Development of analytical methods: Validation of MRM extraction methods for high protein content pulses

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1. Aim and scope

This document reports the validation data at LOQ for 258 pesticides, most of them included in the European Union Multi Annual Control Program (EU-MACP) [1] and the Working Document SANCO/12745/2013 [2], using multiresidue methods and analyzed by liquid chromatography coupled to triple quadrupole mass spectrometry (LC-MS/MS). Four different approaches have been validated for the extraction of pesticide residues in white beans.

2. Short description

New approaches for the extraction of pesticide residues in white beans, within the high starch and/or protein content and low water and fat content commodity group, have been developed and evaluated. With that purpose, homogeneous samples have been spiked at 0.010 mg/kg concentration level and extracted with different modifications of the QuEChERS citrate method. (M01) QuEChERS citrate extraction, (M02) QuEChERS citrate increasing time of shaking, (M03) QuEChERS citrate without hydratation, and (M04) QuEChERS citrate with sodium docecyl sulphate (SDS) addition, all methods using freezing-out at the pre-clean-up stage. The obtained extracts have been analyzed by LC-MS/MS.
The validation of the extraction methods has been performed in terms of accuracy (recovery at 0.010 mg/kg), repeatability (5 replicates) and matrix effect.

3. Apparatus and consumables

- Automatic pipettes, suitable for handling volumes from 1 µL to 5000 µL and from 1 mL to 5 mL.
- Graduated 10 mL pipette.
- 50 mL and 15 mL PTFE centrifuge tubes.
- Vortex Shaker IKATM 4 Basic.
- Axial shaker Agytax SR1 CP57.
- Centrifuge Orto Alresa Consul 21, suitable for the centrifuge tubes employed in the procedure and capable of achieving at least 4000 rpm.
- Injection vials, 2 mL, suitable for LC and GC auto-sampler.

4. Chemicals

- Acetonitrile ultra-gradient grade (AcN)
- Trisodium citrate dihydrate
- Disodium hydrogenocitrate sesquihydrate
- Sodium chloride

- Anhydrous magnesium sulphate
- Anhydrous calcium chloride
- Primary secondary amine (PSA)
- Sodium dodecyl sulphate (SDS)
- Ammonium formate
- Ultra-pure water
- Formic acid
- Pesticide analytical standards
- Dry ice

5. Procedure

5.1. Sample preparation

The study matrix is dry white beans. It is a type of matrix with low water content and high protein content. To carry out the grinding of the samples and their reduction to an analytical sample, all parts of the sample are taken as specified in Annex I to COMMISSION Regulation (EU) No 752/2014 of 24 June 2014 (replacing Annex I to Regulation (EC) No 396/2005).

5.2. Pesticide stock solutions and working mix solutions.

Individual pesticide stock solutions (1000–2000 mg/L) were prepared in acetonitrile and were stored in screw-capped glass vials in the dark at -20 °C.

For spiking, representative portions of the previously homogenized samples were spiked homogenously with the appropriate amount of the working standard solution in acetonitrile. The validation methods were performed at a fortification level of 0.010 mg/kg. Five replicates were analysed at each level.

5.3. Extraction procedure

Method M01:

1. Weigh 5 g ± 0.05 g of sample in 50 mL PTFE centrifuge tube.

2. Add 10 mL of water for hydrate the sample.

3. Add 10 mL of acetonitrile and 50 µL of 1 mg/L carbendazim-d3, malathion-d10 and Dichlorvos D6 (procedure internal standards).

4. Shake the sample using an automatic axial shaker for 6 min.

5. Add 6.5 g ± 0.1 g of the prepared salt mixture or from the individual reagents (sodium chloride (1 g ± 0.1), magnesium sulphate (4 g ± 0.1), disodium hydrogen citrate 1 ½ hydrate (0.5 g ± 0.1), sodium citrate (1 g ± 0.1)).

6. Shake the samples again in the automatic shaker for 6 min.

7. Centrifuge the tubes at 3700 rpm for 10 min.

8. Take 8 mL of the supernatant with a micropipette and transfer to a 15 mL Falcon tube. Place the tube on dry ice for 20 min and centrifuge for 1 min at 3700 rpm.

9. Transfer 5 mL of the supernatant to a 15 mL PTFE tube containing 750 mg magnesium sulphate and 125 mg PSA.

10. Vortex the tube for 30 sec.

11. Centrifuge the tubes at 3700 rpm for 5 min.

12. Transfer the supernatant to a 4 ml vial, add 10 µl per ml extract of 5% formic acid solution in acetonitrile.

13. Analysis: LC-MS/MS: dilute 100 µL extract with 400 µL of water with 62.5 ppb Dimethoate-D6 (Injection Internal Standard) and 750 ppm of ascorbic acid.

Method M02:

1. Weigh 5 g ± 0.05 g of sample in 50 mL PTFE centrifuge tube.

2. Add 10 mL of water for hydrate the sample.

3. Add 10 mL of acetonitrile and 50 µL of 1 mg/L carbendazim-d3, malathion-d10 and Dichlorvos D6 (procedure internal standards).

4. Shake the sample using an automatic axial shaker for 30 min.

5. Add 6.5 g ± 0.1 g of the prepared salt mixture or from the individual reagents (sodium chloride (1 g ± 0.1), magnesium sulphate (4 g ± 0.1), disodium hydrogen citrate 1 ½ hydrate (0.5 g ± 0.1), sodium citrate (1 g ± 0.1)).

6. Shake the samples again in the automatic shaker for 6 min.

7. Centrifuge the tubes at 3700 rpm for 10 min.

8. Take 8 mL of the supernatant with a micropipette and transfer to a 15 mL Falcon tube. Place the tube on dry ice for 20 min and centrifuge for 1 min at 3700 rpm.

9. Transfer 5 mL of the supernatant to a 15 mL PTFE tube containing 750 mg magnesium sulphate and 125 mg PSA.

10. Vortex the tube for 30 sec.

11. Centrifuge the tubes at 3700 rpm for 5 min.

12. Transfer the supernatant to a 4 ml vial, add 10 µl per ml extract of 5% formic acid solution in acetonitrile.

13. Analysis: LC-MS/MS: dilute 100 µL extract with 400 µL of water with 62.5 ppb Dimethoate-D6 (Injection Internal Standard) and 750 ppm of ascorbic acid.

Method M03:

1. Weigh 5 g ± 0.05 g of sample in 50 mL PTFE centrifuge tube.

2. Add 10 mL of acetonitrile and 50 µL of 1 mg/L carbendazim-d3, malathion-d10 and Dichlorvos D6 (procedure internal standards).

3. Shake the sample using an automatic axial shaker for 6 min.

4. Add 6.5 g ± 0.1 g of the prepared salt mixture or from the individual reagents (sodium chloride (1 g ± 0.1), magnesium sulphate (4 g ± 0.1), disodium hydrogen citrate 1 ½ hydrate (0.5 g ± 0.1), sodium citrate (1 g ± 0.1)).

5. Shake the samples again in the automatic shaker for 6 min.

6. Centrifuge the tubes at 3700 rpm for 10 min.

7. Take 8 mL of the supernatant with a micropipette and transfer to a 15 mL Falcon tube. Place the tube on dry ice for 20 min and centrifuge for 1 min at 3700 rpm.

8. Transfer 5 mL of the supernatant to a 15 mL PTFE tube containing 750 mg magnesium sulphate and 125 mg PSA.

9. Vortex the tube for 30 sec.

10. Centrifuge the tubes at 3700 rpm for 5 min.

11. Transfer the supernatant to a 4 ml vial, add 10 µl per ml extract of 5% formic acid solution in acetonitrile.

12. Analysis: LC-MS/MS: dilute 100 µL extract with 400 µL of water with 62.5 ppb Dimethoate-D6 (Injection Internal Standard) and 750 ppm of ascorbic acid.

Method M04:

1. Weigh 5 g ± 0.05 g of sample in 50 mL PTFE centrifuge tube.

2. Add 10 ml of water with 0.25 g of dissolved SDS to hydrate the sample.

3. Add 10 mL of acetonitrile and 50 µL of 1 mg/L carbendazim-d3, malathion-d10 and Dichlorvos D6 (procedure internal standards).

4. Shake the sample using an automatic axial shaker for 6 min.

5. Add 6.5 g ± 0.1 g of the prepared salt mixture or from the individual reagents (sodium chloride (1 g ± 0.1), magnesium sulphate (4 g ± 0.1), disodium hydrogen citrate 1 ½ hydrate (0.5 g ± 0.1), sodium citrate (1 g ± 0.1)).

6. Shake the samples again in the automatic shaker for 6 min.

7. Centrifuge the tubes at 3700 rpm for 10 min.

8. Take 8 mL of the supernatant with a micropipette and transfer to a 15 mL Falcon tube. Place the tube on dry ice for 20 min and centrifuge for 1 min at 3700 rpm.

9. Transfer 5 mL of the supernatant to a 15 mL PTFE tube containing 750 mg magnesium sulphate and 125 mg PSA.

10. Vortex the tube for 30 sec.

11. Centrifuge the tubes at 3700 rpm for 5 min.

12. Transfer the supernatant to a 4 ml vial, add 10 µl per ml extract of 5% formic acid solution in acetonitrile.

13. Analysis: LC-MS/MS: dilute 100 µL extract with 400 µL of water with 62.5 ppb Dimethoate-D6 (Injection Internal Standard) and 750 ppm of ascorbic acid.

5.4. Methodology

All samples were analysed by LC-MS/MS system operated in multiple reaction monitoring mode (MRM). Selected reaction monitoring (SRM) experiments were carried out to obtain the maximum sensitivity for the detection of the target molecules. Two SRM transitions and a correct ratio between the abundance of the two optimised SRM transitions (SRM2/SRM1) were used for confirmation of the studied compounds, along with retention time matching. The mass transitions used are presented in **Appendix I (Table A1)**.

5.5. Instrumentation and analytical conditions for the LC- MS/MS system

5.5.1. UHPLC (Thermo ScientificTM TranscendTM DUO LX-2-LC)

- Column: Accucore C18 2.1x100 mm and 2.6 µm particle size (Thermo Scientific™)
- Mobile phase A: Water (0.1 % formic acid, 5 mM ammonium formate, 2 % MeOH)
- Mobile phase B: Methanol (0.1 % formic acid, 5 mM ammonium formate, 2 % water)
- Column temperature: 30 °C
- Flow rate: 0.35 ml/min
- Injection volume: 2.5 µL
- Autosampler temperature: 10 °C

Mobile phase gradient for pesticides analysis:

min	Mobile phase A%	Mobile phase B %
0	100	0
1	100	0
2	70	30
3	50	50
11	0	100
14.85	0	100
14.95	100	0
20.9	100	0
Data window [min]	1.1-11.55	

5.5.2. Triple quadrupole system (Thermo Scientific™ TSQ Altis™)
Ion source: Opta Max NG

- Positive ion spray voltage: 3500 V
- Negative ion spray voltage: 2500 V
- Sheath gas: 50
- Aux gas: 10
- Sweep gas: 1
- Ion transfer tube temperature: 325 °C
- Vaporizer temperature: 350 °C

6. Results

6.1. Method validation

6.1.1. Recoveries and within-laboratory reproducibility:

The results corresponding to the mean recovery (n = 5) and within-laboratory reproducibility in terms of relative standard deviation (RSDr) at a fortification level of 0.010 mg/kg are summarized in **Appendix I (Table A2)**.

The relative standard deviation between the five replicates was found to be less than 20 % for most of the compounds. In the case of M02 it is the method with the highest number of compounds exceeding 20 % relative standard deviation (10 compounds). Most of the recovery results are within the range 70-120 %.

