
Iprodione (626)	93	11	30				Meets criteria
Iprovalicarb (C46)				127	27	54	Meets criteria
Isocarbamid (A36)	63	29	28	60	26	53	Meets criteria
Isocarbophos (C77)	156	15	35	110	25	50	Use LC
Isoprocarb (637)	103	17	45	101	9	18	Meets criteria
Isoprothiolane (855)	86	10	23	102	10	19	Meets criteria
Ivermectin (C61)							
Karbutilate (D33)				74	14	27	Meets criteria
Kresoxim methyl (B42)	93	11	21				Meets criteria
Kresoxim-methyl (B42)				94	9	19	Meets criteria
Linuron (129)				96	13	27	Meets criteria
Lufenuron (C34)				57	30	60	
Malathion (052)	122	5	15	92	13	27	Use LC
Mandipropamide (C94)				102	10	20	Meets criteria
Mepanipyrim (C55)	97	25	55	97	10	19	Meets criteria
Mepronil (D23)				100	9	19	Meets criteria
Metaflumizone (C40)				59	23	46	
Metalaxyl (607)	88	18	28	100	9	19	Meets criteria
Metaldehyde (B07)	56	9	12				
Metconazole (C54)				70	28	56	Meets criteria
Methamidophos (170)				60	23	46	
Methfuroxam (D20)							
Methidathion (197)	124	10	34	97	19	39	Use LC
Methiocarb (195)				95	10	20	Meets criteria
Methomyl (159)				80	8	17	Meets criteria
Methoprene (C14)	94	16	49				Meets criteria
Methoxychlor-o,p' (274)	106	9	18				Meets criteria
Methoxychlor-p,p' (275)	93	13	16				Meets criteria
Methoxyfenozide (C15)				107	15	30	Meets criteria

	GC			LC			
Compound	**Recovery**	**RSD**	**Uncertainty**	**Recovery**	**RSD**	**Uncertainty**	**Notes**
Methyl Parathion (057)	121	10	25				Meets criteria
Metolachlor (283)	92	6	18				Meets criteria
Metoxuron (844)				131	59	117	
Metrafenone (D13)	86	24	29	96	8	16	Meets criteria
Metribuzin (181)	60	17	40	85	23	46	Use LC
Metsulfuron-methyl (C18)							
Mevinphos (069)	80	15	27				Meets criteria
Mevinphos (578)				93	12	24	Meets criteria
Mexacarbate (681)				94	9	17	Meets criteria
MGK-264 (058)	80	11	49				Meets criteria
Monocrotophos (343)				91	8	15	Meets criteria
Moxidectin (C63)				50	12	24	
Myclobutanil (679)	75	17	18	73	25	51	Meets criteria
Napropamide (594)	82	19	23				Meets criteria
Nitenpyram (C51)				60	26	52	Meets criteria
Nitrofen (158)	102	12	19				Meets criteria
Novaluron (C35)				63	24	48	Meets criteria
Nuarimol (800)	64	17	16	63	37	74	
Omethoate (178)				85	8	16	Meets criteria
Oxadixyl (A46)	90	7	14	84	11	23	Meets criteria
Oxamyl (537)				103	15	29	Meets criteria
Oxyfluorfen (713)	127	13	40				Meets criteria
Paclobutrazol (A48)				85	20	40	Meets criteria
Parathion (065)	125	15	26	88	17	34	Use LC
Penconazole (956)	58	37	40	84	17	33	Use LC
Pencycuron (C95)				86	9	17	Meets criteria
Pendimethalin (230)	96	22	30				Meets criteria
Pentachloroaniline (351)	44	25	20				
Pentachloroanisole (376)	52	9	13				
Pentachlorobenzene (387)	40	13	11				
Pentachlorobenzonitrile (396)	42	7	12				
Pentachlorothioanisole(388)	24	17	11				
Penthiopyrad (D34)	99	7	15				Meets criteria
Permethrin-cis (222)	85	9	19				Meets criteria
Permethrin-trans (223)	78	9	27				Meets criteria
Phenmedipham (791)				72	15	31	Meets criteria
Phenylphenol-o (083)	93	29	48				Meets criteria
Phorate Sulfone (189)	117	12	26	100	14	27	Use LC
Phosalone (166)	105	15	34				Meets criteria
Phosmet (165)	154	42	130				
Phoxim (247)				101	9	17	Meets criteria
Picoxystrobin (C68)				106	20	39	Meets criteria
Piperonyl Butoxide (070)	83	10	16	102	15	30	Meets criteria
Pirimacarb (580)	76	28	27				Meets criteria
Pirimicarb (580)				92	12	25	Meets criteria
Pirimiphos Ethyl (587)	89	8	21				Meets criteria
Pirimiphos-methyl (562)	89	13	21	108	11	22	Meets criteria
Prochloraz (833)	34	35	19	89	12	25	Chromatography on GC poor. Use LC
Procymidone (593)	89	11	22				Meets criteria
Profenofos (224)	70	16	27	74	25	50	Meets criteria
Prometryn (249)	80	12	16	100	7	14	Use LC
Pronamide (540)	98	9	17				Meets criteria

	GC			LC			
Compound	Recovery	RSD	Uncertainty	Recovery	RSD	Uncertainty	Notes
Propamocarb (C42)				49	25	51	
Propanil (341)	89	8	18				Meets criteria
Propargite (623)	120	14	142	94	13	26	Use LC
Propiconazole (264)	63	18	30	81	20	39	Use LC
Propoxur (162)				97	11	23	Meets criteria
Proquinazid (D35)				73	6	11	Meets criteria
Prothiophos-Tok (613)	80	11	24				Meets criteria
Pymetrozine (C22)				31	23	47	
Pyraclostrobin (B61)	92	19	36	102	8	16	Chromatography on GC poor. Use LC
Pyridaben (B56)	82	6	15	80	6	13	Meets criteria
Pyrimethanil (B16)	70	21	25	109	11	22	Meets criteria
Pyriproxyfen (B24)	83	5	11	82	17	34	Meets criteria
Quinalphos (661)	102	15	38				Meets criteria
Quinoxyfen (B57)	62	7	13	76	11	23	Meets criteria
Quintozene (304)	66	15	18				Meets criteria
Resmethrin (556)	74		39				Meets criteria
Rotenone (020)				102	43	86	
Siduron (C24)				91	10	20	Meets criteria
Simazine (149)	100	14	35				Meets criteria
Spinetoram (C88)				95	14	28	Meets criteria
Spinosad (C25)				92	8	16	Meets criteria
Spinosyn D (D03)				90	11	22	Meets criteria
Spirodiclofen (B85)	76	32	40	64	34	67	Low response on GC. Use LC.
Spiromesifen (C60)	59	24	34	108	41	83	Use LC
Spirotetramat (C96)				73	47	94	
Spiroxamine (C98)				88	7	15	Meets criteria
Sulfotep (311)							
Sulfoxaflor+NH4 (D48)				73	22	45	Meets criteria
Tebuconazole (A58)	46	40	33	79	17	34	Chromatography on GC poor. Use LC
Tebufenozide (C56)				101	32	64	
Tebufenpyrad (C39)	73	14	25	60	29	58	Meets criteria
Tebuthiuron (780)				93	13	25	Meets criteria
Tecnazene (147)	68	10	15				Meets criteria
Teflubenzuron (C99)				54	33	67	
Temephos (749)				84	16	32	Meets criteria
Tepraloxydim (D36)				65	34	68	
Terbuthylazine (678)	82	8	18				Meets criteria
Tetraconazole (B72)	89	14	20	86	19	38	Meets criteria
Tetradifon (108)	81	7	18				Meets criteria
Tetramethrin Total (947)	104	32	67				
Thiabendazole (157)				72	19	38	Meets criteria
Thiacloprid (B68)				79	20	39	Meets criteria
Thiamethoxam (B43)	101	22	31	49	17	34	Use GC
Thifluzamide (F27)	100	11	21	90	14	28	Meets criteria
Thiobencarb (726)	80	11	16				Meets criteria
Thiodiarb (943)				98	7	14	Meets criteria
Thiophanate-methyl (611)							
THPI (624)	117	29	67				Meets criteria
Tolclofos-methyl (B70)	89	6	20				Meets criteria
Tolfenpyrad (D52)	82	16	24	57	31	61	Use GC
Tolylfluanid (649)							

	GC			LC			
Compound	**Recovery**	**RSD**	**Uncertainty**	**Recovery**	**RSD**	**Uncertainty**	**Notes**
Triadimefon (608)	91	10	21	82	17	34	Meets criteria
Triadimenol (638)	60	46	54	79	19	39	Use LC
Triallate (621)	66	6	13				Meets criteria
Triazophos (536)	124	6	20	97	25	50	Use LC
Trichlorfon (130)							
Tricyclazole (804)	21	76	19	81	15	29	
Tridemorph (795)				77	10	20	Chromatography on GC poor. Use LC
Trifloxystrobin (B79)	110	24	55	102	10	20	Meets criteria
Triflumizole (A61)	50	58	35	70	31	62	
Trifluralin (151)	90	6	11				Use LC
Triflusulfuron methyl ester (A62)				41	61	123	
Triticonazole (C33)				86	15	30	Meets criteria
Vinclozolin (529)	82	11	15				Meets criteria
Zoxamide (B44)				85	18	36	Meets criteria

3.3 Precision Results by Platform

3.3.1 GC-MS/MS precision results

An upper limit of 30% relative percent difference (RPD) was specified in the study proposal, which applies to either platform. The average RPD for all analytes on the GC-MS/MS was 16%.

