EURL for Cereals and Feeding stuff
National Food Institute
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Validation Report 31A

Determination of pesticide residues in rice based babyfood by LC-MS/MS and GC-MS/MS

(QuEChERS method)

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CONTENT:

1. Introduction

EFSA drafted a Scientific Opinion on pesticides in foods for infants and young children (published 28 June 2018)[1], which concluded that the default MRL 0.01 mg/kg might now not be sufficiently protective for infants below the age of 16 weeks. This default MRL cor-responds to an ADI of 0.0026 mg/kg bw (based on a 260 g/kg bw intake of infant formula. The Commission requested from the EURLs information on the lowest achievable LOQs for the pesticides on the EFSA list. Prioritised substances were chlorpyrifos, cyhalothrin, cypermethrin, fluquinconazole, ethoprophos and emamectin. The EURL-CF has consequently validated rice based baby food although this is not recommended for infant up to 16 weeks. Our first approach was to see if the standard QuEChERS method could fulfil the requirement if the extracts were analysed on the most sensitive instruments. This report describes, consequently, the validation of the QuEChERS method combined with GC-MS/MS and LC-MS/MS. The method was tried validated for 255 pesticides and metabolites by both LC-MSMS and GC-MSMS in rice based baby food.

2. Principle of analysis

Sample preparation: The baby food was analysed as ready to consume, so 25 g powder was mixed with 25 ml 50 ºC MiliQ water, that was boiled and then cooled to 50 ºC.

The extraction procedure is outlines in Appendix 4 and described briefly in the following.

Extraction: 5 g porridge was added 5 ml water and mixed well using ceramic homogenizers. The samples was then extracted with 10 ml acetonitrile using a Geno grinder and a salt and buffer mixture is added and the sample is shaken again.

Clean-up: After centrifugation the supernatant is transferred to a clean tube and put in -80 degree freezer for minimum 15 minutes. The extracts are then allowed to thaw until almost liquid state and then centrifuged. At this point an aliquot is withdrawn and filtered, diluted 1:1 with acetonitrile and analysed by LC-MS/MS. The rest of the supernatant is transferred to a tube containing PSA and $MgSO_4$. After shaking and an additional centrifugation step the final extract is diluted 1:1 with acetonitrile to obtain the same matrix concentration as in the matrix matched calibration standards.

Quantification and qualification: The final extracts are analysed by GC-MS/MS. Crude extract withdrawn before PSA clean-up was analysed by LC-MS/MS.

GC-MS/MS: The GC used was Trace 1300 series with an TriPlus RSH Autosampler-GC Liquids. The column was TG-5SILMS 30mx0.25mmx0.25µm. Injection volume was 1 µL on a PTV. Mass spectrometer was Thermo TSQ 8000 Evo, upgrade of the MS to TSQ 9000 Evo with a new ion source, the Advanced Electron Ionization source, AEI. The quadrupole was operating in the multiple reaction

monitoring mode (MRM) with electron energy at 50 eV, source temperature at 250°C and transfer line at 250°C. For each pesticide minimum two sets of precursor and product ions were determined. One for quantification and one-three for qualification. The MRM transitions for the pesticides and degradation products are given in Appendix 1a.

LC-MS/MS: The pesticide residues are separated on a reversed-phase column and detected by tandem mass spectrometry (MS/MS) by electrospray (ESI), Bruker EVOQ. The validation includes pesticides determined in positive and negative mode. For each pesticide or metabolite a precursor ion and 2 product ions were determined. One product ion for quantification and one-two for qualification. The MRM transitions for the pesticides and degradation products sought validated are given in Appendix 1b.

3. Validation design

The method was sought validated for 255 pesticides or metabolites in rice based babyfood, see **Appendix 1**. The validation was performed on 3 times 6 replicates of the four spiking levels; 0.001, 0.002, 0.005 and 0.01 mg/kg. A blank sample was included.

4. Calibration curves and linearity

The calibration curve is determined by the analysis of each of the analysts at least 4 calibration levels within the range of 0.1 to 33 ng/ml. The quantification was performed from the mean of two bracketing calibration curves. The calibration curves were fitted to a linear curve. The majority of the correlation coefficients (R) were higher or equal to 0.99 but none were lower than 0.97. Thus, good linearity was observed within the relevant concentration range.

5. Specificity

The ion ratios for sample extracts were within ±30% (relative) of average of relevant calibration standards from same sequence. The ion ratios may vary slightly depending on concentration level and in some cases the average of calibration standard are based on the lower calibration levels for the low spike samples.

6. Precision – repeatability and internal reproducibility

Repeatability was calculated for all pesticides and degradation products on all three spiking levels (0.001, 0.002, 0.005 and 0.01 mg/kg). Repeatability is given as the relative standard deviation on the

result from two or more analysis at the same sample, done by the same technician, on the same instrument and within a short period of time.

Repeatability (RSD_r) in this validation was calculated from the 5-6 replicate determinations. Repeatability were calculated as given in ISO 5725-2[2].

Accuracy – Recovery
The accuracy was determined from recovery studies in which samples were spiked at three concentration levels (0.001, 0.002, 0.005 and 0.01 mg/kg) with the relevant pesticides, isomers and degradation products.