Method M01 has the lowest percentage of recoveries in the range 70-120 % with 69 % of compounds. M02 and M03 have obtained 88 and 91% respectively and method M04 is the one that has obtained the best percentage of recoveries between 70-120 % with 93 % of the compounds (**Table 1**).

Table 1. Percentage of target analytes with recoveries in the 70-120 % range.

Extraction methods	***M01***	***M02***	***M03***	***M04***
% Of compounds with recovery (70-120 %)	69	88	91	93

Figure 1 summarises the recovery results obtained by the extraction method M04.

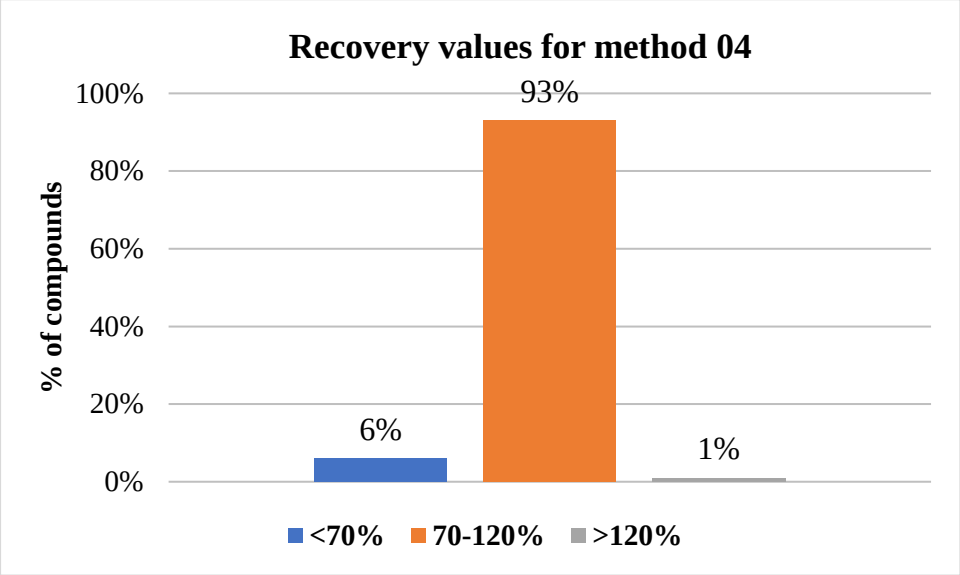

Figure 1. Percentage of compounds in each recovery range for method 04

6.1.2. Matrix effects:

Matrix effects were assessed by comparison of the slopes of four-point matrix-matched calibration curves with the slopes of the calibration curves in solvent. For values (in absolute terms) between 0 and 20 %, the matrix effect is considered low; between 20 % and 50 % there is a moderate matrix effect, and for compounds with a value over 50 % matrix effect is classified as strong. These values of matrix effects are summarized in **Appendix I**, **Table A2**, and they are also represented in **Figure 2**.

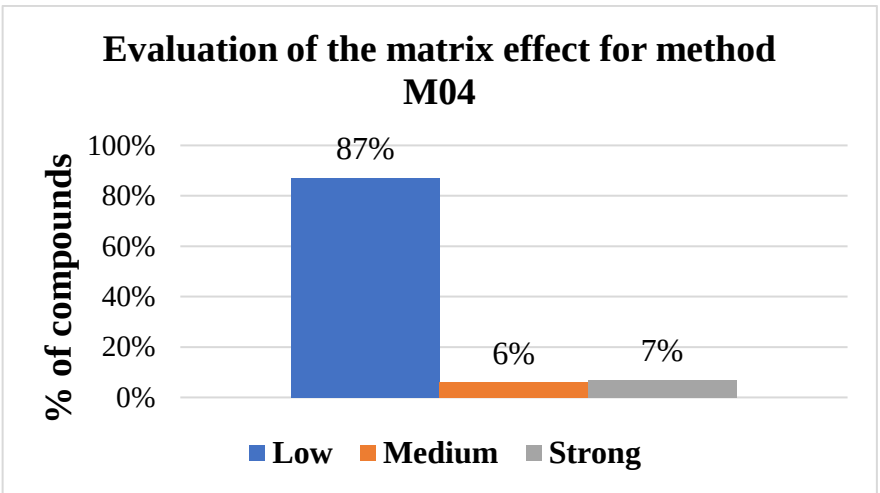

Figure 2. Matrix effects for the target analytes using the method M04.

6.2. Comparison of extraction methods:

The extraction method M04 has been compared with the extraction methods M01, M02 and M03, in terms of method precision (as recovery (%) of the compounds included in the scope), as well as matrix effects. The results for all pesticide residues included in the LC-MS/MS range are summarised in **Figure 3** in terms of recovery. The percentage of compounds with recovery values between 70 and 120 % is higher with the addition of SDS as represented by M04. Method M01 has the lowest percentage of compounds with recovery values between 70-120 %. Improvement is observed by extending the agitation time, enhancing percentage of compounds with good recoveries between 70-120 %. Without hydrating the sample (M03), due to protein emulsion is not formed, increase the percentage of compounds with good recoveries.

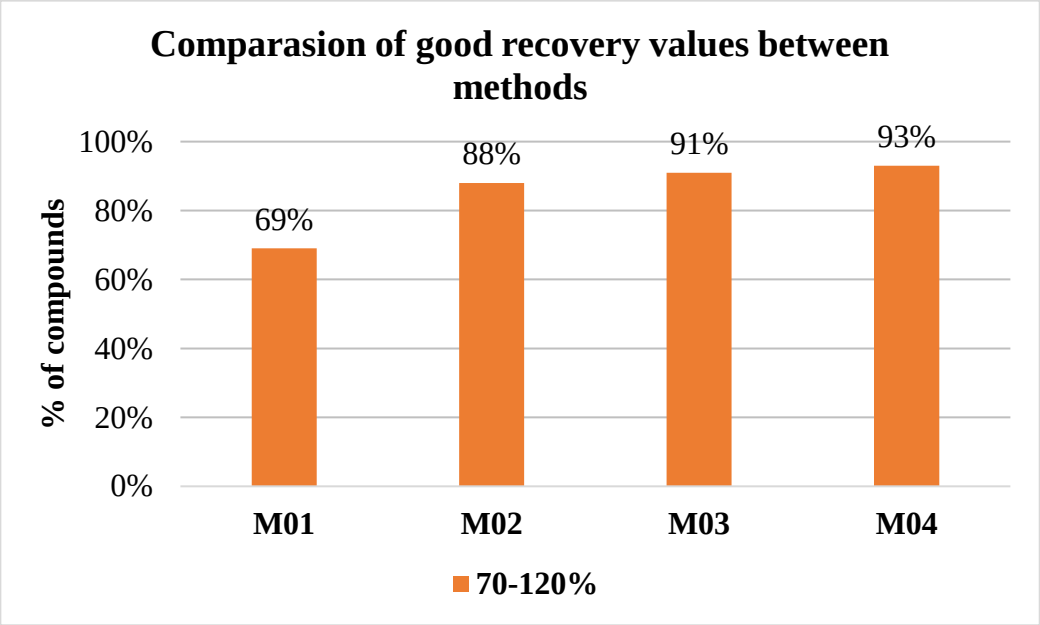

Figure 3. Recovery values for different extraction methods.

Some compounds could not be recovered in the extraction process. Thus, they were not detected, in **Table A2,** they are shown as ND (not detected). Both M01 and M02 were not able to recover 6 compounds out of 258, and M03 failed to detect 7 compounds out of 258. However, M04 successfully detected 257 out of 258 compounds.

The results for all pesticide residues included in the LC-MS/MS range are summarised in **Figure 4** in terms of matrix effect. Methods that present compounds with less matrix effects are M04 and M01, which have the highest percentage of

compounds with a low or negligible matrix effect (87%). All four methods have a similar percentage of compounds with a strong matrix effect from 5 to 8 %.

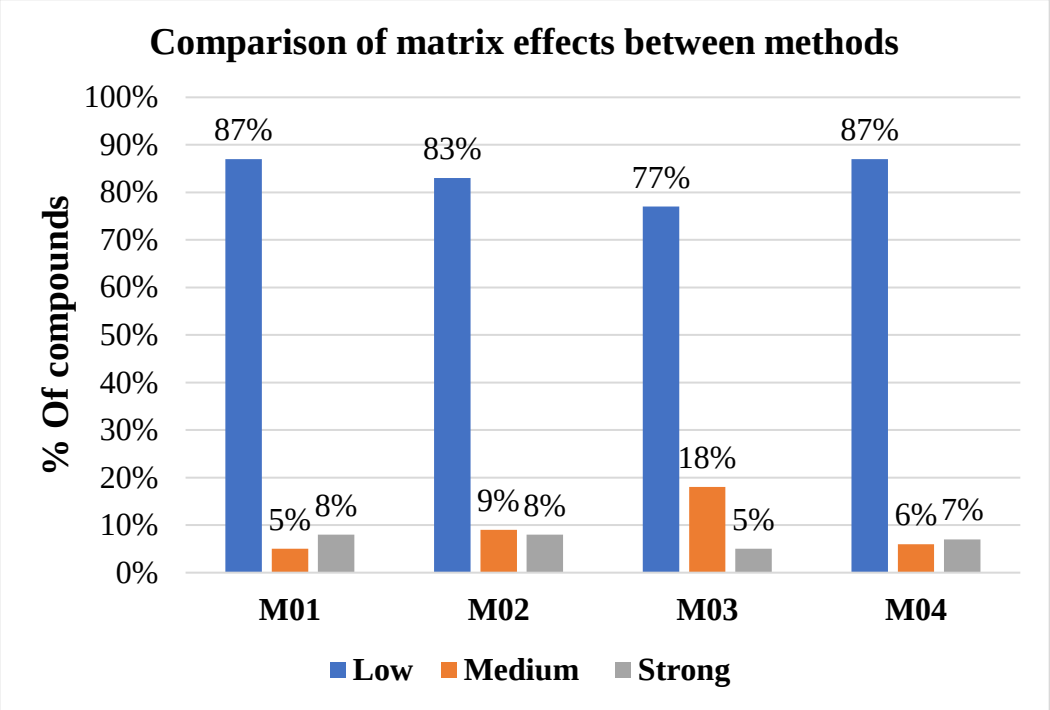

Figure 4. Matrix effects for different extraction methods

Figure 5 shows the appearance of the extracts after the freezing-out step. As can be seen in the figure, M04 obtains a higher precipitate because the SDS denatures the proteins present in the matrix and causes them to precipitate.

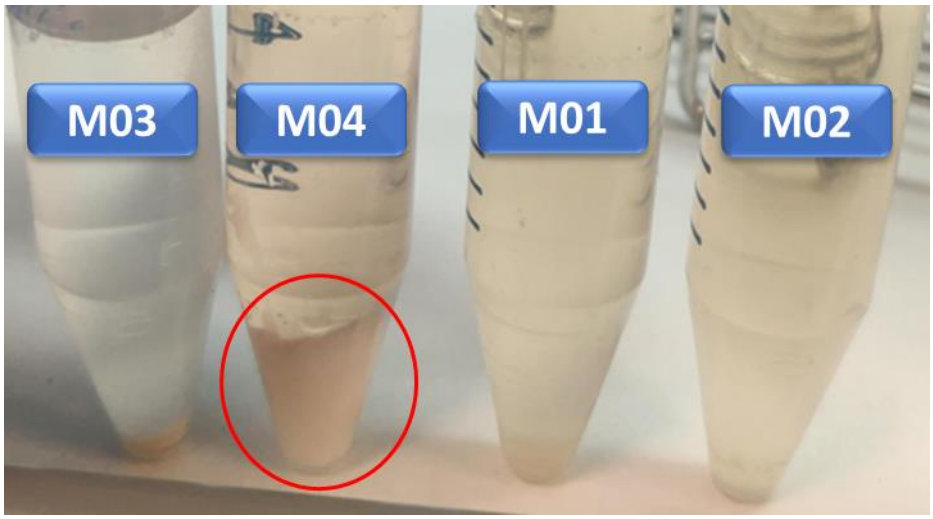

Figure 5. Extracts of white bean samples with the method M03, M04, M01 and M02, which includes a freezing-out step.