In all, there were 19 of 194 compounds exceeding the criteria. The RPD was not determined for the compounds that did not show a recovery or that did not have a matching spike sample.

3.3.2 LC-MS/MS precision results

The average RPD for all analytes on the LC-MS/MS was 17%. In all, there were 22 of the 209 pesticide residues evaluated for this study that exceeded the criteria. These compounds include aldicarb, amitraz, bifenazate, butocarboxim, chlorimuron-ethyl, chlorpyrifos-methyl, clethodim, cycloxydime, ethiprole, ethirimol, flubendiamide, fludioxinil, formetanate, imazalil, imazasulfuron, nitenpyram, spiromesifen, spirotetramat, tebufenozide, tepraloxydim, triflumizole, and triflusulfuron methyl ester.

3.4 Uncertainty Results by Platform

3.4.1 GC-MS/MS uncertainty results

Uncertainty was calculated from the relative standard deviation (RSD) results for each platform. The SOP, KAN-LAB-QMS.126, "Estimation of Uncertainty Measurement," requires the determination of uncertainty at the 95% confidence level using the relative standard deviation (RSD) from at least ten (10) spiked laboratory control samples at different spike levels, not the RPD. All 18 spikes in this study were used for the determination. The coverage factor is two (2), or twice the RSD.

Very high values were obtained for 2,6-DIPN, bifenazate, dieldrin, dimethachlor, diphenylamine, fenamidone, fenamiphos, fenhexamid, fenthion, hexythiazox, mepanipyrim, phosmet, propargite, tetramethrin, THPI, triadimenol, and trifloxystrobin. High RSD values for 2,6-DIPN, diphenylamine, tetramethrin, and triadimenol are at least partly attributable to matrix interferences as a background peak is present in most samples.

3.4.2 LC-MS/MS uncertainty results

Using the same methodology, uncertainty was calculated from the RSD results for the LC/MS/MS platform.

Very high values were obtained for amitraz, carboxin, chlorimuron-ethyl, clethodim, cycloxydime, ethirimol, imazasulfuron, metoxuron, rotenone, spiromesifen, spirotetramat and triflusulfuron methyl ester.

3.5 Selectivity Results by Platform

3.5.1 GC-MS/MS selectivity results

Identification was done using ion ratios and retention time criteria per KAN-LAB-PES.075. Some ions were not used due to matrix interferences. However, at least one confirmation ion was still available for confirmation of identity. Also, several compounds had retention time shifts due to matrix interferences. Additional information such as peak shapes, ion ratios and the absence of these peaks in the unspiked matrix was used to confirm the identification for these compounds.

3.5.2 LC-MS/MS selectivity results

Identification was done using ion ratios and retention time criteria per KAN-LAB-PES.072 from all 18 spikes. Ion ratio criteria were high (>140%) for aminocarbazone, butocarboxim, cyantraniliprole eprinomectin, fenarimol, nuarimol, and parathion, and propamocarb. This was mostly due to low sensitivity at the lower spike levels.

3.6 LOD/LOQ

A default value of 10 ng/g for the LOQ will be used for those analytes meeting accuracy and precision criteria. This reporting level has generally been used as the action level for residues with no tolerances. Actual LODs and LOQs may be lower than this value.

3.7 Ruggedness/Robustness

As per the study proposal, the single matrix method extension was conducted over four days as stated in Appendix 3, part C of the referenced FDA FVM program guidelines. Three analysts performed the initial extractions over a four-day period.

For the GC-MS/MS analysis, an adjustment in the cleanup step was made, as allowed by the method. An additional tube of the Z-sep material was used to provide a better cleanup. The contents of two tubes were combined.

System suitability requirements for GC-MS/MS were met on each day of analysis.

No adjustments were made for the cleanup used in the LC-MS/MS cleanup. System suitability requirements for LC-MS/MS were met on each day of analysis.

4. ACKNOWLEDGEMENT

Stacy Hetz, David Graham and Daniel Sagardia-Vazquez performed the initial extractions and GC-MS/MS cleanup. David Graham set up and analyzed the samples for the GC-MS/MS platform with review by David Eide. Mark Ross performed the LC-MS/MS cleanup and analysis. Robert E. Smith used the SOP that was written by David Eide when he wrote the LIB.

REFERENCES

1. Gunderson EL. FDA Total Diet Study, July 1986-April 1991, dietary intakes of pesticides, selected elements, and other chemicals. *J. AOAC Int*. **78**: 1353-1363, 1995.

2. Lehotay SJ, Mastovska K, Lightfield AR. Use of buffering and other means to improve results of problematic pesticides in a fast and easy method for residue analysis of fruits and vegetables. *J. AOAC Int*. **88**: 615-629, 2005.

3. Pryzbylski C, Hommet F. Evaluation of some parameters affecting troublesome pesticide analysis in gas chromatography–ion-trap mass spectrometry. *J. Chromatogr. A* **1201:** 78-90, 2008.

4. Mastovska K, Lehotay SJ. Evaluation of common organic solvents for gas chromatographic analysis and stability of multiclass pesticide residues. *J. Chromatogr. A* **1040:** 259-272, 2004.

5. SUPELCO. Analysis of compounds in fatty matrices. 2014. https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Supelco/General_Information/1/12023_T414114_Compounds_in_Fatty_Matrices.pdf

6. Mastovska K, Lehotay SJ, Anastassiades M. Combination of analyte protectants to overcome matrix effects in routine GC analysis of pesticide residues in food matrixes. *Anal. Chem*. **77:** 8129-8137, 2005.

7. Koesukwiwat U, Lehotay SJ, Leepipatpiboon N. Fast, low-pressure gas chromatography triple quadrupole tandem mass spectrometry for analysis of 150 pesticide residues in fruits and vegetables. *J. Chromatogr. A* **1218:** 7039-7050, 2011.

8. KAN-LAB-PES.053 - Analysis of Pesticides and Industrial Chemical Analysis by the QuEChERS Procedure.

9. KAN-LAB-PES.072 - Determination of Pesticides and Industrial Chemicals by LC-MS/MS Using AB Sciex 4000 and 5000 QTraps.

10. KAN-LAB-PES.075, Determination of Pesticides and Industrial Chemicals using Agilent 7000 GC/MS Triple Quad.

11. Official Methods of Analysis of AOAC International, AOAC int., Gaithersburg, MD, Method 2007.1, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.

12. Sack C, Smoker M, Chamkasem N, Thompson R, Satterfield G, MacMahon S, Masse C, Mercer G, Neuhaus B, Cassias I, Chang E, Wong J, Zhang K. Laboratory Information Bulletin, No. 4465, Collaboration of the QuEChERS Procedure for the Multiresidue Determination of Pesticides by LC-MS/MS In Raw Agricultural Commodities.

13. Mercer G, Neuhaus B, Jones J, Wong M. Laboratory Information Bulletin, No. 4521, GC-MS/MS Determination of Over 200 Pesticides Commonly Found in Regulatory Samples.

14. KAN-LAB-WI.044, version 2.1, QuEChERS Procedure for LC and GC.

15. Mercer G, Jones J, Olson M, Mabry-Smith R. Laboratory Information Bulletin, No. 4594, A QuEChERS Procedure Focused on Pesticides and Chemical Contaminants that Require Determination by Gas Chromatography.

Attachment A: GC-MS/MS Instrument Parameters

These parameters are established for the use of the GC Triple Quad as a Pesticide Analyzer. Other parameters not listed are set by the software during the tuning parameters. The Pesticide Analyzer is configured for the analysis of the compounds listed in Attachment A. Alternate settings may be used for targeted compounds for confirmation.

Mass Spectrometer settings

1. Ion Source Temperature: 300° C.
2. MS1 quadrupole temperature: 150° C.
3. MS2 quadrupole temperature: 150° C.
4. Transfer Line temperature: 290° C.
5. Electron energy: -70 eV.
6. Emission current: 35 μA.
7. Collision cell: a. He Quench gas – 2.25 ml/min.
   b. N_2 gas – 1.5 ml/min.
8. Solvent Delay: 3 min.

Note: All other settings such as lens voltages and multiplier voltages are set through the instrument Autotune.

GC settings

1. Inlet Liner: 2mm dimpled glass, Agilent #5190-2296 or equivalent.
2. Inlet start temp: 60° C Hold: 0.2 min Rate: 600° C/min to 290° C.

   Compressed air is used to assist in cooling the inlet. The GC is configured as N_2.

3. Injection mode: Splitless Purge Flow: 30 ml/min at 3 min
4. Injection volume: 1 ul extract and 1 ul internal standard.
5. Total flow: 50-60 ml/min using ultrapure helium as carrier gas.

6. GC columns: The columns have a DB-5 phase with a 0.25 mm ID and 0.25 um film thickness and a length of 15 meters.

   Note: The GC is equipped with a backflush valve located between two columns of identical phase and length.