Robustness
The QuEChERS method has, in connection with the development of the method, been shown to be robust by Anastassiades et al. 2003[2].

Limit of quantification, LOQ
The quantification limits (LOQ) was determined as the lowest spike level for which the acceptance criteria (se Section 6) were meet.

7. Criteria for the acceptance of validation results

For the pesticides to be accepted as validated the following criteria for precision and trueness must to be fulfilled:

1. The relative standard deviation of the repeatability and internal reproducibility should be ≤20%[4].
2. The average relative recovery must be between 70 and 120%[3].

If the above mentioned criteria have been meet, the quantification limits, LOQs is stated.

The expanded uncertainty is calculated to demonstrate that it is less than 50%. The expanded uncertainty is given by:

$$U = \sqrt{RSD^2 + Bias^2 + (RSD^2/n)} * 2$$

Where RSD is the intra-laboratory uncertainty (RSD_R),
Bias is 100 minus the recovery,
RSD^2/n is the uncertainty of the bias,
n is the number of recoveries included in the bias and
2 is the coverage factor corresponding to 95% confidence level.

If the expanded uncertainty is higher than 50%, the analytical results must be corrected for recovery and the combined uncertainty is then given by:

$$U_c = \sqrt{RSD^2 + (RSD^2/n)}$$

Where RSD, in this validation, is the repeatability uncertainty (RSD_r),
RSD^2/n is the uncertainty of the bias,
n is the number of recoveries included in the bias and
2 is the coverage factor corresponding to 95% confidence level.

The bias/recovery used for correction will be the bias/recoveries determined for the individual analytes during the initial validation and/or ongoing method validation. However, if it is evaluated that the type of sample being analysed is significantly different from the matrices employed for the method validation it is possible to correct for bias/recoveries based on recovery from spiked samples included in the analytical batch in question. However, minimum of 5 recovery samples must be included then.

The obtained results including recovery, RSD_r, RSD_R, expanded uncertainty (U, Uc and limit of quantification (LOQ) are presented in Appendix 2.

8. Results and conclusion

The validation results obtained for the 255 pesticides or metabolites using LC-MSMS and GC-MSMS are presented in Appendix 2. In total, 231 compounds were validated, 192 at 0.001 mg/kg, 20 at 0.002 mg/kg, 10 at 0.005 mg/kg and 9 at 0.01 mg/kg. However, 24 compound was not validated.

Generally the combined uncertainties were lower than 50%, indicating that recovery for correction is not needed. However, it has been decided at our laboratory that all results shall be corrected for recovery when possible, regardless of the expanded uncertainty and the combined uncertainty will therefore apply.

The EFSA list of pesticides that needs lower MRLs than 0.01 mg/kg, see Appendix 3, includes 66 pesticides. Of them, 18 has to be analysed by so-called Single Residues Methods (SRM) which then cannot be analysed by Multi Residue Methods (MRM) like the QuEChERS method. This validation study validated 36 of the pesticides on the list were validated. However, 13 did not obtain LOQs low enough to fulfil the MRLs need (Aldrin, carbofuran, Chlordane, diazinon, dichlorvos, dieldrin, endrin, fipronil, cyhalothrin-lambda, monocrotophos, parathion, disulfoton and nitrofen). For some of the pesticides the lowest, spike level were too high and for other pesticide the instruments were not

sensitive enough. However, some pesticides failed due to siloxane interferences leaking from the vial lids.

The remaining 12 pesticides were not included in the study (carbophenothion, dioxathion, fluometuron, gamma-cyhalothrin, heptachlor, isofenphos, methomyl, phorate, quinoclamine, tembotrione, topramezone and triazoxide).

The pesticides that fulfilled the requirement for the very low MRLs were: cadusafos, chlordane, chlorpyrifos, DDTs, demeton-S-methyl, dicofol, dimethoate, ethion, ethoprophos, fenamiphos, fensulfothion, fluquinconazole, flusilazole, mecarbam, methamidophos, methidathion, methomyl, nitrofen, omethoate, oxamyl, oxydemeton-methyl, phosphamidon and triazophos.

As 12 compounds was lacking in the pesticide mixture used for validation and 13 compounds was not validated at a sufficiently low level, it was then decided to start a new validation study which included these compound, but only include two low spiking levels; 0.0005 and 0.001 mg/kg. To obtain better results, the instrument methods will be optimized e.g. by injecting higher volume of the extractions into the instrument than the 1µl used in this study; 5µl for the GCMSMS and 2 µl for the LCMSMS.

Although, it is possible to validate a row of compound at an LOQ level of 0.001 mg/ml, it is still requires a lot of skills and carefulness to analyse pesticide residues in babyfood. Due to relatively low levels of pesticide residue, the data often requires a manual evaluation of the response. The ion ratios are difficult to fulfill because the ion counts are low and often the qualifier is not sensitive enough to give a sufficient response. Consequently, the most sensitive instruments are needed to detect and quantify at this low levels. Hopefully, by increasing the injection volume the requirement needed for all the pesticides will be fulfilled.

9. References

1 Scientific opinion on pesticides in foods for infants and young children. EFSA Panel on Plant Protection Products and their Residues (PPR). EFSA Journal 2018;16(6):5286.
DOI: 10.2903/j.efsa.2018.5286.