6.3. Conclusion

Method approaches with longer agitation, no water addition and SDS addition improve recoveries for 16-23 % of the pesticides. Best results are obtained for methods with no water addition and SDS addition, with more than 90 % of pesticides with good recoveries, between 70-120 %. The modified QuEChERS citrate method by adding SDS in the hydration step performed better than any method with 87% of the target analytes within the 70-120 % recovery range and up to 87% of them showing a low or negligible matrix effect.

8. References

[1] COMMISSION IMPLEMENTING REGULATION (EU) 2021/601 of 13 April 2021 concerning a coordinated multiannual control programme of the Union for 2022, 2023 and 2024 to ensure compliance with maximum residue levels of pesticides and to assess the consumer exposure to pesticide residues in and on food of plant and animal origin.

[2] Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin (SANCO/12745/2013).

[1] ANALYTICAL QUALITY CONTROL AND METHOD VALIDATION PROCEDURES FOR PESTICIDE RESIDUES ANALYSIS IN FOOD AND FEED. Document Nº SANTE/11312/2021. EUROPEAN UNION REFERENCE LABORATORIES.

APPENDIX I: MASS TRANSITIONS AND VALIDATION RESULTS

Table A1. Detection parameters for the selected compounds analysed by LC-MS/MS.

Compound	Retention Time (min)	RT Window (min)	Polarity	Precursor (m/z)	Product (m/z)	Collision Energy (V)	RF Lens (V)
Acephate	1.42	0.5	Positive	184.019	48.845	20.58	30
Acephate	1.42	0.5	Positive	184.019	142.863	10.23	30
Acetamiprid	2.74	0.5	Positive	223.074	55.786	16.52	45
Acetamiprid	2.74	0.5	Positive	223.074	125.702	21.18	45
Alachlor	6.30	2	Positive	270.125	162	20.35	33
Alachlor	6.30	2	Positive	270.125	238	10.23	33
Albendazole	4.79	0.5	Positive	266.095	190.845	32.86	50
Albendazole	4.79	0.5	Positive	266.095	233.929	19.67	50
Aldicarb	3.21	0.5	Positive	116.053	61.082	13.72	30
Aldicarb	3.21	0.5	Positive	116.053	88.97	9.51	30
Ametoctradin	7.43	0.5	Positive	276.218	176	37.71	70
Ametoctradin	7.43	0.5	Positive	276.218	176.97	28.99	70
Anilofos	6.91	0.5	Positive	368.03	124.845	31.27	45
Anilofos	6.91	0.5	Positive	368.03	198.762	14.21	45
Atrazine	4.38	0.5	Positive	216.101	103.917	28.35	50
Atrazine	4.38	0.5	Positive	216.101	173.97	17.35	50
Azinphos-ethyl	6.07	0.5	Positive	346.044	131.917	16.07	33
Azinphos-ethyl	6.07	0.5	Positive	346.044	260.887	10.23	33
Azinphos-methyl	5.02	0.5	Positive	318.013	131.804	14.3	36
Azinphos-methyl	5.02	0.5	Positive	318.013	167.016	11.3	36
Azoxystrobin	5.19	0.5	Positive	404.124	343.929	25.35	45
Azoxystrobin	5.19	0.5	Positive	404.124	371.929	14.74	45
BAC 10	5.79	0.5	Positive	276.268	91.054	27.96	50
BAC 10	5.79	0.5	Positive	276.268	184.292	19.45	50
BAC 8	4.29	0.5	Positive	248.237	91.054	26.82	50
BAC 8	4.29	0.5	Positive	248.237	156.208	18.4	50
Benalaxyl	6.88	0.5	Positive	326.175	148	21.75	40
Benalaxyl	6.88	0.5	Positive	326.175	293.929	10.23	40
Bendiocarb	3.70	0.5	Positive	224.091	108.774	17.47	33
Bendiocarb	3.70	0.5	Positive	224.091	166.857	10.23	33
Benzovindiflupyr	7.00	0.5	Positive	398.150	342.125	16.88	33
Benzovindiflupyr	7.00	0.5	Positive	398.150	378.054	13.26	33
Bitertanol	7.27	0.5	Positive	338.186	69.929	10.23	36
Bitertanol	7.27	0.5	Positive	338.186	269.012	10.23	36
Bixafen	6.76	0.5	Positive	416.122	396.054	13.47	33
Bixafen	6.76	0.5	Positive	416.22	267.899	23.28	33

Boscalid	5.52	0.5	Positive	343.039	270.94	31.76	90
Boscalid	5.52	0.5	Positive	343.039	306.845	19.86	90
Bromacil	3.69	0.5	Positive	261.023	187.762	28.54	40
Bromacil	3.69	0.5	Positive	261.023	204.762	14.47	40
Bromuconazole	6.30	1.5	Positive	375.961	69.917	20.8	60
Bromuconazole	6.30	1.5	Positive	375.961	158.899	28.16	60
Bupirimate	5.98	0.5	Positive	317.164	165.929	24.67	60
Bupirimate	5.98	0.5	Positive	317.164	237.012	19.89	60
Buprofezin	8.01	0.5	Positive	306.163	115.845	16.56	40
Buprofezin	8.01	0.5	Positive	306.163	200.857	12.54	40
Butoxycarboxim	1.90	0.5	Positive	223.074	62.845	10.99	30
Butoxycarboxim	1.90	0.5	Positive	223.074	105.845	11.48	30
Carbaryl	3.96	0.5	Positive	202.086	126.929	28.96	30
Carbaryl	3.96	0.5	Positive	202.086	144.929	10.23	30
Carbendazim	2.18	0.5	Positive	192.076	131.929	30.7	40
Carbendazim	2.18	0.5	Positive	192.076	159.929	18.15	40
Carbendazim-d3	2.20	0.5	Positive	195.095	160.042	18.02	52
Carbendazim-d3	2.20	0.5	Positive	195.095	132.0	30.78	52
Chlorantraniliprole	4.93	0.5	Positive	481.978	283.875	12.69	50
Chlorantraniliprole	4.93	0.5	Positive	481.978	450.815	16.82	50
Chlorbromuron	5.52	0.5	Positive	292.968	181.845	16.79	50
Chlorbromuron	5.52	0.5	Positive	292.968	203.69	19.71	50
Chlorfenvinphos	7.01	0.5	Positive	358.976	98.774	28.92	45
Chlorfenvinphos	7.01	0.5	Positive	358.976	154.899	12.88	45
Chlorfluazuron	9.11	0.5	Positive	539.97	157.97	19.06	60
Chlorfluazuron	9.11	0.5	Positive	539.97	382.833	20.08	60
Chloridazon	2.79	0.5	Positive	222.042	76.929	33.73	60
Chloridazon	2.79	0.5	Positive	222.042	103.845	23.16	60
Chlorotoluron	4.32	0.5	Positive	213.078	71.857	18.95	45
Chlorotoluron	4.32	0.5	Positive	213.078	139.845	25.09	45
Chloroxuron	6.01	0.5	Positive	291.089	71.917	20.77	50
Chloroxuron	6.01	0.5	Positive	291.089	163.929	17.24	50
Chlorpyriphos	8.51	0.5	Positive	349.933	197.762	19.4	50
Chlorpyriphos	8.51	0.5	Positive	349.933	321.679	12.05	50
Chromafenozide	6.04	0.5	Positive	395.232	174.982	16.96	36
Chromafenozide	6.04	0.5	Positive	395.232	339.149	7.4	36
Clofentezine	7.27	0.5	Positive	303.019	101.845	34.41	36
Clofentezine	7.27	0.5	Positive	303.019	137.845	14.97	36
Clomazone	4.97	0.5	Positive	240.078	88.899	46.28	40
Clomazone	4.97	0.5	Positive	240.078	124.845	21.11	40
Coumaphos	6.91	0.5	Positive	363.021	226.774	26.07	60
Coumaphos	6.91	0.5	Positive	363.021	306.762	17.85	60
cyantraniliprole	4.12	0.5	Positive	475.098	285.958	14.27	59

cyantraniliprole	4.12	0.5	Positive	475.098	444.00	18.23	59
Cyazofamid	6.33	0.5	Positive	325.052	107.774	14.28	40
Cyazofamid	6.33	0.5	Positive	325.052	260.929	10.23	40
Cyflufenamid	7.24	0.5	Positive	413.128	240.917	23.19	50
Cyflufenamid	7.24	0.5	Positive	413.128	294.899	15.5	50
Cyflumetofen	7.90	0.5	Positive	465.250	173.042	24.29	66
Cyflumetofen	7.90	0.5	Positive	465.250	173.042	13.72	66
Cymoxanil	2.84	0.5	Positive	199.082	127.970	10.23	40
Cymoxanil	2.84	0.5	Positive	199.082	110.917	10.23	40
Cyproconazole	5.83	0.5	Positive	292.121	124.899	29.87	30
Cyproconazole	5.83	0.5	Positive	292.121	88.970	52.57	30
Cyprodinil	6.24	0.5	Positive	226.133	92.857	34.98	60
Cyprodinil	6.24	0.5	Positive	226.133	107.857	26.68	60
Cyromazine	0.01	0.5	Positive	167.103	67.988	34.57	40
Cyromazine	0.01	0.5	Positive	167.103	125.0	19.14	40
DEET	4.49	0.5	Positive	192.138	90.929	29.64	45
DEET	4.49	0.5	Positive	192.138	118.845	17.89	45
Demeton-S-methyl	3.77	0.5	Positive	231.027	60.917	30.36	27
Demeton-S-methyl	3.77	0.5	Positive	231.027	88.899	10.23	27
Demeton-S-methylsulfone	2.19	0.5	Positive	263.017	124.845	23.5	50
Demeton-S-methylsulfone	2.19	0.5	Positive	263.017	168.845	16.37	50
Demeton-S-methylsulfoxide	2.10	0.5	Positive	247	109.042	27.07	40
Demeton-S-methylsulfoxide	2.10	0.5	Positive	247	169.054	14.27	40
Diazinon	6.93	0.5	Positive	305.11	153.208	21.26	50
Diazinon	6.93	0.5	Positive	305.11	169.137	21.09	50
Dichlorvos	3.58	0.5	Positive	220.938	108.774	17.85	50
Dichlorvos	3.58	0.5	Positive	220.938	144.8	14.02	50
Dichlorvos-d6	3.65	0.5	Positive	226.990	115.095	17.85	86
Dichlorvos-d6	3.65	0.5	Positive	226.990	83.11	26.90	86
Dicrotophos	2.41	0.5	Positive	238.08	112.125	12.71	36
Dicrotophos	2.41	0.5	Positive	238.08	126.97	18.35	36
Diethofencarb	5.18	0.5	Positive	268.154	179.929	18.26	33
Diethofencarb	5.18	0.5	Positive	268.154	225.929	10.23	33
Difenoconazole	7.51	0.5	Positive	406.071	250.845	25.7	60
Difenoconazole	7.51	0.5	Positive	406.071	336.845	17.62	60
Difenoxuron	4.66	0.5	Positive	287	71.929	20.35	50
Difenoxuron	4.66	0.5	Positive	287	122.899	19.89	50
Diflubenzuron	6.60	0.5	Positive	311.039	140.917	32.1	36
Diflubenzuron	6.60	0.5	Positive	311.039	157.97	14.06	36
Dimethoate	2.77	0.5	Positive	230.006	124.845	21.75	33
Dimethoate	2.77	0.5	Positive	230.006	198.762	10.23	33
Dimethomorph	5.50	1.5	Positive	388.131	164.929	31.61	60
Dimethomorph	5.50	1.5	Positive	388.131	300.845	20.73	60