7. GC Oven Program

   A. Temperature Program:

      1) Initial Temperature: 60° C. Hold 1 minute.

      2) 40°C/min from 60° to 170° C

      3) 10° C/minute from 170° to 310° C. Hold time is 1.25 minutes. Total run time is 19 min.

      4) Post-run time: 2 minutes with reversed flow at approximately 3.4ml/minute.

   B. Flow rates: Column 1 – Approx. 1 ml/min. Column 2 Approx. 1.2 ml/min.

Note: The flow rate for column 1 is set so that the retention time for chlorpyrifos is approximately 9.96 minutes and all analyte peaks are in the MRM window. The flow rate for column 2 is 0.2 ml/min greater than the flow rate in column 1.

   C. Backflush: Post run flow rate: -3.4 ml/min for 2 minutes.

   D. Transfer Line temperature: 300° C.

   E. Collision cell: Helium quench gas is at 2.25 mls per minute to control metastable helium. N2 collision gas flow is 1.5 mls /min.

Attachment B: LC-MS/MS Instrument Parameters

Scheduled MRM	Yes
Polarity Ion	Positive
Probe Horizontal Position	2 mm
Probe Vertical Position	5 mm
Curtain Gas	40
CAD	Medium
Ion Source Gas 1	60
Ion Source Gas 2	60
Temperature	325 °C
Resolution Q1	Unit
Resolution Q2	Unit
Settling Time	0 ms
Pause between mass ranges	5 ms
Target scan time	0.5 sec

HPLC System Parameters:

Column: Agilent Zorbax Eclipse Plus C18 4.6 mm x 75 mm, 1.8 µm or equivalent

Column Temp: 40 °C

Column Flow 0.5 mL/min.

Injection Volume: 2 – 10 µL

Rinsing Volume: 200 µL

Needle Stroke: 52 mm

Rinsing Speed: 35 µL /sec

Sampling Speed: 5.0 µL /sec

Purge Time: 25 min

Rinse Dip Time: 3 sec

Rinse Mode: Before and after aspiration

Cooler Enabled: Yes

Cooler Temperature: 15 °C

HPLC Mobile Phase:

Mobile Phase A: 4 mM ammonium formate and 0.1% formic acid in water

Mobile Phase B: 4 mM ammonium formate and 0.1% formic acid in methanol

Time (minutes)	Parameter	Value
0	Pump B Conc.	5
3.5	Pump B Conc.	75
3.5	Pump B Curve	3
6.5	Pump B Conc.	95
9.0	Pump B Conc.	98
15.0	Pump B Conc.	98
16.0	Pump B Conc.	5
16.1	Stop	

Attachment C: GC-MS/MS Compound List and Transitions

MRM	Ret.			Quant MRM			Qual MRM	
Start	Time	Compound	Q1	Q3	CE	Q1	Q3	CE
3.4	3.736	Metaldehyde	89	45	2	87	43	2
4.0	4.373	*Propoxur IP Frag*	152	110	7	110	63	30
	4.469	*Clofentezine frag*	137	102	15	137	75	30
	4.470	Ethiolate	161	100	1	161	72	15
	4.473	D8 Naphthalene	136.1	84	26	136.1	82	36
	4.700	Dichlorvos	184.9	93	14	184.9	109	17
5.23	5.273	Dichlobenil	170.9	100	30	170.9	136	15
5.52	5.617	Mevinphos	192	127	10	127	95	16
	5.855	Etridiazole	210.9	182.9	9	210.9	139.9	26
5.93	5.990	THPI	151	79	27	151	122	4
	6.104	d10-Acenaphthene	164.1	160.1	38	164.1	134.1	45
	6.267	o-Phenylphenol	170	115	45	170	141	30
	6.310	Oxamyl	162	115	7	162	145	0
	6.338	Isoprocarb	136	103	26	136	77	38
	6.370	Pentachlorobenzene	249.9	214.9	21	249.9	141.9	50
6.55	6.722	Flonicamid	174	69	44	174	126	22
	6.762	Fluensulfone	119	59	24	119	92	10
	6.842	Propoxur	110	63	30	152	110	7
	6.906	Tecnazene	260.9	202.9	13	214.9	178.9	10
	6.972	Diphenylamine	169	66	32	169	51	55
	7.028	Ethoprop	158	97	16	200	158	1
	7.055	Cycloate	154	83	6	154	55	22
	7.078	2356-Tetrachloroaniline	230.9	159.9	24	230.9	168.9	25
	7.112	Chloropropham	213	171	5	213	127	16
7.19	7.250	Trifluralin	306	264	7	306	160	25
	7.282	Benfluralin	292	160	22	292	206	12
	7.381	Sulfotep	322	146	27	322	202	8
	7.433	Cadusafos	159	97	24	158	81	15
7.55	7.648	BHC-alpha	219	183	7	181	145	15
	7.783	2,6-DIPN	197	155	12	197	167	32
7.72	7.788	Hexachlorobenzene	283.8	213.9	40	283.8	248.9	22
	7.800	Ethoxyquin	202	174	18	202	145	35
	7.811	Dicloran	206	176	13	206	124	27
	7.815	Simazine	201	138	11	201	172	13

MRM	Ret.			Quant MRM			Qual MRM	
Start	**Time**	**Compound**	**Q1**	**Q3**	**CE**	**Q1**	**Q3**	**CE**
	7.835	Pentachloroanisole	265	237	15	280	265	12
	7.893	Atrazine	200	104	20	200	122	9
	8.089	Isocarbamid	142	70	22	142	42	34
	8.154	BHC-gamma	219	183	8	217	181	7
	8.180	Pronamide	173	74	50	173	109	30
	8.236	Quintozene	248.9	213.9	16	294.9	236.9	20
	8.268	Pentachlorobenzonitrile	274.9	239.9	20	274.9	204.9	36
	8.285	Diazinon	304	179	13	179	137	20
	8.290	Pyrimethanil	198	118	40	198	158	22
8.35	8.406	Dinitramine	261	195	23	261	241	10
8.47	8.513	BHC-delta	219	183	8	217	181	7
	8.568	Triallate	268	183.9	20	268	226	12
	8.585	Chlorothalonil	265.9	133	53	265.9	169.9	28
8.65	8.690	Iprobenfos (IBP)	204	121	38	204	91	6
	8.734	Pirimacarb	238	166	7	166	55	22
8.83	8.919	Pentachloroaniline	262.9	191.9	25	264.9	193.9	28
	8.933	Propanil	161	99	30	217	161	7
	8.992	Dimethachlor	134	105	15	197	148	10
	9.006	Metribuzin	198	82	18	198	89	15
9.06	9.116	Vinclozolin	212	172	16	187	124	22
	9.143	Methyl Parathion	263	109	12	263	79	32
	9.143	Chlorpyrifos-methyl	285.9	93	24	285.9	208	15
	9.233	Ametryn	227.1	58	12	227.1	185.1	4
	9.236	Tolclofos-methyl	265	93	26	265	109	52
	9.284	Prometryn	241.1	58	15	241.1	184.1	10
	9.336	Metalaxyl	234.1	146.1	21	249.1	190.1	4
9.47	9.592	Pirimiphos-methyl	290	125	24	290	233	10
	9.597	Fenitrothion	277	260	4	260	125	12
	9.608	Ethofumesate	286	207	5	207	161	5
	9.740	Malathion	173	99	14	173	127	2
	9.760	Pentachlorothioanisole	295.8	262.9	16	295.8	245.8	37
	9.763	Dichlofluanid	224	123.1	11	226	123	12
	9.773	Diethofencarb	225.1	96.1	38	225.1	168.1	10
	9.786	Thiobencarb	257	100	5	257	72	22
9.84	9.876	Phorate sulfone	199	143	9	199	97	23
	9.895	D10-Chlorpyrifos	324	260	16	260	167	25
	9.900	Metolachlor	238.1	162.1	13	162.1	133.1	15
	9.918	Fenthion	278	109	20	278	169	18