2 EN 15662:2008. Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE - QuEChERS-method

3 ISO 5725-2:1994. Accuracy (trueness and precision) of measurement methods and results – Part2. Basic method for the determination of repeatability and reproducibility of standard measurement method. First edition. December 1994.

4 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed, Document SANTE/12682/2019, European Commission, Brussels, 2019.

Appendix 1a. MRM transitions for GC-MS/MS

Name	RT	Parent Mass	Product Mass	Collision Energy
2-phenylphenol	9.58	169.1	115.1	25
2-phenylphenol	9.58	169.1	141.1	10
2-phenylphenol	9.58	170.1	169.1	10
Acrinathrin(I)	23.5	181	152	22
Acrinathrin(I)	23.5	208.1	180.9	8
Acrinathrin(I)	23.5	289	93.1	22
Acrinathrin(II)	23.89	181	152	22
Acrinathrin(II)	23.89	208.1	180.9	8
Acrinathrin(II)	23.89	289	93.1	22
Aldrin	14.67	219.9	150	30
Aldrin	14.67	292.9	185.9	30
Aldrin	14.67	292.9	257.9	10
Atrazine	11.68	200.1	104.1	20
Atrazine	11.68	215.1	173.1	10
Atrazine	11.68	215.1	200.1	10
Azoxystrobin	29.8	344.1	172.1	25
Azoxystrobin	29.8	344.1	329.1	20
Azoxystrobin	29.8	388.1	345.1	15
Azoxystrobin-d4 (P-IS)	29.75	348	332	30
Bifenthrin	21.69	181.1	141	22
Bifenthrin	21.69	181.1	165.1	15
Bifenthrin	21.69	181.1	166.1	15
Bitertanol	25	170.1	115.1	25
Bitertanol	25	170.1	141.1	20
Bitertanol	25	171.2	142.1	20
Bixafen	27.37	159	139	10
Bixafen	27.37	413.1	159	12
Bixafen	27.37	415.1	159	12
Boscalid	26.69	342	140	15
Boscalid	26.69	344	142	15
Bromophos-ethyl	16.36	302.8	284.8	14
Bromophos-ethyl	16.36	358.9	302.8	14
Bromophos-ethyl	16.36	358.9	330.9	10
Bromopropylate	21.71	184.9	75.5	30
Bromopropylate	21.71	340.8	185	14
Bromopropylate	21.71	342.7	185	15
Bromuconazole(I)	21.46	173	109	25
Bromuconazole(I)	21.46	173	145	15
Bromuconazole(I)	21.46	294.9	173	10
Bromuconazole(II)	22.25	172.9	145	16
Bromuconazole(II)	22.25	293	173	10
Bromuconazole(II)	22.25	295	175	10
Bupirimate	17.72	273.1	193.1	10

Name	RT	Parent Mass	Product Mass	Collision Energy
Bupirimate	17.72	316.2	208.1	10
Cadusafos	10.97	159	97	20
Cadusafos	10.97	213.1	89	12
Carbofuran	11.51	164.1	149.1	10
Carbofuran	11.51	221.1	164.1	5
Carboxin	17.76	235.1	87	15
Carboxin	17.76	235.1	143	15
Chlordane-cis	16.81	236.9	142.9	24
Chlordane-cis	16.81	271.7	236.9	8
Chlordane-trans	16.46	271.7	236.8	12
Chlordane-trans	16.46	372.8	265.9	18
Chlordane-trans	16.46	374.7	265.9	20
Chlorfenapyr	17.99	248.9	112	24
Chlorfenapyr	17.99	363.8	247.2	30
Chlorfenapyr	17.99	363.8	282.1	20
Chlorfenson	17.14	111	75	14
Chlorfenson	17.14	175	75	28
Chlorfenson	17.14	175	111	10
Chlorfenvinphos	15.72	266.9	159	15
Chlorfenvinphos	15.72	268.9	161	12
Chlorfenvinphos	15.72	323	267	15
Chlormephos	8.8	154	121	5
Chlormephos	8.8	234	121	14
Chlorobenzilate	18.51	139	74.9	26
Chlorobenzilate	18.51	139	111	12
Chlorobenzilate	18.51	251	139	14
Chlorpropham	10.68	213.1	127	15
Chlorpropham	10.68	213.1	171	10
Chlorpyrifos	14.45	196.7	168.9	12
Chlorpyrifos	14.45	313.9	257.9	12
Chlorpyrifos	14.45	316.1	260	15
Chlorpyrifos-d10 (I-IS)	14.32	200	109	36
Chlorpyrifos-d10 (I-IS)	14.32	200	172	12
Chlorpyrifos-methyl	13.24	285.9	93	20
Chlorpyrifos-methyl	13.24	285.9	270.9	25
Chlorpyrifos-methyl	13.24	287.9	272.9	25
Clofentezine	22.79	102	50.9	12
Clofentezine	22.79	102	74.9	12
Clofentezine	22.79	137.6	102	12
Clomazone	11.71	125	89	15
Clomazone	11.71	204	107	15
Cyflutrin	26.25	206	151	12
Cyflutrin	26.25	226	206	10
Cyhalothrin-lambda(I)	23.2	180.9	151.9	22
Cyhalothrin-lambda(I)	23.2	197	141.1	10