Dimethylvinphos	5.93	0.5	Positive	331	127.042	13.51	50
Dimethylvinphos	5.93	0.5	Positive	331	170.042	36.34	50
Diniconazole	7.46	0.5	Positive	326.082	69.929	25.47	70
Diniconazole	7.46	0.5	Positive	326.082	158.887	30.47	70
Dinotefuran	1.88	0.5	Positive	203.100	157.125	7.27	30
Dinotefuran	1.88	0.5	Positive	203.100	129.042	11.44	30
Diuron	4.70	0.5	Positive	233.024	71.917	18.87	50
Diuron	4.70	0.5	Positive	233.024	159.815	26.95	50
DMF	3.41	0.5	Positive	150.091	106.125	33.14	55
DMF	3.41	0.5	Positive	150.091	107.125	21.43	55
DMPF	2.33	0.5	Positive	163.173	122.054	16.50	30
DMPF	2.33	0.5	Positive	163.173	107.125	24.25	30
Dodine	7.19	0.5	Positive	228.264	56.929	23.99	60
Dodine	7.19	0.5	Positive	228.264	59.929	23.8	60
Edifenphos	6.76	0.5	Positive	311.032	108.845	32.18	45
Edifenphos	6.76	0.5	Positive	311.032	282.845	13.79	45
Emamectin B1a	8.36	0.5	Positive	886.618	158.125	34.15	98
Emamectin B1a	8.36	0.5	Positive	886.618	82.12	40.80	98
Epoxiconazole	6.25	0.5	Positive	330.08	100.917	43.32	50
Epoxiconazole	6.25	0.5	Positive	330.08	120.899	21.3	50
Ethiofencarb	4.14	0.5	Positive	226.089	106.845	15.72	30
Ethiofencarb	4.14	0.5	Positive	226.089	164	10.23	30
Ethion	8.34	0.5	Positive	384.994	142.762	25.13	40
Ethion	8.34	0.5	Positive	384.994	198.774	10.23	40
Ethiprole	5.40	0.5	Positive	350.598	350.58	20.42	94
Ethiprole	5.40	0.5	Positive	396.990	254.98	34.70	94
Ethirimol	3.15	0.5	Positive	210.16	97.97	27.44	50
Ethirimol	3.15	0.5	Positive	210.16	140	22.55	50
Ethoprofos	6.15	0.5	Positive	243.063	130.905	20.35	36
Ethoprofos	6.15	0.5	Positive	243.063	172.958	14.63	36
Etoxazol	8.69	0.5	Positive	360.176	140.845	30.93	60
Etoxazol	8.69	0.5	Positive	360.176	303.929	18.23	60
Famoxadone	7.11	0.5	Positive	392.16	237.911	17.28	36
Famoxadone	7.11	0.5	Positive	392.16	330.911	10.23	36
Fenamidone	5.42	0.5	Positive	312.116	91.899	24.71	50
Fenamidone	5.42	0.5	Positive	312.116	235.929	15.08	50
Fenamiphos	6.51	0.5	Positive	304.113	216.845	23.27	50
Fenamiphos	6.51	0.5	Positive	304.113	233.917	17.13	50
Fenamiphos-sulfone	3.87	0.5	Positive	336.102	265.917	20.12	60
Fenamiphos-sulfone	3.87	0.5	Positive	336.102	307.917	15.72	60
Fenamiphos-sulfoxide	3.71	0.5	Positive	320.107	232.845	25.05	50
Fenamiphos-sulfoxide	3.71	0.5	Positive	320.107	291.899	16.26	50
Fenarimol	6.14	0.5	Positive	331.039	188.845	47.19	70

Fenarimol	6.14	0.5	Positive	331.039	267.917	22.93	70
Fenazaquin	9.27	0.5	Positive	307.18	146.917	19.74	50
Fenazaquin	9.27	0.5	Positive	307.18	161.054	16.98	50
Fenbendazole	5.93	0.5	Positive	300.030	268.042	20.33	30
Fenbendazole	5.93	0.5	Positive	300.030	159.072	34.78	30
Fenbuconazole	6.46	0.5	Positive	337.121	88.899	55	60
Fenbuconazole	6.46	0.5	Positive	337.121	124.845	30.24	60
Fenhexamide	6.07	0.5	Positive	302.07	55.054	34.23	60
Fenhexamide	6.07	0.5	Positive	302.07	97.208	23.28	60
Fenobucarb	5.21	0.5	Positive	208.133	94.917	15.08	30
Fenobucarb	5.21	0.5	Positive	208.133	151.97	10.23	30
Fenoxycarb	6.60	0.5	Positive	302.138	87.97	18.98	40
Fenoxycarb	6.60	0.5	Positive	302.138	115.857	10.68	40
Fenpicoxamid	7.9	0.5	Positive	615.305	239.054	26.69	30
Fenpicoxamid	7.9	0.5	Positive	615.305	515.280	16.29	30
Fenpropidin	4.76	0.5	Positive	274.252	116.857	52.12	70
Fenpropidin	4.76	0.5	Positive	274.252	146.929	29.07	70
Fenpropimorph	4.95	0.5	Positive	304.263	130.054	25.43	70
Fenpropimorph	4.95	0.5	Positive	304.263	147.054	28.88	70
Fenpyrazamine	5.85	0.5	Positive	332.142	230.083	19.25	45
Fenpyrazamine	5.85	0.5	Positive	332.142	231.054	18.11	45
Fenpyroximate *E*	8.98	0.5	Positive	422.294	366.137	13.97	30
Fenpyroximate *E*	8.98	0.5	Positive	422.294	138.000	30.70	30
Fensulfothion	4.65	0.5	Positive	309.126	253.042	17.13	45
Fensulfothion	4.65	0.5	Positive	309.126	157.054	23.79	45
Fenthion	6.89	0.5	Positive	279.027	168.899	18.3	60
Fenthion	6.89	0.5	Positive	279.027	246.845	13.37	60
Fenthion-sulfone	4.07	0.5	Positive	311.017	124.917	20.05	70
Fenthion-sulfone	4.07	0.5	Positive	311.017	278.917	17.92	70
Fenthion-sulfoxide	3.86	0.5	Positive	295.022	263.845	16.75	60
Fenthion-sulfoxide	3.86	0.5	Positive	295.022	279.845	18.98	60
Fenuron	2.6	0.5	Positive	165.102	71.917	15.84	30
Fenuron	2.6	0.5	Positive	165.102	76.899	30.43	30
Fipronil	6.67	0.5	Negative	434.945	329.845	15.46	90
Fipronil	6.67	0.5	Negative	434.945	398.815	10.23	90
Flazasulfuron	5.02	0.5	Positive	408.058	139	39.12	50
Flazasulfuron	5.02	0.5	Positive	408.058	181.97	18.42	50
Flonicamid	2.17	0.5	Positive	230.053	173.917	18.34	50
Flonicamid	2.17	0.5	Positive	230.053	202.845	17.35	50
Florpyrauxifen-benzyl	7.46	0.5	Positive	439.130	91.125	21.85	68
Florpyrauxifen-benzyl	7.46	0.5	Positive	439.130	65.00	55.00	68
Fluacypirim	7.49	0.5	Positive	427.062	144.982	24.92	36
Fluacypirim	7.49	0.5	Positive	427.062	205.196	9.34	36

Fluazifop-P-butyl	8.12	0.5	Positive	384.280	282.155	19.79	74
Fluazifop-P-butyl	8.12	0.5	Positive	384.280	328.1	15.49	74
Flubendiamide	6.79	0.5	Positive	683.03	273.833	31.38	50
Flubendiamide	6.79	0.5	Positive	683.03	407.887	10.23	50
Fludioxonil	5.57	0.5	Negative	247.032	168.857	32.78	100
Fludioxonil	5.57	0.5	Negative	247.032	179.857	28.69	100
Flufenacet	6.19	0.5	Positive	364.073	151.97	18.87	40
Flufenacet	6.19	0.5	Positive	364.073	193.929	10.23	40
Flufenoxuron	8.84	0.5	Positive	489.043	140.917	42.83	60
Flufenoxuron	8.84	0.5	Positive	489.043	157.97	18.95	60
Fluometuron	4.24	0.5	Positive	233.089	71.917	19.1	45
Fluometuron	4.24	0.5	Positive	233.089	159.899	27.44	45
Fluopicolide	5.63	0.5	Positive	382.97	109.042	55	60
Fluopicolide	5.63	0.5	Positive	382.97	173.042	23.87	60
Fluopyram	6.03	0.5	Positive	397.053	172.845	28.69	60
Fluopyram	6.03	0.5	Positive	397.053	207.845	21.9	60
Fluquinconazole	6.03	0.5	Positive	376.016	307.042	26.23	60
Fluquinconazole	6.03	0.5	Positive	376.016	349.042	19.7	60
Flusilazole	6.56	0.5	Positive	316.107	164.929	27.25	70
Flusilazole	6.56	0.5	Positive	316.107	246.929	18.34	70
Flutriafol	4.49	0.5	Positive	302.109	94.917	47.91	50
Flutriafol	4.49	0.5	Positive	302.109	122.845	28.01	50
Fluxapyroxad	5.74	0.5	Positive	382.097	341.929	21.18	50
Fluxapyroxad	5.74	0.5	Positive	382.097	362	14.4	50
Formetanate-hydrochloride	1.62	0.5	Positive	222.123	92.845	35.44	50
Formetanate-hydrochloride	1.62	0.5	Positive	222.123	165	15.57	50
Fosthiazate	4.17	0.5	Positive	284.053	103.845	21.71	33
Fosthiazate	4.17	0.5	Positive	284.053	227.845	10.23	33
Haloxyfop	6.74	0.5	Positive	362.04	287.845	27.25	60
Haloxyfop	6.74	0.5	Positive	362.04	315.649	18.53	60
Hexaconazole	7.16	0.5	Positive	314.082	69.899	20.77	50
Hexaconazole	7.16	0.5	Positive	314.082	158.845	31.15	50
Hexythiazox	8.48	0.5	Positive	353.108	168.042	24.18	45
Hexythiazox	8.48	0.5	Positive	353.108	227.988	14.66	45
Imazalil	4.17	0.5	Positive	297.055	158.958	23.04	60
Imazalil	4.17	0.5	Positive	297.055	200.863	17.77	60
Imidacloprid	2.53	0.5	Positive	256.059	175.071	18.15	45
Imidacloprid	2.53	0.5	Positive	256.059	209.006	15.72	45
Indoxacarb	7.62	0.5	Positive	528.077	149.97	24.14	60
Indoxacarb	7.62	0.5	Positive	528.077	248.97	16.71	60
Ioxynil	4.84	0.5	Negative	369.823	126.958	33	100
Ioxynil	4.84	0.5	Negative	369.823	214.958	32	100
Iprovalicarb	5.99	0.5	Positive	321.217	119.018	19.48	36