MRM	Ret.			Quant MRM			Qual MRM	
Start	Time	Compound	Q1	Q3	CE	Q1	Q3	CE
	9.959	Chlorpyrifos	313.9	257.9	14	313.9	165.9	43
	9.973	Parathion	291	109	10	291	81	35
	10.004	Triadimefon	208	181	10	208	111	26
	10.036	p,p'-Dicofol IP frag	250	139	16	250	111	42
	10.057	Tetraconazole	336	218	20	336	204	36
	10.060	Dacthal	298.9	164.9	54	300.9	222.9	30
	10.080	Isocarbophos	136	69	37	230	198	8
	10.212	Butralin	266	220.1	10	266	190	12
	10.289	Thiamethoxam	212	139	11	212	182	3
10.19	10.291	Pirimiphos-ethyl	318	166	13	318	182	13
10.35	10.400	Cyprodinil	224.1	208.1	27	224.1	118.1	45
	10.437	MGK-264	164	80	32	164	98	12
10.49	10.527	Pendimethalin	252	162	10	252	191	8
	10.554	Penconazole	248	157	30	248	192	15
10.59	10.627	Heptachlor Epoxide	352.8	262.8	15	352.8	281.9	18
	10.637	Fluopyram	173	145	18	396	223	10
	10.641	Tolylfluanid	238	137	15	137	65	34
	10.650	Fipronil	366.9	212.9	37	366.9	254.9	24
	10.668	Chlorfenvinphos	266.9	159	17	322.9	266.9	12
	10.733	Captan	149	70	22	264	79	25
	10.734	Quinalphos	157	102	28	146	118	10
	10.737	Triadimenol	168	70	8	128	65	24
	10.737	Methoprene	153	111	5	191	107	13
10.79	10.833	Prallethrin	168	123.1	6	168	81	18
	10.848	Procymidone	283	96	10	283	67	37
	10.850	Folpet	259.9	130	16	259.9	102	42
	10.851	Triflumizole	206	179	18	287	68	6
	10.970	Hexythiazox	184	149	4	156	75	53
	11.002	Methidathion	145	85	4	145	58	13
	11.040	Chlordane-trans	372.8	265.9	24	372.8	263.9	26
	11.086	o,p'-DDE	246	176	35	318	246	25
	11.177	Mepanipyrim	222.1	118	44	222.1	206.1	30
11.11	11.275	Endosulfan-I	240.9	205.9	15	195	159	8
	11.301	Chlordane-cis	372.8	265.9	24	372.8	263.9	26
	11.310	Chlorfluazuron frag	320.9	303.9	30	320.9	112.9	50
	11.312	Flutriafol	219	123	16	219	95	40
	11.320	Fenamiphos	303	195	6	303	154	15
	11.394	Flutolanil	173	75	50	173	95	36

MRM	Ret.			Quant MRM			Qual MRM	
Start	**Time**	**Compound**	**Q1**	**Q3**	**CE**	**Q1**	**Q3**	**CE**
	11.408	Napropamide	271.1	72	15	271.1	128	2
	11.446	Hexaconazole	214	159	23	231	175	9
	11.491	Imazalil	215	173	4	215	41	15
	11.494	Isoprothiolane	290	118	12	290	204	3
	11.498	Prothiophos/Tok	309	239	16	309	205	34
	11.543	Fludioxonil	248	127	33	248	154	20
	11.550	Profenofos	336.9	266.9	14	336.9	188	32
	11.586	DEF	202	147	2	202	113	18
	11.621	p,p'-DDE	246	176	35	318	246	25
	11.632	Tricyclazole	189	162	12	189	135	22
11.67	11.717	Oxyfluorfen	252	146	40	252	170	32
	11.727	Dieldrin	262.9	192.9	40	262.9	190.9	38
	11.735	Myclobutanil	179	125	17	179	90	35
	11.745	Carboxin	235	143	8	235	43	36
	11.776	Flusilazole	233	165	22	233	152	20
	11.786	Buprofezin	172	57	14	175	132	12
	11.807	Bupirimate	273.1	193.1	9	273.1	108	15
	11.818	Kresoxim methyl	206	116	3	206	131	12
	11.845	Thifluzamide	449	429	17	447	427	17
11.92	12.007	Nitrofen	283	162	26	283	202	14
	12.038	Cyproconazole	222	125	23	222	82	10
	12.045	Chlorfenapyr	246.9	226.9	18	246.9	200	32
	12.129	Endrin	262.9	192.9	40	262.9	190.9	38
12.2	12.282	Endosulfan-II	195	159	8	240.9	205.9	15
	12.318	Diniconazole	268	232	12	268	136	42
	12.375	Penthiopyrad	177	101	20	177	75	30
	12.436	Ethion	231	129	25	231	175	12
	12.441	o,p'-DDT	235	165	30	235	199	18
	12.442	Oxadixyl	163	132	10	163	117	30
12.55	12.654	Triazophos	161	134	6	257	162	4
12.74	12.874	Benalaxyl	266.1	148	8	234	146	20
	12.929	Quinoxyfen	237	208	32	307	237	24
	12.937	Propiconazole	259	69	10	259	173	20
	12.947	Trifloxystrobin	222	162	7	116	63	30
	13.030	Endosulfan Sulfate	271.9	236.9	15	271.9	116.9	48
	13.041	p,p'-DDT	235	165	30	235	199	18
	13.043	Fenhexamid	177	78	30	177	113	22
	13.102	Fluopicolide	346.9	171.9	30	209	182	19

MRM	Ret.			Quant MRM			Qual MRM	
Start	**Time**	**Compound**	**Q1**	**Q3**	**CE**	**Q1**	**Q3**	**CE**
13.15	13.215	o,p'-Methoxychlor	227	121	15	121	78	26
	13.271	Nuarimol	235	139	18	203	107	13
	13.274	Tebuconazole	250	125	26	125	89	20
	13.325	Propargite	135	107	14	135	77	32
	13.378	Piperonyl Butoxide	176	103	30	176	131	16
	13.412	Resmethrin	171	128	17	171	143	4
13.48	13.580	Epoxiconazole	192	138	12	192	111	27
	13.704	Iprodione	314	56	24	314	245	10
	13.721	Spiromesifen	272.1	254.1	3	272.1	209	14
13.76	13.810	Tetramethrin 1	164	107	14	164	135	10
	13.854	Acetamiprid	152	116	19	152	62	45
	13.911	Phosmet	160	77	27	160	133	14
	13.921	Fluxapyroxad	381.1	159.1	12	159.1	139.1	13
	13.918	Tetramethrin 2	164	107	14	164	135	10
	13.924	Bromopropylate	338.9	182.9	18	342.9	184.9	18
	13.925	Bifenthrin	181	165	35	181	166	12
	13.948	EPN	157	110	14	185	110.1	25
	13.965	Bifenazate	258	199	10	300	258	6
	13.966	D12 Chrysene	240.1	236.1	41	240.1	212.1	33
	14.031	Methoxychlor (p,p')	227	169	28	227	141	40
	14.051	Fenpropathrin	181	152	30	265	210	10
	14.086	Etoxazole	300.1	270.1	26	204.1	161.1	23
	14.107	Tebufenpyrad	333	171	22	318	145	15
	14.162	Fenamidone	268	180	22	238	103	22
14.22	14.424	Tetradifon	353.9	159	12	353.9	227	9
	14.592	Phosalone	182	75	36	182	111	17
	14.613	Azinphos-methyl	160	77	19	160	132	1
	14.626	Pyriproxifen	136	41.1	18	136	78.1	32
	14.795	Amitraz	293.1	162	6	293.1	132	25
	14.888	lambda-Cyhalothrin	197	141	13	181	152	29
14.97	15.097	Fenarimol	219	107	12	251	139	15
	15.302	Ametoctradin	176	121	18	176	65	32
	15.306	Metrafenone	393	363	18	377	283	18
	15.558	Bitertanol	170	115	45	170	141	30
	15.621	cis-Permethrin	183	153	18	183	115	30
	15.664	Spirodiclofen	312	109	19	312	259	11
	15.742	trans-Permethrin	183	153	18	183	115	30
	15.777	Pyridaben	147	117	24	147	132	13

MRM	Ret.			Quant MRM			Qual MRM	
Start	**Time**	**Compound**	**Q1**	**Q3**	**CE**	**Q1**	**Q3**	**CE**
	15.862	Fluquinconazole	340	108	48	340	298	20
	15.869	Coumaphos	362	109	15	362	81	42
	15.927	Prochloraz	310	70.1	15	308	70	15
16.05	16.230	Fenbuconazole	198	129	8	129	102	16
	16.268	Cyfluthrin	163	127	5	226	206	10
	16.585	Cypermethrin	181	152	30	163	127	4
	16.600	Boscalid	140	76	30	342	140	15
	16.666	Flucythrinate	157	107	15	199	157	5
	16.795	Etofenprox	163	107	20	163	135	10
16.94	17.143	Fluridone	328	189	54	328	259	33
	17.421	Fenvalerate 1	167	125	12	125	89	23
	17.466	Pyraclostrobin	132	77	20	164	132	10
	17.580	Fluvalinate	250	55	18	250	200	24
	17.614	Fenvalerate 2	167	125	12	125	89	23
17.68	17.833	Difenoconazole	323	265	17	265	202	20
	17.920	Deltamethrin 1	253	93	18	253	174	5
	18.046	Indoxacarb	203	106	24	203	78	36
	18.134	Deltamethrin	253	93	18	253	174	5
18.24	18.43	Azoxystrobin	344	172	46	344	156	46
	18.464	Famoxadone	330	196	24	330	224	10
	18.481	Dimethomorph 1	301	165	16	301	139	20
	18.660	Tolfenpyrad	383	171	25	383	197	25
	18.791	Dimethomorph 2	301	165	16	301	139	20

Notes:

Q1 is *m/z* of the parent ion (in Da).

Q3 is *m/z* of the parent ion (in Da).

Quant MRM is the Q3 ion that was used in the multiple reaction monitoring for quantitation.

Qual MRM is the Q3 ion that was used in the multiple reaction monitoring for qualitative analysis.

CE is the collision energy (in eV).