Name	RT	Parent Mass	Product Mass	Collision Energy
Cyhalothrin-lambda(I)	23.2	208.1	180.9	8
Cyhalothrin-lambda(II)	23.6	181	151.9	22
Cyhalothrin-lambda(II)	23.6	208.1	151.8	28
Cyhalothrin-lambda(II)	23.6	208.1	180.9	8
Cypermethrin	26.985	163	127	10
Cypermethrin	26.985	181	152	20
Cyproconazole	18.22	383	282	20
Cyproconazole	18.22	383.1	254	20
Cyprodinil	15.46	224.1	118	30
Cyprodinil	15.46	224.1	208.1	30
Cyprodinil	15.46	225.1	210.1	25
DDD-pp	18.82	235	165	20
DDD-pp	18.82	236.8	165	20
DDD-pp	18.82	237	165	20
DDE-pp	17.47	246.1	176	25
DDE-pp	17.47	317.8	248	18
DDE-pp	17.47	317.9	246	20
DDT-op	18.93	235	165	22
DDT-op	18.93	236.8	165.1	20
DDT-op	18.93	237	165	20
DDT-pp	20.03	235	165	20
DDT-pp	20.03	235	165.1	22
DDT-pp	20.03	237	165	20
DEET	9.9	190.1	117	20
DEET	9.9	190.1	145	20
Demeton-S-methyl	10.39	88	59.8	6
Demeton-S-methyl	10.39	109	79	6
Demeton-S-methyl	10.39	141.9	79	12
Diazinon	12.02	179	137	15
Diazinon	12.02	199	93	15
Diazinon	12.02	304.1	179.1	15
Dichlofluanid	14.25	224	123	15
Dichlofluanid	14.25	226	123	15
Dichlorvos	7.58	185	93	12
Dichlorvos	7.58	185	109	17
Dichlorvos	7.58	220	185	10
Dichlorvos-d6 (P-IS)	7.55	191	99	15
Dichlorvos-d6 (P-IS)	7.55	191	115	20
Dicloran	11.54	160	124	10
Dicloran	11.54	206	176	10
Dicloran	11.54	208	178	10
Dicofol-pp	20.47	111	74.9	8
Dicofol-pp	20.47	139	111	15
Dieldrin	17.65	276.9	240.9	12
Dieldrin	17.65	278.9	242.9	12

Name	RT	Parent Mass	Product Mass	Collision Energy
Dieldrin	17.65	280.9	244.9	10
Difenoconazole(I+II)	29.075	323.1	265	15
Difenoconazole(I+II)	29.075	324.9	267	10
Difenoconazole(I+II)	29.075	325.1	267	20
Dimethomorph(I)	29.96	301	139	14
Dimethomorph(I)	29.96	301.1	165	12
Dimethomorph(I)	29.96	387.1	301.1	12
Dimethomorph(II)	30.45	301.1	165	12
Dimethomorph(II)	30.45	387.1	301.1	12
Diphenylamine	10.38	168.1	139	38
Diphenylamine	10.38	168.1	167.1	14
Diphenylamine	10.38	169.2	167.1	22
Disulfoton	12.32	142	109	10
Disulfoton	12.32	274	88	10
Endosulfan-alpha	16.87	195	160	10
Endosulfan-alpha	16.87	240.9	205.9	10
Endosulfan-alpha	16.87	242.9	207.9	10
Endosulfan-beta	18.68	195	160	10
Endosulfan-beta	18.68	240.9	205.9	10
Endosulfan-beta	18.68	242.9	207.9	10
Endosulfan-sulfate	19.89	269.9	234.9	15
Endosulfan-sulfate	19.89	271.9	234.9	10
Endosulfan-sulfate	19.89	273.9	238.9	10
Endrin	18.32	244.8	173	22
Endrin	18.32	262.8	192.9	30
Endrin	18.32	278.9	242.9	12
EPN	21.64	157	110	15
EPN	21.64	169	77	16
Epoxiconazole	20.92	165	138	8
Epoxiconazole	20.92	192	111	10
Epoxiconazole	20.92	192	138	10
Ethion	18.75	231	129	20
Ethion	18.75	384	231	10
Ethoprophos	10.48	158	97	10
Ethoprophos	10.48	200	158	8
Ethoxyquin	11.45	174.1	131.2	18
Ethoxyquin	11.45	174.1	146.1	12
Ethoxyquin	11.45	202.1	174.1	14
Etofenprox	27.21	163.1	107.1	16
Etofenprox	27.21	163.1	135.1	10
Etofenprox	27.21	376.2	163.1	20
Etofenprox-d5 (P-IS)	27.15	168	108	10
Etofenprox-d5 (P-IS)	27.15	168	136	20
Fenarimol	23.78	139	75	26
Fenarimol	23.78	219	107	15