Iprovalicarb	5.99	0.5	Positive	321.217	203.071	10.23	36
Isocarbophos	4.60	0.5	Positive	230.917	230.917	15.35	30
Isocarbophos	4.60	0.5	Positive	307.107	272.9	10.23	30
Isofenphos-methyl	6.73	0.5	Positive	332.107	120.899	33.96	45
Isofenphos-methyl	6.73	0.5	Positive	332.107	230.917	14.44	45
Isofetamid	6.11	0.5	Positive	360.163	124.988	27.28	39
Isofetamid	6.11	0.5	Positive	360.163	210.1	8.41	39
Isoprocarb	4.45	0.5	Positive	194.117	94.97	14.63	30
Isoprocarb	4.45	0.5	Positive	194.117	137.071	10.23	30
Isoprothiolane	5.61	0.5	Positive	291.071	188.887	20.99	33
Isoprothiolane	5.61	0.5	Positive	291.071	230.97	10.23	33
Isoproturon	4.57	0.5	Positive	207.149	71.988	18.42	40
Isoproturon	4.57	0.5	Positive	207.149	165.071	14.09	40
Isoxaflutole	4.61	0.5	Positive	360.051	219.845	38.62	50
Isoxaflutole	4.61	0.5	Positive	360.051	250.917	15.91	50
Kresoxim-methyl	6.68	0.5	Positive	314.138	222.071	10.23	30
Kresoxim-methyl	6.68	0.5	Positive	314.138	267.071	10.23	30
Lenacil	4.51	0.5	Positive	235.144	135.988	31.72	36
Lenacil	4.51	0.5	Positive	235.144	153	15.31	36
Linuron	5.31	0.5	Positive	249.019	159.976	17.58	45
Linuron	5.31	0.5	Positive	249.019	181.988	15.46	45
Lufenuron	8.38	0.5	Negative	508.971	326.042	18.65	80
Lufenuron	8.38	0.5	Negative	508.971	339.113	11.32	80
Malathion	7.38	0.5	Positive	331.043	124.845	28.54	30
Malathion	7.38	0.5	Positive	331.043	126.899	11.71	30
Malathion-d10	5.60	0.5	Positive	341.106	132.042	12.46	38
Malathion-d10	5.60	0.5	Positive	341.106	100.0	23.28	38
Mandipropamid	5.56	0.5	Positive	412.131	328.018	14.28	45
Mandipropamid	5.56	0.5	Positive	412.131	356.018	10.23	45
Matrine	1.50	0.5	Positive	249.321	150.1	31.62	74
Matrine	1.50	0.5	Positive	249.321	247.2	25.35	74
Mebendazole	4.38	0.5	Positive	296.190	264.125	20.00	30
Mebendazole	4.38	0.5	Positive	398.088	182.125	30.32	30
Mefentrifluconazole	7.22	0.5	Positive	398.088	70.071	21.68	68
Mefentrifluconazole	7.22	0.5	Positive	398.088	182.1	30.32	68
Mepanipyrim	5.95	0.5	Positive	224.118	77	37.79	50
Mepanipyrim	5.95	0.5	Positive	224.118	105.929	26	50
Metalaxyl	4.54	0.5	Positive	280.154	220.054	14.4	45
Metalaxyl	4.54	0.5	Positive	280.154	248.054	10.23	45
Metamitron	2.72	0.5	Positive	203.092	103.917	23.34	50
Metamitron	2.72	0.5	Positive	203.092	175	17.09	50
Metconazole	7.20	0.5	Positive	320.152	69.97	24.03	50
Metconazole	7.20	0.5	Positive	320.152	124.97	38.85	50

Methamidophos	0.54	1.5	Positive	142.008	93.917	14.51	36
Methamidophos	0.54	1.5	Positive	142.008	124.917	14.4	36
Methidathion	4.75	0.5	Positive	302.969	144.917	10.23	40
Methidathion	4.75	0.5	Positive	226.089	121.000	37.64	40
Methiocarb	5.41	0.5	Positive	226.089	106.917	37.64	30
Methiocarb	5.41	0.5	Positive	226.089	121	10.23	30
Methiocarb-sulfone	2.83	0.5	Positive	258.079	122.071	18.98	45
Methiocarb-sulfone	2.83	0.5	Positive	258.079	200.97	10.23	45
Methiocarb-sulfoxide	2.60	0.5	Positive	242.084	122	29.03	45
Methiocarb-sulfoxide	2.60	0.5	Positive	242.084	184.97	13.68	45
Methomyl	2.12	0.5	Positive	163.053	87.97	10.23	27
Methomyl	2.12	0.5	Positive	163.053	105.917	10.23	27
Methoxyfenozide	5.76	0.5	Positive	369.217	149	17.28	30
Methoxyfenozide	5.76	0.5	Positive	369.217	312.982	10.23	30
Metobromuron	4.38	0.5	Positive	259.007	148	15.5	50
Metobromuron	4.38	0.5	Positive	259.007	169.887	18.91	50
Metolachlor	6.26	0.5	Positive	284.141	176.054	25.85	40
Metolachlor	6.26	0.5	Positive	284.141	252.054	15.19	40
Metolcarb	3.41	0.5	Positive	166.086	93.97	30.78	30
Metolcarb	3.41	0.5	Positive	166.086	108.929	10.23	30
Metrafenone	7.31	0.5	Positive	409.064	208.929	13.98	50
Metrafenone	7.31	0.5	Positive	409.064	226.845	20.46	50
Monocrotophos	2.31	0.5	Positive	224.068	126.899	15.8	36
Monocrotophos	2.31	0.5	Positive	224.068	192.845	10.23	36
Monolinuron	4.11	0.5	Positive	215.058	125.917	17.89	40
Monolinuron	4.11	0.5	Positive	215.058	148	14.78	40
Monuron	3.58	0.5	Positive	199.063	71.97	16.71	45
Monuron	3.58	0.5	Positive	199.063	125.97	26.11	45
Myclobutanil	5.88	0.5	Positive	289.121	89	54.73	50
Myclobutanil	5.88	0.5	Positive	289.121	124.899	32.59	50
Neburon	6.68	0.5	Positive	275.071	57	21.22	45
Neburon	6.68	0.5	Positive	275.071	88.054	16.56	45
Nitenpyram	2.00	0.5	Positive	271.095	189	14.06	45
Nitenpyram	2.00	0.5	Positive	271.095	225	11.97	45
Novaluron	7.86	0.5	Negative	491.005	304.982	14.97	80
Novaluron	7.86	0.5	Negative	491.005	470.97	12.5	80
Omethoate	1.68	0.5	Positive	214.029	124.917	22.36	36
Omethoate	1.68	0.5	Positive	214.029	182.887	11.36	36
Orthosulfamuron	4.64	0.5	Positive	425.124	199.179	11.28	30
Orthosulfamuron	4.64	0.5	Positive	341.045	229.958	14.81	30
Oxadiargyl	7.15	0.5	Positive	341.045	222.917	16.07	60
Oxadiargyl	7.15	0.5	Positive	341.045	229.958	14.81	60
Oxadixyl	3.31	0.5	Positive	279.133	132	31.15	36

Oxadixyl	3.31	0.5	Positive	279.133	219.054	10.23	36
Oxamyl	1.98	0.5	Positive	237.025	71.97	10.23	27
Oxamyl	1.98	0.5	Positive	237.025	89.97	10.23	27
Oxasulfuron	3.43	0.5	Positive	407	107.196	43.25	50
Oxasulfuron	3.43	0.5	Positive	407	150.196	18.44	50
Oxathiapiprolin	5.72	0.5	Positive	540.274	500.208	22.78	30
Oxathiapiprolin	5.72	0.5	Positive	316.075	191.196	20.84	30
Oxfendazole	3.35	0.5	Positive	316.075	159.071	33.81	77
Oxfendazole	3.35	0.5	Positive	316.075	191.196	20.84	77
Paclobutrazol	5.65	0.5	Positive	294.136	69.97	20.92	50
Paclobutrazol	5.65	0.5	Positive	294.136	124.899	37.37	50
Penconazole	6.81	0.5	Positive	284.071	122.917	49.28	50
Penconazole	6.81	0.5	Positive	284.071	158.917	29.6	50
Pencycuron	7.38	0.5	Positive	329.141	124.917	25.81	50
Pencycuron	7.38	0.5	Positive	329.141	218	16.1	50
Pendimethalin	8.49	0.5	Positive	282.144	211.970	10.23	30
Pendimethalin	8.49	0.5	Positive	318.200	141.083	29.35	30
Penflufen	6.68	0.5	Positive	318.2	141.083	29.35	50
Penflufen	6.68	0.5	Positive	318.2	234.125	15.19	50
Penthiopyrad	6.89	0.5	Positive	360.14	177.125	34.82	45
Penthiopyrad	6.89	0.5	Positive	360.14	276.125	14.6	45
Permethrin	9.70	0.75	Positive	408.112	183.125	19.79	43
Permethrin	9.70	0.75	Positive	408.112	355.125	8.71	43
Phenothrin	9.80	0.5	Positive	351.195	183.125	18.18	30
Phenothrin	9.80	0.5	Positive	321.037	79.000	39.61	30
Phenthoate	6.67	0.5	Positive	321.037	79	39.61	36
Phenthoate	6.67	0.5	Positive	321.037	246.917	10.23	36
Phosalone	7.25	0.5	Positive	367.994	110.917	37.52	50
Phosalone	7.25	0.5	Positive	367.994	181.887	15	50
Phosmet	5.02	0.5	Positive	318.001	77	51.32	36
Phosmet	5.02	0.5	Positive	318.001	159.97	10.23	36
Phoxim	7.13	0.5	Positive	299.061	77.125	29.33	36
Phoxim	7.13	0.5	Positive	299.061	129.113	10.23	36
Pirimicarb	3.26	0.5	Positive	239.15	71.97	21.45	40
Pirimicarb	3.26	0.5	Positive	239.15	181.982	16.1	40
Pirimiphos-methyl	7.08	0.5	Positive	306.103	107.929	30.55	50
Pirimiphos-methyl	7.08	0.5	Positive	306.103	164.054	22.47	50
Prochloraz	7.01	0.5	Positive	376.038	69.929	25.77	40
Prochloraz	7.01	0.5	Positive	376.038	307.815	11.71	40
Profenophos	7.89	0.5	Positive	372.942	302.72	18.68	50
Profenophos	7.89	0.5	Positive	372.942	344.833	13.34	50
Promecarb	5.57	0.5	Positive	208.133	108.929	16.48	30
Promecarb	5.57	0.5	Positive	208.133	151.054	10.23	30