Attachment D: LC-MS/MS Acquisition Parameters

Compound	Q1	Q3	RT	DP	EP	CE	CXP
2,6-Dichlorobenzamide-[272] 1	190	173	3.81	71	10	27	16
2,6-Dichlorobenzamide-[272] 2	190	109	3.81	71	10	49	10
3-Hydroxycarbofuran-[512] 1	238.1	163	4.09	86	10	21	15
3-Hydroxycarbofuran-[512] 2	238.1	181	4.09	86	10	16	15
Acephate-[204] 1	184.1	143	3.02	81	10	13	15
Acephate-[204] 2	184.1	49	3.02	81	10	33	15
Acetamiprid-[B80] 1	223	126	4.08	81	10	29	15
Acetamiprid-[B80] 2	223	99	4.08	81	10	53	15
Acetochlor-[807] 1	270.1	224	7.42	60	10	15	12
Acetochlor-[807] 2	270.1	148	7.37	60	10	27	12
Acibenzolar-S-methyl-[B51] 1	211	136	6.61	66	10	39	15
Acibenzolar-S-methyl-[B51] 2	211	140	6.61	66	10	31	15
Alachlor-[277] 1	270.1	238	7.44	60	10	17	18
Alachlor-[277] 2	270.1	162	7.45	60	10	31	12
Aldicarb Sulfoxide-[169] 1	207.1	132.1	3.2	50	10	10	15
Aldicarb Sulfoxide-[169] 2	207.1	89.1	3.2	50	10	19	15
Aldicarb-[167] 1	208.11	116	4.71	56	10	11	15
Aldicarb-[167] 2	208.1	89	4.71	56	10	23	15
Aldoxycarb-[168] 1	223.1	86.1	3.32	72	10	21	15
Aldoxycarb-[168] 2	223.1	148	3.31	72	10	13	15
Ametoctradin [D27] 1	276.2	176.1	8.47	86	10	51	12
Ametoctradin [D27] 2	276.2	149.1	8.47	86	10	51	12
Ametryn-[156] 1	228.2	186.2	6.09	66	10	23	15
Ametryn-[156] 2	228.2	96.1	6.09	66	10	33	15
Amicarbazone-[D05] 1	242.2	143.1	5.02	51	10	15	24
Amicarbazone-[D05] 2	242.2	85.2	5.02	51	10	45	14
Amitraz [233] 1	294.2	163.2	9.62	46	10	21	4
Amitraz [233] 2	294.2	107.1	9.61	46	10	57	4
Atrazine [305] 1	216.1	174	5.8	66	10	23	10
Atrazine [305] 2	216.1	104	5.8	66	10	36	10
Avermectin B1a-[948] 1	890.9	305.4	9.75	91	10	35	15
Avermectin B1a-[948] 2	890.9	567.7	9.75	96	10	23	15
AvermectinB1b [948] 1	876.5	291	9.3	41	10	35	4
AvermectinB1b [948] 2	876.5	145	9.67	41	10	43	4
Azinphos-methyl-[042] 1	318	132	5.96	35	10	21	15
Azinphos-methyl-[042] 2	318.1	160	5.96	35	10	13	15
Azoxystrobin-[B48] 1	404.1	372.1	5.96	71	10	19	15
Azoxystrobin-[B48] 2	404.1	344.1	5.96	71	10	27	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Benalaxyl-[B45] 1	326.2	148.1	8.01	91	10	31	15
Benalaxyl-[B45] 2	326.2	294.1	8.01	91	10	17	15
Bendiocarb-[658] 1	224.1	167.1	5.02	81	10	15	15
Bendiocarb-[658] 2	224.1	109	5.02	81	10	27	15
Bifenazate-[B82] 1	301.1	198.1	6.78	81	10	15	15
Bifenazate-[B82] 2	301.1	170.1	6.78	81	10	29	15
Bitertanol-[850] 1	338.2	269.2	8.16	71	10	13	15
Bitertanol-[850] 2	338.2	99.1	8.16	62	10	21	4
Boscalid-[B75] 1	343	307	6.38	111	10	27	15
Boscalid-[B75] 2	343	140	6.38	111	10	27	15
BromuconazoleA-[B20] 1	378	159	6.93	81	10	37	15
BromuconazoleA-[B20] 2	378	70	6.93	81	10	43	15
BromuconazoleB-[B20] 1	378.1	159.1	7.71	81	10	37	15
BromuconazoleB-[B20] 2	378.1	161	7.71	81	10	39	9
Bupirimate-[872] 1	317	166.1	7.31	106	10	33	15
Bupirimate-[872] 2	317	108	7.31	106	10	37	15
Buprofezin-[B52] 1	306.2	201.1	8.9	66	10	17	15
Buprofezin-[B52] 2	306.2	116.2	8.9	66	10	21	15
Butachlor-[806] 1	312.1	238	9	60	10	31	12
Butachlor-[806] 2	312.1	162	9	60	10	15	12
Butocarboxim-[857] 1	208.1	116	4.75	80	10	13	9
Butocarboxim-[857] 1	213.1	75	4.69	70	10	20	15
Butocarboxim-[857] 2	208.1	75	4.75	80	10	13	9
Butocarboxim-[857] 2	213.1	116	4.69	70	10	13	15
Carbaryl-[102] 1	202.1	145	5.24	76	10	15	15
Carbaryl-[102] 2	202.1	127	5.24	76	10	41	15
Carbendazim-[666] 1	192.2	160.2	3.72	100	10	24	15
Carbendazim-[666] 2	192.2	132.1	3.72	100	10	41	15
Carbetamide-[875] 1	237.1	192	4.83	76	10	13	15
Carbetamide-[875] 2	237.1	118.1	4.82	76	10	19	15
Carbofuran-[180] 1	222.1	123	5.08	86	10	31	15
Carbofuran-[180] 2	222.1	165.1	5.08	86	10	19	15
Carboxin-[210] 1	236	143	5.29	80	10	22	15
Carboxin-[210] 2	236	87	5.29	80	10	35	15
Chlorantraniliprole-[C86] 1	484	452.9	5.86	86	10	23	15
Chlorantraniliprole-[C86] 2	484	285.9	5.86	86	10	19	15
Chlorfenvinphos-[B74] 1	359	155	7.99	85	10	18	15
Chlorfenvinphos-[B74] 2	359	99	7.99	85	10	41	15
Chlorfluazuron-[C72] 1	540	383	9.21	111	10	27	15
Chlorfluazuron-[C72] 2	540	158	9.21	111	10	27	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Chlorimuron-ethyl-[717] 1	415	185	6.43	100	10	35	12
Chlorimuron-ethyl-[717] 2	415	186	6.43	100	10	29	16
Chlorotoluron [862] 1	213.1	72.2	5.59	61	10	31	4
Chlorotoluron [862] 2	213.1	46.2	5.59	61	10	27	4
Chlorpyrifos-[160] 1	350	96.9	9.11	76	10	47	4
Chlorpyrifos-[160] 2	350	198	9.11	56	10	25	15
Chlorpyrifos-d10 1	360	199	9.14	71	10	29	13
Chlorpyrifos-d10 2	360	99	9.14	71	10	53	13
Chlorpyrifos-methyl-[235] 1	322	125.1	8.49	81	10	42	15
Chlorpyrifos-methyl-[235] 2	324	125.1	8.49	81	10	45	15
Clethodim-[B93] 1	360.1	164	8.69	81	10	29	15
Clethodim-[B93] 2	360.1	268.1	8.69	81	10	17	15
Clofentezine-[699] 1	303	138	8.23	81	10	23	15
Clofentezine-[699] 2	303	102	8.23	81	10	53	15
Clothianidin-[B95] 1	250	132	3.95	71	10	21	15
Clothianidin-[B95] 2	250	169	3.95	71	10	17	15
Coumaphos-[124] 1	362.9	227	7.94	71	10	37	10
Coumaphos-[124] 2	362.9	306.9	7.94	71	10	25	10
Cumyluron-[D14] 1	303.1	125	6.81	50	10	50	15
Cumyluron-[D14] 2	303.1	185.2	6.81	46	10	19	10
Cyantraniliprole-[D46] 1	475.01	286	5.18	66	10	19	20
Cyantraniliprole-[D46] 2	475.01	284	5.18	66	10	19	18
Cyazofamid-[C48] 1	325	108	7.15	81	10	21	15
Cyazofamid-[C48] 2	325	261.1	7.15	81	10	15	15
Cycloxydime-[D28] 1	326.2	280	6.1	66	10	19	14
Cycloxydime-[D28] 2	326.2	180	6.1	66	10	29	16
Cymoxanil-[877] 1	199	128	4.37	80	10	13	15
Cymoxanil-[877] 2	199	111	4.37	80	10	25	15
CyproconazoleA-[A22] 1	292	70	6.73	86	10	39	15
CyproconazoleA-[A22] 2	292	125	6.73	86	10	45	15
CyproconazoleB-[A22] 1	292.1	70.1	7	86	10	39	15
CyproconazoleB-[A22] 2	292.1	125.1	7	86	10	45	15
Cyprodinil-[B22] 1	226	93	7.8	121	10	51	15
Cyprodinil-[B22] 2	226	77	7.8	121	10	65	15
Cyromazine-[255] 1	167.1	85.1	2.67	81	10	27	15
Cyromazine-[255] 2	167.1	125.1	2.67	81	10	27	15
Daimuron-[D06] 1	269.2	151.1	6.73	46	10	19	10
Daimuron-[D06] 2	269.2	91	6.73	46	10	57	10
Desmedipham-[786] 1	318.1	136	5.71	61	10	36	15
Desmedipham-[786] 2	318.1	182	5.71	61	10	19	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Diafenthiuron-[D10] 1	385.6	329.3	9.28	76	10	27	15
Diafenthiuron-[D10] 2	385.6	278.1	9.28	76	10	43	15
Diazinon-[024] 1	305	169.1	8.09	86	10	31	10
Diazinon-[024] 2	305	153.1	8.09	86	10	29	10
Dichlorvos-[338] 1	221	109	5.01	61	10	25	15
Dichlorvos-[338] 2	221	127	5.01	61	10	27	15
Dicrotophos-[209] 1	238.1	193	3.73	66	10	15	14
Dicrotophos-[209] 2	238.1	112.1	3.73	66	10	19	8
Diethofencarb-[B62] 1	268.1	226.1	6.23	81	10	15	15
Diethofencarb-[B62] 2	268.1	124	6.23	81	10	45	15
Difenoconazole-[B58] 1	406.1	251.1	8.36	101	10	37	15
Difenoconazole-[B58] 2	408.2	253.1	8.36	96	10	33	15
Diflubenzuron-[651] 1	311	158.2	7.35	91	10	23	15
Diflubenzuron-[651] 2	311	141.1	7.35	91	10	45	15
Dimethoate-[171] 1	230	199	4.17	70	10	14	15
Dimethoate-[171] 2	230	125	4.17	70	10	27	15
DimethomorphA-[B77] 1	388.1	301	6.21	86	10	25	15
DimethomorphA-[B77] 2	388.1	165.1	6.21	86	10	45	15
DimethomorphB-[B77] 1	388.2	301.1	6.52	86	10	25	15
DimethomorphB-[B77] 2	388.2	165.2	6.52	86	10	45	15
Diniconazole-[D01] 1	326	70	8.37	76	10	45	15
Diniconazole-[D01] 2	326	159	8.37	71	10	39	15
Dinotefuran-[C75] 1	203.1	129.2	3.23	71	10	19	15
Dinotefuran-[C75] 2	203.1	157.2	3.23	71	10	13	15
Dioxacarb-[656] 1	224.1	123	4.16	71	10	23	15
Dioxacarb-[656] 2	224.1	167	4.16	71	10	13	15
Dithianon-[XAL] 1	297	181	5.01	56	10	21	14
Dithianon-[XAL] 2	297	280	5.01	56	10	11	12
Diuron-[032] 1	235.1	72.1	5.81	76	10	39	15
Diuron-[032] 2	233.1	72	5.82	76	10	33	15
DMST-[D30] 1	215	106	5.1	81	10	21	10
DMST-[D30] 2	215	77	5.1	81	10	57	10
DNOC-[029] 1	199.2	117	4.56	76	10	41	15
DNOC-[029] 2	199.2	46.1	3.97	76	10	39	15
Dodine-[104] 1	228	57	7.68	86	10	35	13
Dodine-[104] 2	228	60	7.67	86	10	35	13
Doramectin-[C62] 1	916.9	331.5	10.2	81	10	31	15
Doramectin-[C62] 2	916.9	593.6	10.2	86	10	19	15
Emamectin-Benzoate-[C58] 1	886.5	82.1	8.64	131	10	127	15
Emamectin-Benzoate-[C58] 2	886.5	158.1	8.64	131	10	51	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