Name	RT	Parent Mass	Product Mass	Collision Energy
Fenarimol	23.78	251	139	15
Fenazaquin	22.29	145.1	117.1	12
Fenazaquin	22.29	160	117.1	20
Fenazaquin	22.29	160	145.1	8
Fenbuconazole	25.91	129	77.8	18
Fenbuconazole	25.91	129	102	15
Fenbuconazole	25.91	198.1	129	15
Fenitrothion	14.03	277	109	20
Fenitrothion	14.03	277	260	10
Fenoxycarb	21.77	116	44	16
Fenoxycarb	21.77	116	88	8
Fenoxycarb	21.77	255.1	186.1	10
Fenpropathrin	21.99	265.1	89	20
Fenpropathrin	21.99	265.1	210.1	15
Fenpropimorph	14.68	128.1	110.1	15
Fenpropimorph	14.68	303.3	128.1	15
Fenson	15.07	268	77	20
Fenson	15.07	268	141	10
Fensulfothion	18.58	293	97	10
Fensulfothion	18.58	293	125	15
Fenthion	14.58	278	109	18
Fenthion	14.58	278	169	14
Fenvalerate(I+II)	28.48	125	89	18
Fenvalerate(I+II)	28.48	167	89	32
Fenvalerate(I+II)	28.48	419.1	225.1	10
Fluazifop-P-butyl	18.2	383.1	254.1	20
Fluazifop-P-butyl	18.2	383.1	282.1	15
Fludioxonil	17.27	248	127	20
Fludioxonil	17.27	248	154	20
Fludioxonil	17.27	248	182	15
Flufenoxuron	12.24	267.9	170	24
Flufenoxuron	12.24	267.9	241	12
Flufenoxuron	12.24	331	276	15
Fluquinconazole	25.21	340	108.1	36
Fluquinconazole	25.21	340	298	22
Fluquinconazole	25.21	340	313	14
Flusilazole	17.66	233	151.9	14
Flusilazole	17.66	233	164.9	16
Flusilazole	17.66	233.1	183.1	25
Flutriafol	16.93	123	75	15
Flutriafol	16.93	219.1	123	15
Fluvalinate-tau(I+II)	28.615	180.8	152.1	22
Fluvalinate-tau(I+II)	28.615	250	199.9	18
Fluvalinate-tau(I+II)	28.615	250.1	55	15
Formothion	12.8	93	63	10

Name	RT	Parent Mass	Product Mass	Collision Energy
Formothion	12.8	126	93	8
Formothion	12.8	170	93	5
HCH-alpha	11.25	182.9	147	12
HCH-alpha	11.25	216.9	180.9	8
HCH-alpha	11.25	218.9	182.9	8
HCH-beta	11.74	182.9	147	12
HCH-beta	11.74	216.9	180.9	8
HCH-beta	11.74	218.7	183	8
Heptachlorepoxide-cis	15.86	134.9	99	15
Heptachlorepoxide-cis	15.86	288.9	218.9	15
Heptachlorepoxide-cis	15.86	288.9	252.9	15
Heptachlorepoxide-trans	15.77	352.8	252.9	15
Heptachlorepoxide-trans	15.77	352.8	262.9	16
Heptachlorepoxide-trans	15.77	352.8	281.9	15
Heptenophos	9.93	124	89	10
Heptenophos	9.93	250	89	25
Hexachlorobenzene	11.3	248.8	213.9	14
Hexachlorobenzene	11.3	283.8	248.8	20
Hexachlorobenzene	11.3	285.8	250.8	20
Hexaconazole	17.2	213.9	159	18
Hexaconazole	17.2	231.1	175	10
Hexaconazole	17.2	256.1	159	10
Hexythiazox	16.39	184	59	20
Hexythiazox	16.39	184	149	6
Hexythiazox	16.39	227	149.1	8
Indoxacarb	29.37	203	134	20
Indoxacarb	29.37	264	176	20
Iprodione	21.34	314	245	15
Iprodione	21.34	314	271	10
Iprodione	21.34	316	247	15
Iprovalicarb(I)	17.58	118.9	117.1	8
Iprovalicarb(I)	17.58	134.1	42	20
Iprovalicarb(II)	17.89	118.9	117.1	8
Iprovalicarb(II)	17.89	134.1	42	20
Isofenphos-methyl	15.26	199	121	10
Isofenphos-methyl	15.26	241.1	199	8
Isoprothiolane	17.24	204	85	25
Isoprothiolane	17.24	204	118	7
Isoprothiolane	17.24	290.1	118	15
Jodofenfos	17.14	125	47	12
Jodofenfos	17.14	376.8	361.8	16
Jodofenfos	17.14	379	364	20
Kresoxim-methyl	17.72	116	63	24
Kresoxim-methyl	17.72	116	89	14
Kresoxim-methyl	17.72	206.1	116.1	15