Prometryn	5.30	0.5	Positive	242.143	157.97	23.65	60
Prometryn	5.30	0.5	Positive	242.143	199.929	18.76	60
Propamocarb	1.70	0.5	Positive	189.159	101.917	17.58	40
Propamocarb	1.70	0.5	Positive	189.159	144	13.41	40
Propaquizafop	8.10	0.5	Positive	444.132	99.929	18.76	60
Propaquizafop	8.10	0.5	Positive	444.132	371	16.29	60
Propazine	5.19	0.5	Positive	230.116	145.917	23.31	45
Propazine	5.19	0.5	Positive	230.116	187.899	17.85	45
Propiconazole	7.02	0.5	Positive	342.077	69	20.12	60
Propiconazole	7.02	0.5	Positive	342.077	158.917	29.14	60
Propoxur	3.64	0.5	Positive	210.112	110.917	14.28	30
Propoxur	3.64	0.5	Positive	210.112	168.054	10.23	30
Propyzamide	5.69	0.5	Positive	256.03	172.958	22.86	33
Propyzamide	5.69	0.5	Positive	256.03	190.042	14.14	33
Proquinazid	9.05	0.5	Positive	373.04	288.851	23.46	50
Proquinazid	9.05	0.5	Positive	373.04	330.774	14.47	50
Prosulfocarb	7.74	0.5	Positive	252.141	90.97	22.55	40
Prosulfocarb	7.74	0.5	Positive	252.141	128.054	12.65	40
Pymetrozine	1.62	0.5	Positive	218.103	77.929	38.4	50
Pymetrozine	1.62	0.5	Positive	218.103	104.917	20.24	50
Pyraclostrobin	7.13	0.5	Positive	388.105	162.982	23.72	40
Pyraclostrobin	7.13	0.5	Positive	388.105	193.899	12.5	40
Pyretrins I	8.87	0.5	Positive	329	143	16.14	36
Pyretrins I	8.87	0.5	Positive	329	161.042	10.23	36
Pyretrins II	7.54	0.5	Positive	373.000	161.054	10.23	30
Pyretrins II	7.54	0.5	Positive	365.144	147.054	24.41	30
Pyridaben	9.20	0.5	Positive	365.144	147.054	24.41	40
Pyridaben	9.20	0.5	Positive	365.144	308.988	12.09	40
Pyridalyl	10.26	0.5	Positive	489.975	108.887	27.21	70
Pyridalyl	10.26	0.5	Positive	489.975	182.917	17.35	70
Pyridaphenthion	5.87	0.5	Positive	341.071	188.97	21.41	60
Pyridaphenthion	5.87	0.5	Positive	341.071	204.899	21.03	60
Pyrifoenone	7.33	0.5	Positive	366.11	184.042	23.96	60
Pyrifoenone	7.33	0.5	Positive	366.11	209.125	24.46	60
Pyrimethanil	4.84	0.5	Positive	200.118	106.917	24.18	60
Pyrimethanil	4.84	0.5	Positive	200.118	183.042	24.03	60
Pyriproxyfen	8.34	0.5	Positive	322.143	95.988	15.61	45
Pyriproxyfen	8.34	0.5	Positive	322.143	227.071	14.51	45
Quinalphos	6.61	0.5	Positive	299.061	147	21.71	60
Quinalphos	6.61	0.5	Positive	299.061	162.97	21.11	60
Quinoclamine	3.47	0.5	Positive	208.015	76.97	36.35	45
Quinoclamine	3.47	0.5	Positive	208.015	104.917	24.97	45
Quinoxyphen	8.45	0.5	Positive	308.003	161.988	45.29	60

Quinoxyphen	8.45	0.5	Positive	308.003	196.833	31.87	60
Quizalofop-P-ethyl	7.91	0.5	Positive	373.094	271	25.54	60
Quizalofop-P-ethyl	7.91	0.5	Positive	373.094	298.899	18.98	60
Rotenone	6.38	0.5	Positive	395.148	191.917	23.27	60
Rotenone	6.38	0.5	Positive	395.148	212.929	22.28	60
Simazine	3.63	0.5	Positive	202.09	124.125	18.56	45
Simazine	3.63	0.5	Positive	202.09	132.054	19.53	45
Spinetoram J	7.50	0.5	Positive	748.499	97.946	42.38	83
Spinetoram J	7.50	0.5	Positive	748.499	142	28.8	83
Spinetoram L	7.93	0.5	Positive	760.499	97.946	42.38	83
Spinetoram L	7.93	0.5	Positive	760.499	142	28.8	83
Spinosyn A	7.06	0.5	Positive	732.46	97.94	42.19	94
Spinosyn A	7.06	0.5	Positive	732.46	141.982	29.49	94
Spinosyn D	7.46	0.5	Positive	746.483	97.94	42.19	94
Spinosyn D	7.46	0.5	Positive	746.483	141.982	29.49	94
Spirodiclofen	8.94	0.5	Positive	411.112	71	16.22	36
Spirodiclofen	8.94	0.5	Positive	411.112	312.917	10.68	36
Spiromesifen	8.64	0.5	Positive	371.221	255.054	23.69	33
Spiromesifen	8.64	0.5	Positive	371.221	273.054	10.23	33
Spirotetramat	6.04	0.5	Positive	374.196	302.054	16.94	50
Spirotetramat	6.04	0.5	Positive	374.196	330.065	15.5	50
Spiroxamine	5.31	0.5	Positive	298.274	99.929	30.81	45
Spiroxamine	5.31	0.5	Positive	298.274	144.054	20.58	45
Sulfoxaflor	2.86	0.5	Positive	278.056	153.97	28.39	33
Sulfoxaflor	2.86	0.5	Positive	278.056	173.97	10.23	33
Tau-Fluvalinate	9.55	0.5	Positive	503.084	181.196	25.77	30
Tau-Fluvalinate	9.55	0.5	Positive	308.152	124.899	37.14	30
Tebuconazole	6.88	0.5	Positive	308.152	69.97	22.93	50
Tebuconazole	6.88	0.5	Positive	308.152	124.899	37.14	50
Tebufenozide	6.58	0.5	Positive	353.222	132.97	19.4	30
Tebufenozide	6.58	0.5	Positive	353.222	296.982	10.23	30
Tebufenpyrad	8.04	0.5	Positive	334.168	116.917	35.78	70
Tebufenpyrad	8.04	0.5	Positive	334.168	144.97	27.14	70
Teflubenzuron_	8.40	0.5	Negative	379	196.042	21.26	45
Teflubenzuron	8.40	0.5	Negative	379	339.042	9.72	45
Terbuthylazine	5.37	0.5	Positive	230.116	103.845	32.18	45
Terbuthylazine	5.37	0.5	Positive	230.116	173.97	17.17	45
Terbuthylazine-desethyl	3.88	0.5	Positive	202.08	104.042	28.13	40
Terbuthylazine-desethyl	3.88	0.5	Positive	202.08	146.113	16.75	40
Tetraconazole	6.29	0.5	Positive	372.028	69.97	22.28	60
Tetraconazole	6.29	0.5	Positive	372.028	158.887	29.79	60
Tetramethrin	7.90	0.5	Positive	332.185	164.125	23.74	30
Tetramethrin	7.90	0.5	Positive	192.026	97.982	29.64	30

Thiabendazole	2.43	0.5	Positive	202.043	130.97	32.82	70
Thiabendazole	2.43	0.5	Positive	202.043	174.97	25.54	70
Thiacloprid	2.98	0.5	Positive	253.03	89.97	35.63	50
Thiacloprid	2.98	0.5	Positive	253.03	125.917	20.92	50
Thiamethoxam	2.17	0.5	Positive	292.026	210.970	11.36	30
Thiamethoxam	2.17	0.5	Positive	258.071	89.000	47.23	30
Thiobencarb	7.29	0.5	Positive	258.071	89	47.23	36
Thiobencarb	7.29	0.5	Positive	258.071	124.97	19.14	36
Tolfenpyrad	8.20	0.5	Positive	384.147	170.982	24.63	70
Tolfenpyrad	8.20	0.5	Positive	384.147	197.208	25.35	70
Triadimefon	5.77	0.5	Positive	294.1	196.899	15.35	50
Triadimefon	5.77	0.5	Positive	294.1	224.929	13.07	50
Triallate	8.56	0.5	Positive	304.009	86.113	16.29	60
Triallate	8.56	0.5	Positive	304.009	142.833	27.02	60
Triazophos	5.95	0.5	Positive	314.072	118.929	34	50
Triazophos	5.95	0.5	Positive	314.072	162	18.45	50
Trichlorfon	2.75	0.5	Positive	256.892	109.125	19.15	54
Trichlorfon	2.75	0.5	Positive	256.892	220.905	9.76	54
Tricyclazole	3.11	0.5	Positive	190.043	135.917	28.5	50
Tricyclazole	3.11	0.5	Positive	190.043	162.97	22.59	50
Trifloxystrobin	7.60	0.5	Positive	409.136	144.917	43.06	50
Trifloxystrobin	7.60	0.5	Positive	409.136	185.917	17.39	50
Triflumizole	7.70	0.5	Positive	346.092	73	16.37	33
Triflumizole	7.70	0.5	Positive	346.092	277.97	10.23	33
Triflumuron	7.28	0.5	Positive	359.04	138.917	30.47	45
Triflumuron	7.28	0.5	Positive	359.04	155.988	15.46	45
Triticonazole	6.17	0.5	Positive	318.136	69.917	18.3	45
Triticonazole	6.17	0.5	Positive	318.136	124.97	32.9	45
Tritosulfuron	5.02	0.5	Positive	446.035	194.917	18.64	50
Tritosulfuron	5.02	0.5	Positive	446.035	220.9	18.72	50
Valifenalate	5.87	0.5	Positive	399.256	155.000	33.52	30
Valifenalate	5.87	0.5	Positive	180.101	94.929	20.01	30
XMC	4.15	1.5	Positive	180.101	94.929	20.01	30
XMC	4.15	1.5	Positive	180.101	122.929	12.16	30
Zoxamide	7.02	0.5	Positive	336.031	158.917	39	50
Zoxamide	7.02	0.5	Positive	336.031	186.815	22.17	50

Table A2. Accuracy data (as recovery values (%)), precision data (as repeatability RSD_r, n=5) at 0.010 mg/kg and matrix effects for dry white beans with different extraction methods (ND= No detected).

No.	Compound	M01		M02		M03		M04	
		Recovery % (RSD %)	Matrix Effects	Recovery % (RSD %)	Matrix Effects	Recovery % (RSD %)	Matrix Effects	Recovery % (RSD %)	Matrix Effects
1	Acephate	87(2)	1	85(2)	0	90(4)	1	84(2)	3
2	Acetamiprid	107(4)	2	101(2)	-1	100(1)	0	98(5)	3
3	Alachlor	115(0)	1	103(4)	-2	101(5)	-7	110(4)	-3
4	Albendazole	107(2)	-2	97(1)	-1	101(4)	-3	92(3)	-3
5	Aldicarb_116	103(1)	5	99(1)	4	103(2)	1	97(4)	1
6	Ametoctradin	108(3)	-2	100(3)	-1	99(1)	-4	79(7)	-69
7	Anilofos	136(2)	-6	101(1)	-9	99(9)	-21	100(4)	-1
8	Atrazine	104(3)	0	102(0)	0	99(3)	-2	100(3)	0
9	Azinphos-ethyl	107(5)	-11	113(8)	-12	88(8)	-26	104(2)	-5
10	Azinphos-methyl	120(5)	-4	106(5)	1	112(3)	-4	98(3)	-2
11	Azoxystrobin	114(4)	-2	100(3)	-3	102(4)	-1	108(4)	-2
12	BAC-C10	111(3)	9	106(3)	8	86(5)	10	108(1)	-69
13	BAC-C8	109(0)	4	105(1)	4	82(4)	-3	91(5)	-93
14	Benalaxyl	118(5)	-2	100(1)	-3	98(5)	-8	97(8)	0
15	Bendiocarb	105(4)	3	98(1)	1	102(5)	1	99(5)	5
16	Benzovindiflupyr	116(4)	-8	92(1)	-8	90(6)	-18	97(13)	1
17	Bifenazate	90(21)	-3	62(18)	-5	96(3)	-9	105(7)	-8
18	Bitertanol	137(11)	-9	105(10)	-4	109(1)	2	87(2)	2
19	Bixafen	118(1)	-5	101(1)	-5	98(4)	-10	99(5)	2
20	Boscalid	112(4)	1	105(2)	0	101(8)	-5	98(6)	1
21	Bromacil	97(3)	0	101(2)	-1	94(3)	-2	99(4)	2
22	Bromuconazole	116(6)	-1	90(3)	-5	100(5)	-6	92(3)	1
23	Bupirimate	120(2)	2	105(2)	4	111(2)	-1	97(4)	2
24	Buprofezin	138(3)	-1	116(2)	-3	120(3)	-5	94(3)	1
25	Butoxycarboxim	101(6)	-3	91(4)	-5	105(4)	-2	94(5)	0
26	Carbaryl	104(4)	3	99(3)	-2	102(3)	0	100(7)	3
27	Carbendazim	97(1)	4	90(0)	-2	92(2)	0	90(3)	0
28	Carbendazim-d3	94(1)	1	91(2)	-1	92(1)	0	90(4)	-2
29	Chlorantraniliprole	106(3)	2	103(6)	-1	95(5)	-5	100(5)	-2
30	Chlorbromuron	110(2)	0	102(4)	-4	96(8)	-3	90(2)	1
31	Chlorfenvinphos	115(2)	-3	109(4)	-3	97(4)	-7	102(4)	-1
32	Chlorfluazuron	ND	ND	ND	ND	ND	ND	123(16)	-19
33	Chloridazon	104(2)	-1	101(1)	-3	103(2)	-5	98(2)	-3
34	Chlorotoluron	102(3)	3	99(2)	-2	102(4)	-4	99(3)	2
35	Chloroxuron	114(5)	-3	94(1)	-2	103(3)	-8	101(6)	-1
36	Chlorpyrifos	>140 (9)	-55	129(14)	-55	100(16)	-54	119(18)	-18