EPN-[035] 1	324	296	8.5	56	10	19	4
EPN-[035] 2	324	157.1	8.5	56	10	31	4
Epoxiconazole-[B53] 1	330	121	7.22	56	10	27	15
Epoxiconazole-[B53] 2	330	101	7.22	61	10	63	15
Eprinomectin-[C59] 1	914.5	186.2	9.53	96	10	27	15
Eprinomectin-[C59] 2	914.5	154.2	9.55	96	10	59	15
Ethiofencarb sulfoxide-[D31] 1	242	107.1	3.88	91	10	27	10
Ethiofencarb sulfoxide-[D31] 2	242	185	3.88	91	10	13	14
Ethion-[107] 1	385	199	8.94	36	10	17	15
Ethion-[107] 2	385	142.9	8.94	86	10	39	8
Ethiprole-[C64] 1	397.3	350.9	6.2	101	10	29	15
Ethiprole-[C64] 2	397.3	255.2	6.2	101	10	49	15
Ethirimol-[802] 1	210.2	140.1	4.83	101	10	31	15
Ethirimol-[802] 2	210.2	98.1	4.83	101	10	39	15
Ethofumesate-[945] 1	304	121	6.27	56	10	27	15
Ethofumesate-[945] 2	304	161	6.27	31	10	31	15
Ethoprop-[175] 1	243	97	7.33	66	10	43	10
Ethoprop-[175] 2	243	173	7.33	61	10	21	15
Etoxazole-[B84] 1	360.1	141	9.19	96	10	45	15
Etoxazole-[B84] 2	360.1	57.2	9.19	96	10	45	15
Famoxadone-[C01] 1	392	331	7.82	51	10	15	15
Famoxadone-[C01] 2	392	238	7.82	56	10	23	15
Fenamidone-[B64] 1	312.1	92	6.3	86	10	39	15
Fenamidone-[B64] 2	312.1	236.1	6.3	86	10	21	15
Fenarimol-[271] 1	331	268	7.08	66	10	31	15
Fenarimol-[271] 2	331	81	7.08	66	10	47	15
Fenazaquin-[B73] 1	307.1	161.1	9.84	91	10	25	15
Fenazaquin-[B73] 2	307.1	147	9.84	91	10	29	15
Fenbuconazole-[A30] 1	337	124.9	7.21	101	10	41	15
Fenbuconazole-[A30] 2	337	70	7.21	101	10	39	15
Fenbutatin oxide-[639] 1	517.3	118	11.3	100	10	129	12
Fenbutatin oxide-[639] 2	517.3	195.2	11.3	100	10	70	14
Fenhexamid-[B41] 1	302	97	7.04	86	10	35	15
Fenhexamid-[B41] 2	302	55	7.04	86	10	71	15
Fenobucarb-[C91] 1	208.1	95.1	6.21	81	10	21	15
Fenobucarb-[C91] 2	208.1	152.1	6.21	81	10	13	15
Fenoxycarb-[811] 1	302.1	88	7.45	86	10	31	15
Fenoxycarb-[811] 2	302.1	116.1	7.45	86	10	17	15
Fenpropimorph-[886] 1	304	147	5.97	66	10	39	15
Fenpropimorph-[886] 2	304	117	5.97	81	10	71	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
FenpyroximateE-[C73] 1	422	366.1	9.38	76	10	23	15
FenpyroximateE-[C73] 2	422	135.1	9.38	76	10	43	15
Fenuron-[840] 1	165.1	72.1	4.19	76	10	25	15
Fenuron-[840] 2	165.1	46	4.19	76	10	29	15
Flonicamid-[C92] 1	230.1	174	3.62	75	10	35	15
Flonicamid-[C92] 2	230.1	203.1	3.62	75	10	35	15
Flubendiamide-[C87] 1	683	408	7.44	76	10	17	15
Flubendiamide-[C87] 2	683	274	7.44	76	10	43	15
Fludioxonil-[B23] 1	266	229	6.29	61	10	23	15
Fludioxonil-[B23] 2	266	227.1	6.29	61	10	13	15
Flufenoxuron-[C71] 1	489	158	9	106	10	29	15
Flufenoxuron-[C71] 2	489	141.1	9	106	10	63	15
Fluopyram [D32] 1	397	208	6.92	81	10	31	12
Fluopyram [D32] 2	397	173	6.92	81	10	39	14
Fluoxastrobin-[C66] 1	459.2	427.2	6.8	75	10	28	15
Fluoxastrobin-[C66] 2	459.2	188	6.8	75	10	35	15
Fluquinconazole-[B78] 1	376	307	6.98	71	10	33	10
Fluquinconazole-[B78] 2	376	349	6.98	76	10	27	15
Fluridone [736] 1	330.1	310.1	5.92	56	10	41	24
Fluridone [736] 2	330.1	309.1	5.92	56	10	51	24
Flusilazole-[950] 1	316.1	247.1	7.37	101	10	27	15
Flusilazole-[950] 2	316.1	165.1	7.37	101	10	39	15
Flutolanil-[B63] 1	324.1	262.1	6.43	96	10	27	15
Flutolanil-[B63] 2	324.1	242.1	6.43	96	10	37	15
Flutriafol-[C53] 1	302.1	70.1	5.55	86	10	37	15
Flutriafol-[C53] 2	302.1	123	5.55	86	10	41	15
Fluxapyroxad-[D45] 1	382.1	362.1	6.5	91	10	19	18
Fluxapyroxad-[D45] 2	382.1	342.1	6.5	91	10	29	12
Fomesafen-[D19] 1	456	344	6.05	66	10	20	10
Fomesafen-[D19] 2	456	223	6.05	66	10	40	10
Forchlorfenuron-[B32] 1	248	129.1	5.7	72	10	25	15
Forchlorfenuron-[B32] 2	248	93.1	5.7	72	10	48	15
Formetanate-[E19] 1	222.1	165	2.91	96	10	23	15
Formetanate-[E19] 2	222.1	120	2.92	80	10	39	8
Fuberidazole-[887] 1	185	157	4.15	101	10	33	15
Fuberidazole-[887] 2	185	65	4.15	101	10	67	15
Furalaxyl-[D22] 1	302.1	95	6.15	76	10	41	18
Furalaxyl-[D22] 2	302.1	242.1	6.15	76	10	23	16
Halofenozide-[C93] 1	331.1	105.1	6.3	61	10	25	15
Halofenozide-[C93] 2	331.1	275	6.3	61	10	11	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Hexaconazole [954] 1	314.1	159	8.2	46	10	37	4
Hexaconazole [954] 2	314.1	70	8.2	56	10	39	4
Hexythiazox-[B10] 1	353.1	228	9.11	81	10	23	15
Hexythiazox-[B10] 2	353.1	168	9.12	81	10	37	15
Imazalil-[604] 1	297	159	5.23	86	10	33	15
Imazalil-[604] 2	297	201	5.22	86	10	27	15
Imazasulfuron-[T07] 1	413	153	6.46	70	10	17	16
Imazasulfuron-[T07] 2	413	156	6.45	70	10	27	12
Imidacloprid-[967] 1	256	209.1	3.87	81	10	23	15
Imidacloprid-[967] 2	256	175.1	3.87	81	10	29	15
Indoxacarb-[C12] 1	528	203	8.34	106	10	55	15
Indoxacarb-[C12] 2	528	150.1	8.34	116	10	30	4
Iprovalicarb-[C46] 1	321.2	119	7.05	86	10	29	15
Iprovalicarb-[C46] 2	321.2	203.1	7.05	86	10	13	15
Isocarbamid-[A36] 1	186.2	87.1	4.45	61	10	23	15
Isocarbamid-[A36] 2	186.2	44.2	4.45	61	10	49	15
Isocarbophos-[C77] 1	273.2	231	5.68	80	10	31	10
Isocarbophos-[C77] 2	273.2	199	5.69	80	10	31	10
Isoprocarb-[637] 1	194.1	95	5.63	81	10	23	15
Isoprocarb-[637] 2	194.1	137	5.62	81	10	13	15
Isoprothiolane-[855] 1	291	189	6.62	61	10	31	15
Isoprothiolane-[855] 2	291	231.1	6.62	61	10	17	15
Ivermectin-[C61] 1	897.7	753.6	10.7	85	10	65	8
Ivermectin-[C61] 2	897.7	609.5	10.7	85	10	65	18
Karbutilate-[D33] 1	280	181	4.97	170	10	17	14
Karbutilate-[D33] 2	280	72	4.97	170	10	53	10
Kresoxim-methyl-[B42] 1	314	267.2	7.77	71	10	10	4
Kresoxim-methyl-[B42] 2	314	116	7.76	71	10	21	15
Linuron-[129] 1	249.1	160	6.3	81	10	23	15
Linuron-[129] 2	249.1	182.1	6.3	81	10	21	15
Lufenuron-[C34] 1	511.1	158.1	8.76	81	10	27	15
Lufenuron-[C34] 2	511.1	141.2	8.76	81	10	67	15
Malathion-[052] 1	331	127	6.59	91	10	19	10
Malathion-[052] 2	331	285	6.59	91	10	11	6
Mandipropamide-[C94] 1	412.1	328.1	6.23	101	10	21	15
Mandipropamide-[C94] 2	412.1	125.1	6.23	80	10	60	4
Mepanipyrim-[C55] 1	224	77	7.13	106	10	59	15
Mepanipyrim-[C55] 2	224	106	7.13	106	10	37	15
Mepronil-[D23] 1	270	119.1	6.67	65	10	32	15
Mepronil-[D23] 2	270	91.1	6.67	65	10	61	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Metaflumizone-[C40] 1	507.1	287.1	8.65	121	10	37	15
Metaflumizone-[C40] 2	507.1	178.1	8.65	121	10	39	15
Metalaxyl-[607] 1	280.1	220.2	5.75	81	10	21	15
Metalaxyl-[607] 2	280.1	192.2	5.75	81	10	27	15
Metconazole-[C54] 1	320.1	70	8.15	101	10	51	15
Metconazole-[C54] 2	320.1	125	8.15	101	10	59	15
Methamidophos-[170] 1	142	94	2.83	75	10	20	15
Methamidophos-[170] 2	142	125	2.83	75	10	19	15
Methfuroxam-[D20] 1	230	137	6	76	10	29	15
Methfuroxam-[D20] 2	230	111.1	6	76	10	23	15
Methidathion-[197] 1	303	145.1	5.92	56	10	15	4
Methidathion-[197] 2	303	85.2	5.92	56	10	29	4
Methiocarb-[195] 1	226.1	169.1	6.38	81	10	13	15
Methiocarb-[195] 2	226.1	121.1	6.38	81	10	27	15
Methomyl-[159] 1	163.1	106	3.57	55	10	13	15
Methomyl-[159] 2	163.1	88.1	3.57	55	10	12	15
Methoxyfenozide-[C15] 1	369.1	149.1	6.55	76	10	25	15
Methoxyfenozide-[C15] 2	369.1	313.2	6.55	76	10	13	15
Metoxuron-[844] 1	229.1	72	4.62	86	10	31	10
Metoxuron-[844] 2	229.1	156	4.62	86	10	35	16
Metrafenone-[D13] 1	409	209.1	8.35	31	10	21	10
Metrafenone-[D13] 2	409	227.1	8.35	31	10	27	10
Metribuzin [181] 1	215.1	187.1	5.2	71	10	27	10
Metribuzin [181] 2	215.1	84.1	5.2	71	10	29	10
Metsulfuron-methyl-[C18] 1	382	167	4.87	70	10	23	14
Metsulfuron-methyl-[C18] 2	382	77	4.87	70	10	73	10
MevinphosE-[579] 1	225.1	127.1	4.09	75	10	20	15
MevinphosE-[579] 2	225.1	193.2	4.09	75	10	10	15
MevinphosZ-[578] 1	225	127	4.39	75	10	20	15
MevinphosZ-[578] 2	225	193.1	4.39	75	10	10	15
Mexacarbate-[681] 1	223.1	166.1	5.48	66	10	23	12
Mexacarbate-[681] 2	223.1	151	5.48	66	10	35	10
Monocrotophos-[343] 1	224.1	127.1	3.64	71	10	23	15
Monocrotophos-[343] 2	224.1	98	3.64	71	10	17	15
Moxidectin-[C63] 1	640.5	528.5	10.3	81	10	11	15
Moxidectin-[C63] 2	640.5	498.5	10.3	81	10	17	15
Myclobutanil-[679] 1	289	70	6.65	91	10	37	15
Myclobutanil-[679] 2	289	125	6.65	91	10	47	15
Nitenpyram-[C51] 1	271	225.2	3.39	71	10	17	15
Nitenpyram-[C51] 2	271	126	3.39	71	10	39	15