Name	RT	Parent Mass	Product Mass	Collision Energy
Lindane	11.9	182.9	147	12
Lindane	11.9	216.9	180.9	8
Lindane	11.9	218.9	182.9	8
Linuron	14.21	248.1	61	15
Linuron	14.21	250.1	61	15
Metalaxyl	13.58	206.1	132.1	20
Metalaxyl	13.58	234.1	174.1	10
Metalaxyl	13.58	249.1	190.1	10
Methacrifos	9.29	240	180	5
Methacrifos	9.29	240	208	10
Methidathion	16.33	145	58	15
Methidathion	16.33	145	85	10
Metribuzin	13.19	198.1	82	17
Metribuzin	13.19	198.1	89	16
Metribuzin	13.19	198.1	110.1	10
Mevinphos	8.75	127	95	14
Mevinphos	8.75	127	109	10
Mevinphos	8.75	192	127	10
Myclobutanil	17.6	179.1	125.1	15
Myclobutanil	17.6	179.1	152.1	8
Myclobutanil	17.6	288.1	179.1	10
Nitrofen	18.27	283	162	15
Nitrofen	18.27	283	202	20
Nuarimol	20.47	235.1	139	15
Nuarimol	20.47	314.1	139	15
Oxadixyl	18.76	163	117	30
Oxadixyl	18.76	163.1	132.1	8
Oxadixyl	18.76	233.1	146.1	10
Oxychlordane	15.78	115	50.9	22
Oxychlordane	15.78	184.9	84.9	26
Oxychlordane	15.78	184.9	121	12
Paclobutrazol	16.59	125	89	18
Paclobutrazol	16.59	236.1	125.1	15
Paclobutrazol	16.59	238.1	127.1	15
Parathion	14.69	235	139	8
Parathion	14.69	291	81	20
Parathion	14.69	291	109	8
Parathion-methyl	13.37	125	47	12
Parathion-methyl	13.37	263	79	22
Parathion-methyl	13.37	263	109	11
Penconazole	15.61	248	157	25
Penconazole	15.61	248	192	13
Pencycuron	11.72	125	89	15
Pencycuron	11.72	125	99	16
Pendimethalin	15.44	252.1	162.1	12

Name	RT	Parent Mass	Product Mass	Collision Energy
Pendimethalin	15.44	252.1	191.1	12
Pendimethalin	15.44	281.1	252.1	12
Permethrin(I+II)	25.1	183	153	15
Permethrin(I+II)	25.1	183.1	165.1	12
Permethrin(I+II)	25.1	183.1	168.1	12
Phenthoate	15.88	121	77	22
Phenthoate	15.88	246	121	8
Phenthoate	15.88	274	121	10
Phosalone	22.78	182	111	15
Phosalone	22.78	367	182	10
Phosmet	21.54	160	77	20
Phosmet	21.54	160	133	10
Pirimicarb	12.61	166.1	71	25
Pirimicarb	12.61	166.1	96.1	10
Pirimicarb	12.61	238.1	166.1	15
Pirimicarb-desmethyl	12.87	152.1	96.1	15
Pirimicarb-desmethyl	12.87	224.1	152.1	10
Pirimiphos-ethyl	15.055	304.1	168.1	15
Pirimiphos-ethyl	15.055	318.1	166.1	13
Pirimiphos-ethyl	15.055	333	168	15
Pirimiphos-methyl	13.98	290.1	125	15
Pirimiphos-methyl	13.98	290.1	233.1	10
Pirimiphos-methyl	13.98	305.1	180	15
Prochloraz	25.34	180.1	138.1	12
Prochloraz	25.34	308	266	10
Prochloraz	25.34	310	268	10
Procymidone	16.03	283	67	15
Procymidone	16.03	283	96	10
Procymidone	16.03	283	96.1	8
Profenofos	17.37	337	267	20
Profenofos	17.37	339	269	20
Propoxur	10.14	110	62.9	24
Propoxur	10.14	110	64.1	16
Propoxur	10.14	152.1	110	8
Propyzamide	12	172.9	74	38
Propyzamide	12	172.9	109	26
Propyzamide	12	172.9	145	14
Prothiofos	17.25	267	239	10
Prothiofos	17.25	309	239	5
Pyrazophos	23.84	232	204	10
Pyrazophos	23.84	265.1	210.1	10
Pyrazophos	23.84	373	232	12
Pyridaben	25.26	147.1	117	10
Pyridaben	25.26	147.1	132.1	15
Pyridaben	25.26	309.1	147.1	15

Name	RT	Parent Mass	Product Mass	Collision Energy
Pyridaphenthion	21.32	340.1	199	10
Pyridaphenthion	21.32	340.1	203	25
Pyrimethanil	12.18	198.1	118.1	35
Pyrimethanil	12.18	199.1	198.1	10
Pyriproxyfen	23.14	136.1	78	20
Pyriproxyfen	23.14	136.1	96	10
Quinoxyfen	19.83	307	237.1	15
Quinoxyfen	19.83	307	272	10
Quinoxyfen	19.83	309	237.1	15
Simazine	11.57	172.7	138	6
Simazine	11.57	186	91	8
Simazine	11.57	201.1	138.1	10
Tebuconazole	20.47	125	89	16
Tebuconazole	20.47	250	125	20
Tebuconazole	20.47	252.1	127.1	20
Tebufenpyrad	22.18	276.1	171.1	15
Tebufenpyrad	22.18	333.2	171	20
Tebufenpyrad	22.18	333.2	276	10
Tecnazene	10.2	214.8	143.6	20
Tecnazene	10.2	214.8	178.7	10
Tecnazene	10.2	214.8	179.9	15
Tefluthrin	12.32	177	127	15
Tefluthrin	12.32	177	137	15
TEPP	8.88	98.8	73	15
TEPP	8.88	161.1	90.3	15
TEPP	8.88	161.1	98.9	20
Terbufos	11.93	231	157	15
Terbufos	11.93	288	231	5
Tetraconazole	14.75	336	204	20
Tetraconazole	14.75	336	218	20
Tetradifon	22.58	355.9	159	15
Tetradifon	22.58	355.9	228.9	10
Thiometon	11.36	88	45	20
Thiometon	11.36	88	60	6
Thiometon	11.36	125	47	14
Tolclofos-methyl	13.42	265	220	20
Tolclofos-methyl	13.42	265	250	15
Tolclofos-methyl	13.42	267	252	15
Tolyfluanid	15.7	137	65.1	28
Tolyfluanid	15.7	137	91.1	18
Tolyfluanid	15.7	238	137	10
TPP (I-IS)	20.65	325.07	169.04	25
TPP (I-IS)	20.65	326.07	215.05	25
TPP (I-IS)	20.65	326.07	325.07	10
Triadimefon	14.78	208.1	127	10