37	Chromafenozide	113(3)	2	97(4)	0	102(11)	-2	104(7)	3
38	Clofentezine	>140 (2)	-41	92(9)	-20	99(4)	-44	119(13)	2
39	Clomazone	109(1)	0	99(1)	2	100(2)	-2	101(5)	0
40	Coumaphos	>140 (2)	-12	101(10)	-18	89(7)	-26	99(12)	-2
41	Cyantraniliprole	113(3)	-112	105(2)	-99	98(7)	-102	99(6)	-100
42	Cyazofamid	>140 (5)	-3	98(4)	-8	102(5)	-18	104(3)	3
43	Cyflufenamid	>140 (2)	-10	107(7)	-20	85(10)	-29	132(5)	-1
44	Cyflumetofen	>140 (18)	-240	117(24)	-85	97(10)	-433	113(12)	-199
45	Cymoxanil	99(1)	0	98(1)	0	99(2)	-1	107(2)	2
46	Cyproconazole	112(1)	-4	105(5)	-3	95(3)	-5	106(9)	-2
47	Cyprodinil	127(5)	1	101(2)	-2	118(1)	-6	91(2)	-5
48	Cyromazine	37(4)	1	47(10)	0	57(2)	-1	56(3)	6
49	DEET	105(1)	0	102(2)	-1	101(4)	-3	99(3)	1
50	Demeton-S-methyl	104(7)	4	96(2)	-2	104(5)	0	100(5)	1
51	Demeton-S-methylsulfone	106(4)	0	99(2)	-3	102(4)	-4	100(4)	0
52	Demeton-S-methylsulfoxide	97(3)	2	93(3)	0	94(2)	-2	92(4)	2
53	Diazinon	120(2)	-10	98(1)	-11	90(5)	-17	94(5)	-5
54	Dichlorvos	74(16)	6	66(16)	3	103(11)	-1	90(10)	7
55	Dichlorvos-d6	69(18)	3	54(16)	1	97(4)	2	90(4)	10
56	Dicrotophos	102(3)	1	101(2)	1	100(1)	-1	96(3)	3
57	Diethofencarb	111(5)	2	96(1)	3	101(1)	1	104(3)	1
58	Difenoconazole	131(2)	-11	96(5)	-11	101(6)	-19	102(9)	-42
59	Difenoxuron	107(4)	1	91(2)	-1	103(5)	-2	97(5)	1
60	Diflubenzuron	120(4)	-5	101(5)	-6	102(4)	-17	102(5)	-1
61	Dimethoate	113(2)	2	111(3)	-2	108(2)	-2	103(3)	1
62	Dimethomorph	111(3)	2	98(1)	3	100(3)	-4	99(6)	4
63	Dimethylvinphos	108(4)	4	100(2)	4	101(7)	-3	99(2)	2
64	Diniconazole	111(1)	-6	100(5)	-8	94(2)	-11	96(6)	-45
65	Dinotefuran	99(5)	-2	91(0)	-2	103(1)	-4	95(3)	-2
66	Diuron	107(3)	4	99(3)	0	101(6)	-1	99(5)	2
67	DMF	99(2)	49	99(1)	6	97(2)	-3	103(5)	-11
68	DMPF	89(2)	54	87(1)	12	73(1)	-4	90(3)	11
69	Dodine	67(4)	-1	105(7)	-5	20(12)	6	77(22)	-15
70	Edifenphos	120(2)	-3	97(3)	-4	100(4)	-9	98(5)	0
71	Emamectin B1a	110(16)	26	105(7)	25	95(8)	23	108(5)	26
72	Epoxiconazole	116(0)	-3	110(5)	-2	103(2)	-4	104(4)	48
73	Ethiofencarb	104(3)	0	103(5)	1	98(4)	-1	96(4)	2
74	Ethion	>140 (1)	-56	132(24)	-58	85(12)	-53	134(16)	-20
75	Ethiprole	111(4)	0	104(1)	-3	99(3)	-3	103(4)	3
76	Ethirimol	89(4)	6	82(3)	0	79(4)	-1	90(1)	3
77	Ethoprop (Ethoprofos)	111(6)	2	99(3)	0	103(2)	-2	100(5)	0
78	Etoxazol	>140 (9)	-42	125(16)	-54	98(9)	-38	76(15)	-73

79	Famoxadone	>140 (6)	-34	115(4)	-26	88(7)	-33	101(7)	-6
80	Fenamidone	116(3)	-1	98(2)	-1	102(2)	-3	105(5)	4
81	Fenamiphos	107(2)	-3	102(3)	-3	100(4)	-7	100(4)	-1
82	Fenamiphos-sulfone	104(3)	0	97(3)	0	97(3)	-4	103(4)	0
83	Fenamiphos-sulfoxide	108(1)	5	98(2)	3	100(4)	0	98(3)	7
84	Fenarimol	120(4)	-1	108(6)	-7	111(4)	-10	87(3)	0
85	Fenazaquin	>140 (2)	-45	114(14)	-48	99(11)	-44	82(8)	-27
86	Fenbendazole	109(1)	-6	104(1)	-8	103(6)	-6	88(5)	-1
87	Fenbuconazole	112(1)	-9	106(1)	-10	104(7)	-8	95(5)	-2
88	Fenhexamid	104(4)	-9	90(2)	-8	56(5)	-12	87(7)	-7
89	Fenobucarb	107(1)	4	99(1)	4	100(3)	1	105(1)	4
90	Fenoxycarb	120(4)	-8	100(3)	-7	95(6)	-19	99(6)	-1
91	Fenpicoxamid	>140 (5)	-9	110(5)	-14	107(8)	-29	80(6)	0
92	Fenpropidin	111(1)	2	100(2)	-1	90(3)	-1	90(8)	-100
93	Fenpropimorph	104(2)	1	100(2)	1	94(4)	-3	90(9)	-98
94	Fenpyrazamine	117(1)	-1	95(1)	-1	104(5)	-4	105(3)	3
95	Fenpyroximate_E	>140 (7)	-52	112(22)	-54	97(13)	-49	118(13)	-4
96	Fensulfothion	109(4)	2	100(2)	1	104(4)	0	103(5)	3
97	Fenthion	>140 (7)	-13	84(9)	-17	87(7)	-26	99(19)	-3
98	Fenthion-sulfone	111(3)	0	96(0)	0	103(2)	-3	98(1)	1
99	Fenthion-sulfoxide	109(2)	3	102(3)	1	105(1)	-1	100(5)	3
100	Fenuron	102(2)	1	103(3)	-3	101(2)	-3	99(5)	-4
101	Fipronil	120(5)	-3	100(4)	-7	98(3)	-11	103(1)	-1
102	Flazasulfuron	69(3)	-10	57(2)	-13	29(5)	-18	75(4)	-12
103	Flonicamid	95(8)	-2	102(7)	5	91(6)	-2	100(8)	2
104	Florpyrauxifen-benzyl	>140 (2)	-8	105(5)	-7	95(14)	-16	103(7)	-6
105	Fluacrypyrim	>140 (6)	-8	104(4)	-11	101(8)	-20	115(10)	-72
106	Fluazifop-P-butyl	>140 (10)	-71	111(10)	-102	92(13)	-76	114(14)	-52
107	Flubendiamide	109(11)	-13	90(19)	-11	81(28)	-19	94(8)	-10
108	Fludioxonil	108(3)	-8	103(4)	-12	88(9)	-10	112(5)	-5
109	Flufenacet	116(4)	0	104(2)	0	99(2)	-2	107(3)	1
110	Flufenoxuron	>140 (26)	-57	138(35)	-66	90(16)	-49	123(14)	53
111	Fluometuron	105(1)	1	103(3)	-1	98(4)	-2	105(3)	-1
112	Fluopicolide	118(1)	0	105(2)	-1	99(5)	-3	103(7)	1
113	Fluopyram	115(1)	-2	100(0)	-3	104(3)	-7	104(6)	-1
114	Flupyradifurone	107(1)	-100	101(5)	331	100(5)	-211	101(3)	-100
115	Fluquinconazole	116(2)	-4	105(10)	-1	102(4)	-5	106(6)	2
116	Flusilazole	113(1)	-5	102(3)	-6	101(3)	-9	95(2)	-4
117	Flutriafol	115(1)	-1	104(6)	-1	101(1)	1	102(7)	1
118	Fluxapyroxad	115(2)	3	103(1)	-1	105(1)	-3	104(5)	1
119	Formetanate-hydrochloride	89(2)	-1	86(3)	-5	84(5)	-8	93(5)	2
120	Fosthiazate	108(1)	1	100(2)	-2	103(2)	-2	101(4)	1

121	Haloxyfop	>140 (2)	-8	75(3)	-8	ND	ND	76(11)	-3
122	Hexaconazole	109(4)	-5	92(1)	-6	97(4)	-6	>140 (18)	-1
123	Hexythiazox	>140 (3)	-52	110(17)	-49	91(13)	-48	118(16)	-18
124	Imazalil	110(1)	-1	97(3)	-2	92(4)	-1	89(6)	-74
125	Imidacloprid	117(4)	12	102(5)	11	106(1)	-1	101(3)	12
126	Indoxacarb	>140 (13)	-16	105(8)	-28	87(7)	-29	106(8)	-4
127	Ioxynil	77(6)	-1	67(4)	0	42(5)	-3	67(1)	3
128	Iprovalicarb	111(5)	0	102(2)	-3	98(6)	-4	102(6)	-1
129	Isocarbophos	120(9)	-4	105(3)	-7	118(17)	-13	86(11)	-2
130	Isofenphos-methyl	131(9)	-8	99(4)	-8	98(11)	-15	101(8)	-3
131	Isofetamid	116(3)	9	102(2)	9	99(2)	-3	106(4)	-5
132	Isoprocarb	109(1)	0	105(2)	1	102(5)	0	104(3)	1
133	Isoprothiolane	120(2)	-2	99(2)	-4	102(5)	-8	104(4)	-2
134	Isoproturon	110(5)	2	100(3)	1	103(5)	-4	96(6)	2
135	Isopyrazam	126(1)	47	102(1)	-71	96(4)	-116	101(5)	-71
136	Isoxaflutole	124(8)	2	100(4)	4	108(5)	-2	105(2)	3
137	Kresoxim-methyl	126(6)	-4	102(7)	-7	96(3)	-16	104(6)	-1
138	Lenacil	100(2)	-1	100(1)	1	100(4)	-3	97(3)	0
139	Linuron	112(1)	0	103(3)	-1	102(0)	-4	103(4)	0
140	Lufenuron	ND	ND	ND	ND	ND	ND	132(25)	-30
141	Malathion	129(6)	0	98(4)	-2	108(2)	-10	104(4)	16
142	Malathion-d10	111(6)	0	106(2)	-3	100(3)	-4	102(6)	2
143	Mandipropamid	115(5)	4	105(3)	1	102(4)	-1	104(6)	2
144	Matrine	55(2)	3	48(5)	2	66(7)	-3	84(2)	6
145	Mebendazole	106(1)	0	98(2)	1	95(4)	-1	92(5)	1
146	Mefentrifluconazole	111(3)	-106	109(6)	-92	98(8)	-110	91(10)	-107
147	Mepanipyrim	99(3)	-6	92(4)	-7	96(8)	-11	88(3)	-3
148	Metalaxyl	111(1)	3	100(0)	1	105(4)	0	103(2)	6
149	Metamitron	>140 (5)	7	139(1)	5	>140 (6)	1	118(4)	4
150	Metconazole	119(1)	-4	99(2)	-1	106(8)	-6	>140 (36)	0
151	Methamidophos	74(1)	0	76(1)	-1	91(2)	-2	76(2)	2
152	Methidathion	116(2)	-1	97(2)	-4	105(1)	-5	102(6)	-1
153	Methiocarb	102(3)	-1	100(4)	-5	97(0)	-3	110(13)	-2
154	Methiocarb-sulfone	97(3)	5	87(1)	1	98(2)	2	98(4)	5
155	Methiocarb-sulfoxide	98(2)	2	95(3)	-2	97(2)	-1	92(5)	1
156	Methomyl	101(4)	-1	97(2)	-4	102(1)	-4	102(1)	-1
157	Methoxyfenozide	114(2)	0	97(5)	-2	104(2)	-6	102(5)	1
158	Metobromuron	106(3)	1	98(1)	0	99(5)	-1	96(2)	4
159	Metolachlor	114(5)	-3	99(1)	-3	99(3)	-8	104(5)	-11
160	Metolcarb	110(2)	1	102(1)	0	106(2)	-2	104(2)	3
161	Metrafenone	>140 (3)	-10	99(4)	-17	94(6)	-24	115(11)	-1
162	Monocrotophos	102(1)	3	101(6)	0	103(7)	-3	92(6)	1