Compound	**Q1**	**Q3**	**RT**	**DP**	**EP**	**CE**	**CXP**
Novaluron-[C35] 1	493	141.1	8.41	91	10	69	15
Novaluron-[C35] 2	493	158.1	8.41	91	10	27	15
Nuarimol-[800] 1	315	252.1	6.25	101	10	31	15
Nuarimol-[800] 2	315	81	6.25	101	10	45	15
Omethoate-[178] 1	214	124.9	3.13	66	10	29	15
Omethoate-[178] 2	214	182.8	3.14	66	10	17	15
Oxadixyl-[A46] 1	279.1	219.1	4.66	81	10	17	15
Oxadixyl-[A46] 2	279.1	132.1	4.65	81	10	43	15
Oxamyl-[537] 1	237.1	72.1	3.34	56	10	25	15
Oxamyl-[537] 2	237.1	90.1	3.34	56	10	12	15
Paclobutrazol-[A48] 1	294	70	6.54	86	10	49	15
Paclobutrazol-[A48] 2	294	125	6.54	81	10	49	15
Parathion-[065] 1	292	236	7.7	76	10	21	18
Parathion-[065] 2	292	94	7.7	76	10	47	10
Penconazole [956] 1	284.1	159	7.81	61	10	39	4
Penconazole [956] 2	284.1	70	7.81	61	10	29	4
Pencycuron-[C95] 1	329.1	125	8.31	96	10	37	22
Pencycuron-[C95] 2	329.1	218.1	8.32	96	10	25	14
Phenmedipham-[791] 1	301.1	136	5.8	70	10	26	15
Phenmedipham-[791] 2	301.1	168.1	5.79	70	10	14	15
Phorate sulfone [189] 1	293	171.1	5.55	36	10	17	14
Phorate sulfone [189] 2	293	97.1	5.55	36	10	41	10
Phosmet [165] 1	318	160	6.03	40	10	19	15
Phosmet [165] 2	318	133	6.03	40	10	50	15
Phoxim-[247] 1	298.9	77	8.08	46	10	47	12
Phoxim-[247] 2	298.9	129	8.08	46	10	21	16
Picoxystrobin-[C68] 1	368	145	7.4	76	10	27	15
Picoxystrobin-[C68] 2	368	205	7.4	76	10	15	15
Piperonyl-Butoxide-[070] 1	356.2	177.2	9.03	71	10	19	15
Piperonyl-Butoxide-[070] 2	356.2	119.1	9.03	71	10	51	15
Pirimicarb-[580] 1	239.2	72.1	5.21	86	10	35	15
Pirimicarb-[580] 2	239.2	182.1	5.21	86	10	23	15
Pirimiphos-methyl-[562] 1	306.2	164.2	8.32	85	10	28	15
Pirimiphos-methyl-[562] 2	306.2	108.2	8.32	85	10	40	15
Prochloraz-[833] 1	376	308	8.1	66	10	17	15
Prochloraz-[833] 2	376	70	8.1	66	10	17	15
Profenofos [224] 1	374.9	304.9	8.8	96	10	27	8
Profenofos [224] 2	372.9	302.9	8.8	136	10	25	22
Prometryn-[249] 1	242.2	158.1	7.05	101	10	33	15
Prometryn-[249] 2	242.2	200.1	7.05	101	10	27	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Propamocarb-[C42] 1	189.2	102	3.13	81	10	25	15
Propamocarb-[C42] 2	189.2	144	3.13	81	10	19	15
Propargite-[623] 1	368.2	231.1	9.19	66	10	15	15
Propargite-[623] 2	368.2	175.1	9.19	66	10	23	15
Propiconazole-[264] 1	342.1	159	8.01	81	10	39	15
Propiconazole-[264] 2	342.1	69	8.01	81	10	37	15
Propoxur-[162] 1	210.1	111	5.04	59	10	19	15
Propoxur-[162] 2	210.1	168.1	5.04	59	10	11	15
Proquinazid-[D35] 1	373	331	9.73	86	10	21	16
Proquinazid-[D35] 2	373	289	9.72	86	10	33	18
Pymetrozine-[C22] 1	218	105	3.06	91	10	27	15
Pymetrozine-[C22] 2	218	78	3.05	91	10	47	15
Pyraclostrobin-[B61] 1	388	194	8.06	51	10	19	15
Pyraclostrobin-[B61] 2	388	163	8.06	51	10	29	15
Pyridaben-[B56] 1	365	309	9.67	66	10	19	15
Pyridaben-[B56] 2	365	147	9.67	66	10	31	15
Pyrimethanil-[B16] 1	200	107	6.41	91	10	33	15
Pyrimethanil-[B16] 2	200	82	6.41	91	10	35	15
Pyriproxyfen-[B24] 1	322	96	9.04	66	10	21	15
Pyriproxyfen-[B24] 2	322	185	9.04	66	10	29	15
Quinoxyfen-[B57] 1	308	197.1	9.13	81	10	45	15
Quinoxyfen-[B57] 2	308	162.1	9.13	81	10	65	15
Rotenone-[020] 1	395.1	213.1	7.42	111	10	33	15
Rotenone-[020] 2	395.1	192.1	7.42	111	10	35	15
Siduron-[C24] 1	233.3	94	6.38	86	10	31	15
Siduron-[C24] 2	233.3	137.2	6.38	86	10	21	15
SpinetoramA-[C88] 1	748.5	142.2	8.27	106	10	45	15
SpinetoramA-[C88] 2	748.5	98.1	8.26	106	10	109	15
SpinetoramB-[C88] 1	760.5	142.2	8.58	116	10	41	15
SpinetoramB-[C88] 2	760.5	98.1	8.58	116	10	101	15
Spinosad A-[C25] 1	732.5	142.2	7.77	131	10	43	15
Spinosad A-[C25] 2	732.5	98.1	7.77	131	10	103	15
Spinosyn D [D03] 1	746.5	142.2	8.2	96	10	43	10
Spinosyn D [D03] 2	746.5	98.1	8.2	96	10	103	18
Spirodiclofen-[B85] 1	411.3	71.3	9.41	91	10	33	15
Spirodiclofen-[B85] 2	411.3	313.3	9.41	91	10	25	15
Spiromesifen-[C60] 1	388.2	273.2	9.16	61	10	19	15
Spiromesifen-[C60] 2	388.2	255.2	9.17	61	10	39	15
Spirotetramat-[C96] 1	374.2	302.2	6.99	86	10	25	15
Spirotetramat-[C96] 2	374.2	330.2	6.99	86	10	23	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Spiroxamine-[C98] 1	298.2	144.2	5.88	96	10	29	15
Spiroxamine-[C98] 2	298.2	100.1	5.87	96	10	47	15
Sulfoxaflor+NH4-[D48] 1	295.08	174	4.2	31	10	19	14
Sulfoxaflor+NH4-[D48] 2	295.08	154	4.2	31	10	45	14
Tebuconazole-[A58] 1	308.2	70	7.84	101	10	49	15
Tebuconazole-[A58] 2	308.2	125	7.85	101	10	51	15
Tebufenozide-[C56] 1	353.2	133	7.46	71	10	25	15
Tebufenozide-[C56] 2	353.2	297.2	7.45	71	10	13	15
Tebufenpyrad [C39] 1	334	117	8.85	71	10	47	15
Tebufenpyrad [C39] 2	334	145	8.85	71	10	37	15
Tebuthiuron-[780] 1	229.1	172.4	5.22	66	10	21	15
Tebuthiuron-[780] 2	229.1	116.1	5.22	66	10	35	15
Teflubenzuron-[C99] 1	381.1	158.2	8.85	86	10	23	15
Teflubenzuron-[C99] 2	381.1	141.2	8.85	86	10	53	15
Temephos-[749] 1	467	405	8.77	121	10	23	15
Temephos-[749] 2	467	419.1	8.77	121	10	29	15
Tepraloxydim-[D36] 1	342	250	7.04	55	10	19	14
Tepraloxydim-[D36] 2	342	166	7.05	55	10	29	12
Tetraconazole-[B72] 1	372	159	6.97	66	10	39	15
Tetraconazole-[B72] 2	372	70	6.96	56	10	47	15
Thiabendazole-[157] 1	202.1	175.1	4.01	105	10	35	15
Thiabendazole-[157] 2	202.1	131.2	4.01	105	10	45	15
Thiacloprid-[B68] 1	253	126	4.28	91	10	31	15
Thiacloprid-[B68] 2	253	99	4.27	91	10	61	15
Thifluzamide-[F27] 1	528.85	148	7.23	136	10	59	12
Thifluzamide-[F27] 2	528.85	107	7.23	136	10	97	12
Thiamethoxam-[B43] 1	292	211	3.59	81	10	19	15
Thiamethoxam-[B43] 2	292	181	3.58	81	10	33	15
Thiodiarb-[943] 1	355	88	5.19	65	10	25	13
Thiodiarb-[943] 2	355	108	5.19	65	10	21	10
Thiophanate-methyl-[611] 1	343	151.1	4.89	81	10	29	15
Thiophanate-methyl-[611] 2	343	311	4.9	81	10	17	15
Tolfenpyrad-[D52] 1	384.15	197.1	8.93	86	10	37	16
Tolfenpyrad-[D52] 2	384.15	154.1	8.93	86	10	57	12
Triadimefon-[608] 1	294	197.1	6.72	86	10	23	15
Triadimefon-[608] 2	294	225	6.73	86	10	19	15
Triadimenol-[638] 1	296.1	70	6.91	46	10	31	15
Triadimenol-[638] 2	298.1	70	6.93	20	10	20	15
Triazophos-[536] 1	314	162	6.78	56	10	25	15
Triazophos-[536] 2	314	119	6.78	56	10	49	15