Name	RT	Parent Mass	Product Mass	Collision Energy
Triadimefon	14.78	208.1	181.1	10
Triallate	12.52	268	184	22
Triallate	12.52	270	186	22
Triazophos	19.31	161	106	12
Triazophos	19.31	161	134	8
Triazophos	19.31	257.1	162	10
Trichlorfon	7.59	145	109	10
Trichlorfon	7.59	185	93	12
Tricyclazole	17.24	162	133.9	8
Tricyclazole	17.24	204.1	118	5
Tricyclazole	17.24	231	189	10
Trifloxystrobin	19.73	190.1	130	10
Trifloxystrobin	19.73	222.1	130	10
Trifloxystrobin	19.73	222.1	162.1	10
Trifluralin	10.63	264.1	160.1	15
Trifluralin	10.63	306.1	206	15
Trifluralin	10.63	306.1	264.1	15
Triticonazole	22.8	182	75.1	30
Triticonazole	22.8	182	111	15
Triticonazole	22.8	235.1	182.1	10
Vinclozolin	13.31	212	145	15
Vinclozolin	13.31	212	172	15
Vinclozolin	13.31	285	212	15

Appendix 1b. MRM transitions for LC-MS/MS

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
3-hydroxycarbofuran	2.67	Positive	238	163	13
3-hydroxycarbofuran	2.67	Positive	238	181	10
Acephate	1.78	Positive	183.8	143	12
Acetamiprid	2.68	Positive	223	126	17
Acetamiprid	2.68	Positive	223	56	10
Aldicarb	4.65	Positive	212.8	116.1	50
Aldicarb	4.65	Positive	212.8	89.2	13
Aldicarb-sulfone	1.96	Positive	240.4	148.2	13
Aldicarb-sulfone	1.96	Positive	240.4	86.3	21
Aldicarb-sulfoxide	1.90	Positive	223.9	132	10
Aldicarb-sulfoxide	1.90	Positive	223.9	89.2	21
Amitraz	2.02	Positive	163	107	26
Amitraz	2.02	Positive	163	122	17
Atrazine	4.57	Positive	216	174	15
Atrazine	4.57	Positive	216	96	20
Atrazine	4.57	Positive	216	104	25
Azinphos-ethyl	5.77	Positive	346	233	12
Azinphos-ethyl	5.77	Positive	346	137	22
Azinphos-methyl	4.88	Positive	318	261	6
Azinphos-methyl	4.88	Positive	318	132	11
Azoxystrobin	5.14	Positive	404	372	15
Azoxystrobin	5.14	Positive	404	344	21
Azoxystrobin	5.14	Positive	404	328	39
Benfuracarb	6.89	Positive	411	252	12
Benfuracarb	6.89	Positive	411	162	32
Bifenthrin	8.28	Positive	440	181	10
Bifenthrin	8.28	Positive	440	166	35
Bitertanol	6.49	Positive	338	70	5
Bitertanol	6.49	Positive	338	99	12
Bitertanol	6.49	Positive	338	268	9
Bixafen	6.18	Positive	414	394	15
Bixafen	6.18	Positive	414	266	25
Bixafen	6.18	Positive	416	396	15
Boscalid	5.41	Positive	343	307	13
Boscalid	5.41	Positive	343	271	24
Bromoxynil	4.43	Negative	276	79	21
Bromoxynil	4.43	Negative	276	80.6	25
Bromoxynil	4.43	Negative	276	274.6	6
Bromuconazole	5.64	Positive	377.9	159	18
Bromuconazole	5.64	Positive	377.9	70	9
Bupirimate	5.62	Positive	317	166	23
Bupirimate	5.62	Positive	317	108	25
Buprofezin	6.97	Positive	306	116	14