163	Monolinuron	108(1)	2	102(1)	1	102(3)	-3	99(3)	2
164	Monuron	105(2)	-3	100(1)	-1	105(4)	-5	96(2)	-1
165	Myclobutanil	105(6)	0	90(4)	5	103(8)	-1	103(8)	0
166	Neburon	120(3)	-9	108(4)	-8	94(2)	-15	103(3)	-2
167	Nitenpyram	93(3)	-3	98(1)	-2	97(4)	-5	ND	ND
168	Novaluron	132(43)	-50	136(28)	-45	90(12)	-49	102(18)	-23
169	Omethoate	93(1)	-2	87(1)	-2	101(5)	-1	87(4)	-2
170	Orthosulfamuron	78(5)	-5	70(4)	-6	34(5)	-10	87(2)	-1
171	Oxadiargyl	>140 (12)	-11	135(13)	-17	116(10)	-33	>140 (6)	-3
172	Oxadixyl	107(4)	0	102(3)	0	100(5)	1	101(2)	2
173	Oxamyl-NH4	99(5)	-1	97(3)	-2	97(4)	-6	100(1)	1
174	Oxasulfuron	87(3)	-3	66(0)	-4	37(3)	-8	91(3)	-4
175	Oxathiapiprolin	114(4)	-2	104(6)	-1	99(3)	-3	103(8)	0
176	Oxfendazole	98(2)	3	89(2)	3	87(3)	-1	91(5)	3
177	Paclobutrazol	110(3)	-1	93(2)	0	105(5)	-4	95(5)	1
178	Penconazole	108(5)	-1	96(2)	-3	98(3)	-5	95(3)	2
179	Pencycuron	>140 (1)	-10	100(3)	-19	92(6)	-24	105(7)	-14
180	Pendimethalin	>140 (6)	-57	106(20)	-53	111(16)	-54	127(11)	-17
181	Penflufen	119(3)	3	103(2)	1	107(2)	-2	100(5)	4
182	Penthiopyrad	114(5)	-4	100(3)	-3	101(3)	-8	97(3)	1
183	Permethrin	ND	ND	ND	ND	ND	ND	127(13)	-4
184	Phenothrin	ND	ND	ND	ND	ND	ND	130(11)	-19
185	Phenthoate	>140 (1)	-11	103(1)	-15	91(2)	-21	104(3)	-1
186	Phosalone	>140 (2)	-8	113(3)	-25	89(9)	-34	107(3)	-5
187	Phosmet	114(4)	-3	102(1)	-4	100(3)	-5	101(4)	1
188	Phoxim	>140 (3)	-11	105(6)	-27	88(6)	-35	>140 (1)	-5
189	Pirimicarb	107(4)	2	99(2)	-1	100(2)	-2	100(2)	1
190	Pirimiphos-methyl	>140 (4)	7	111(2)	-1	121(4)	-1	106(2)	-1
191	Prochloraz	118(4)	3	100(1)	-1	107(4)	-3	90(8)	5
192	Profenofos	>140 (2)	-13	93(3)	-19	88(8)	-26	97(8)	-4
193	Promecarb	117(3)	1	105(0)	2	106(2)	-2	100(5)	1
194	Prometryn	113(1)	0	99(1)	-3	107(4)	-4	96(1)	-5
195	Propamocarb	95(5)	-4	84(2)	-5	61(7)	-8	102(3)	1
196	Propaquizafop	>140 (4)	-12	97(4)	-24	91(9)	-33	105(7)	-5
197	Propazine	107(3)	2	99(3)	2	101(3)	-4	101(3)	4
198	Propiconazole	116(2)	-4	93(2)	-3	103(3)	-7	87(7)	7
199	Propoxur	108(1)	2	104(2)	0	106(2)	-1	104(4)	3
200	Propyzamide	112(2)	1	99(6)	1	103(6)	-2	107(3)	3
201	Proquinazid	>140 (2)	-12	101(13)	-47	85(9)	-49	105(10)	-14
202	Prosulfocarb	>140 (5)	-14	104(3)	-20	86(7)	-28	106(9)	-6
203	Pymetrozine	81(2)	1	66(3)	-1	100(3)	-6	73(2)	1
204	Pyraclostrobin	>140 (5)	-13	98(4)	-14	96(6)	-25	>140 (4)	-2
205	Pyrethrins1	>140 (10)	-54	115(21)	-52	85(26)	-52	105(17)	-14

206	Pyrethrins2	>140 (3)	-19	130(2)	-17	106(7)	-27	101(18)	-12
207	Pyridaben	>140 (6)	-60	126(33)	-67	89(19)	-52	128(15)	-22
208	Pyridalyl	>140 (11)	-14	>140 (52)	-62	90(42)	-29	116(12)	26
209	Pyridaphenthion	119(2)	0	99(1)	1	105(3)	-3	100(6)	2
210	Pyrifoenone	140(5)	-11	96(1)	-10	89(6)	-25	102(7)	-4
211	Pyrimethanil	103(4)	1	103(3)	-1	98(4)	-4	100(4)	0
212	Pyriproxyfen	>140 (2)	-52	122(17)	-51	100(14)	-51	119(12)	-18
213	Quinalphos	>140 (4)	-9	101(1)	-9	98(6)	-22	105(5)	-1
214	Quinoclamine	107(2)	2	95(4)	0	98(2)	-2	105(4)	6
215	Quinoxyphen	>140 (2)	-14	128(5)	-30	130(12)	-37	98(5)	-9
216	Quizalofop-P-ethyl	>140 (3)	-9	102(6)	-21	83(4)	-32	109(9)	-4
217	Rotenone	138(4)	-8	92(5)	-12	101(11)	-17	103(5)	-3
218	Simazine	105(1)	2	98(4)	0	104(1)	-2	98(6)	5
219	Spinetoram J	108(3)	16	96(3)	14	95(3)	11	105(4)	-58
220	Spinetoram L	105(11)	26	98(6)	26	107(6)	30	120(5)	29
221	Spinosyn A	110(8)	12	102(5)	12	97(3)	6	93(12)	14
222	Spinosyn D	117(13)	21	110(5)	16	104(3)	17	97(10)	-58
223	Spirodiclofen	>140 (12)	-60	127(21)	-63	92(17)	-53	125(11)	-24
224	Spiromesifen	>140 (13)	-9	90(11)	-50	125(29)	-51	108(13)	-23
225	Spirotetramat	100(4)	-15	96(3)	-12	90(5)	-16	107(4)	-13
226	Spiroxamine	105(3)	-3	101(3)	-2	90(3)	-5	95(7)	-100
227	Sulfoxaflor	101(3)	2	96(2)	0	99(4)	1	100(4)	2
228	Tau-Fluvalinate	ND	ND	ND	ND	ND	ND	>140 (21)	16
229	Tebuconazole	109(0)	2	108(3)	-2	98(5)	-3	98(3)	4
230	Tebufenozide	116(2)	-5	109(3)	-4	102(5)	-6	99(5)	-3
231	Tebufenpyrad	>140 (3)	-15	97(4)	-22	89(10)	-30	104(6)	-4
232	Teflubenzuron neg	>140 (23)	-23	112(11)	-32	85(13)	-37	112(6)	-11
233	Terbuthylazine	104(3)	-3	97(1)	-1	99(2)	-5	100(4)	0
234	Terbuthylazine-desethyl	101(4)	0	96(2)	-4	100(5)	-3	96(3)	-1
235	Terbutryn(e)	112(2)	3	104(2)	0	104(3)	-4	92(3)	-1
236	Tetraconazole	112(7)	-2	92(2)	-3	102(2)	-4	105(4)	44
237	Tetramethrin	>140 (4)	-10	107(4)	-24	81(9)	-35	104(9)	-1
238	Thiabendazole	94(3)	3	85(4)	2	97(5)	2	88(4)	1
239	Thiacloprid	108(3)	1	104(2)	-2	101(3)	-5	103(4)	-2
240	Thiamethoxam	99(2)	1	102(3)	1	103(4)	-4	93(5)	0
241	Thiobencarb	>140 (3)	-13	103(6)	-16	84(9)	-24	105(9)	-3
242	Tolfenpyrad	>140 (5)	-12	96(10)	-42	85(19)	-45	105(11)	-9
243	Triadimefon	105(2)	3	108(6)	3	96(8)	-2	106(4)	6
244	Triallate	ND	ND	ND	ND	ND	ND	108(4)	-18
245	Triazophos	120(2)	-3	101(1)	-3	99(2)	-8	102(5)	0
246	Trichlorfon	98(4)	4	94(1)	-3	94(4)	-2	100(2)	4
247	Triclocarban	>140 (3)	-51	111(18)	-47	72(10)	-49	107(8)	-2

248	Tricyclazole	98(2)	1	95(1)	0	98(4)	-2	96(1)	1
249	Trifloxystrobin	>140 (4)	-15	104(4)	-21	88(14)	-31	102(8)	-64
250	Triflumizole	126(6)	-6	111(3)	-7	112(3)	-8	79(4)	-20
251	Triflumuron	>140 (6)	-15	109(2)	-19	92(9)	-25	112(6)	0
252	Trinexapac-ethyl	36(6)	-2	26(1)	-6	41(11)	-2	88(6)	-3
253	Trinexapac-methyl	27(7)	-2	24(8)	-6	34(13)	-13	77(4)	-1
254	Triticonazole	101(2)	0	96(6)	0	92(2)	-4	99(8)	4
255	Tritosulfuron	100(7)	-3	86(12)	-3	73(9)	1	100(4)	-1
256	Valifenalate	117(1)	1	107(1)	-1	110(3)	-2	107(7)	1
257	XMC	108(1)	1	100(2)	-1	104(2)	0	101(2)	2
258	Zoxamide	132(4)	-6	100(4)	-9	98(2)	-20	102(4)	3