Compound	Q1	Q3	RT	DP	EP	CE	CXP
Trichlorfon-[130] 1	256.9	109.1	4.21	86	10	25	15
Trichlorfon-[130] 2	256.9	127	4.21	86	10	25	15
Tricyclazole-[804] 1	190	163	4.53	101	10	33	15
Tricyclazole-[804] 2	190	136	4.53	101	10	41	15
Tridemorph-[795] 1	298.1	130	7.65	96	10	33	10
Tridemorph-[795] 2	298.1	98	7.65	96	10	37	13
Trifloxystrobin-[B79] 1	409	186	8.44	51	10	23	15
Trifloxystrobin-[B79] 2	409	206	8.44	51	10	21	15
Triflumizole-[A61] 1	346.1	278.1	8.52	71	10	15	15
Triflumizole-[A61] 2	346.1	73	8.52	71	10	27	15
Triflusulfuron methyl ester [A62] 1	493.1	264	6.26	40	10	35	18
Triflusulfuron methyl ester [A62] 2	493.1	96	6.26	40	10	75	12
Triticonazole-[C33] 1	318.1	70	7.13	86	10	45	15
Triticonazole-[C33] 2	318.1	125	7.14	86	10	41	15
Zoxamide-[B44] 1	336.1	187	8.05	120	10	47	18
Zoxamide-[B44] 2	336.1	159	8.06	120	10	47	18

Q1 and Q3 are the *m/z* values of the parent and transition ions.
RT is the retention time in mins.
DP, EP and CXP are the clustering potential, entrance potential, collision energy and collision exit potentials (in mV), respectively.
CE is the collision energy (in eV).