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Buprofezin	6.97	Positive	306	201	8
Cadusafos	6.65	Positive	271	159	12
Cadusafos	6.65	Positive	271	131	21
Carbaryl	4.12	Positive	202	145	7
Carbaryl	4.12	Positive	202	127	27
Carbendazim	2.00	Positive	192	160	13
Carbendazim	2.00	Positive	192	105	32
Carbendazim	2.00	Positive	192	132	24
Carbofuran	3.85	Positive	222	165	10
Carbofuran	3.85	Positive	222	123	18
Carboxin	4.09	Positive	236	143	11
Carboxin	4.09	Positive	236	87	24
Carboxin	4.09	Positive	236	93	28
Chlorpyrifos	7.30	Positive	351.7	200	18
Chlorpyrifos	7.30	Positive	349.7	198	16
Clethodim	6.86	Positive	360	166	25
Clethodim	6.86	Positive	360	164	18
Clofentezine	6.53	Positive	303	138	12
Clofentezine	6.53	Positive	303	102	30
Clothianidin	2.50	Positive	250	169	13
Clothianidin	2.50	Positive	250	132	50
Cyazofamid	5.93	Positive	325	108	10
Cyazofamid	5.93	Positive	325	261	7
Cypermethrin	7.72	Positive	433	191	14
Cypermethrin	7.72	Positive	435	193	14
Deltamethrin_cis	7.75	Positive	523	281	14
Deltamethrin_cis	7.75	Positive	521	279	14
Demeton-S-methyl	3.88	Positive	231	89	10
Demeton-S-methyl	3.88	Positive	231	61	25
Demeton-S-methylsulfone	2.12	Positive	263	169	14
Demeton-S-methylsulfone	2.12	Positive	263	109	24
Demeton-S-methylsulfone	2.12	Positive	263	121	12
Diazinon	6.31	Positive	305	169	20
Diazinon	6.31	Positive	305	97	30
Diazinon	6.31	Positive	305	153	20
Dichlofluanid	5.82	Positive	333	123	23
Dichlofluanid	5.82	Positive	333	224	10
Dichlofluanid	5.82	Positive	333	121	5
Dichlorprop	5.47	Negative	233	161	10
Dichlorprop	5.47	Negative	233	125	26
Dichlorvos	3.78	Positive	221	109	16
Dichlorvos	3.78	Positive	238	221	4
Difenoconazole	6.71	Positive	406	251	24
Difenoconazole	6.71	Positive	406	188	42

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Difenoconazole	6.71	Positive	406	337	14
Diflubenzuron	6.07	Positive	311	158	8
Diflubenzuron	6.07	Positive	311	141	25
Dimethoate	2.68	Positive	230	199	8
Dimethoate	2.68	Positive	230	125	19
Dimethoate	2.68	Positive	230	171	14
Dinoterb	6.44	Negative	239	207	23
Dinoterb	6.44	Negative	239	136	34
Disulfoton	6.55	Positive	275	89	6
Disulfoton	6.55	Positive	275	61	19
Disulfoton sulfone	4.48	Positive	307	97	26
Disulfoton sulfone	4.48	Positive	307	125.5	17
Disulfoton sulfone	4.48	Positive	307	171	17
Disulfoton sulfoxide	4.38	Positive	291	213	8
Disulfoton sulfoxide	4.38	Positive	291	185	12
Disulfoton sulfoxide	4.38	Positive	291	153	12
Ditalimphos	5.70	Positive	300	148	16
Ditalimphos	5.70	Positive	300	130	30
DMF	3.56	Positive	150	106.8	20
DMF	3.56	Positive	150	132.2	35
DMPF	2.04	Positive	163	122	15
DMPF	2.04	Positive	163	107	25
DMPF	2.04	Positive	163	117	21
DMST	4.00	Positive	215	106.1	13
DMST	4.00	Positive	215	77	43
DMST	4.00	Positive	215	151	5
DNOC	4.37	Negative	196.9	137	17
DNOC	4.37	Negative	196.9	109.1	10
Epoxiconazole	5.86	Positive	330	121	18
Epoxiconazole	5.86	Positive	330	101	30
Ethiofencarb	4.25	Positive	226	107	11
Ethiofencarb	4.25	Positive	226	165	6
Ethion	7.20	Positive	385	199	10
Ethion	7.20	Positive	402	199	15
Ethoprophos	5.84	Positive	243.2	97	23
Ethoprophos	5.84	Positive	243.2	131	31
Etofenprox	8.20	Positive	394	177	14
Etofenprox	8.20	Positive	394	135	23
Etofenprox	8.20	Positive	394	359	11
Fenamiphos	6.02	Positive	304	216.9	21
Fenamiphos	6.02	Positive	304	201.7	35
Fenamiphos-sulfone	4.02	Positive	336	188	31
Fenamiphos-sulfone	4.02	Positive	336	266	50
Fenamiphos-sulfoxide	3.88	Positive	337.2	320.1	5

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Fenamiphos-sulfoxide	3.88	Positive	337.2	171.1	20
Fenazaquin	7.78	Positive	307	161.1	14
Fenazaquin	7.78	Positive	307	57.4	20
Fenazaquin	7.78	Positive	307	147.1	17
Fenbuconazole	5.99	Positive	337	125	25
Fenbuconazole	5.99	Positive	337	70.2	16
Fenhexamid	5.83	Positive	302	302	9
Fenhexamid	5.83	Positive	302	97	19
Fenoxycarb	6.11	Positive	302	116	8
Fenoxycarb	6.11	Positive	302	88	14
Fenoxycarb	6.11	Positive	302	256	12
Fenpropidin	4.25	Positive	274	147	24
Fenpropidin	4.25	Positive	274	117	31
Fensulfothion	4.66	Positive	309	281.3	10
Fensulfothion	4.66	Positive	309	157	27
Fensulfothion	4.66	Positive	309	173	27
Fenthion	6.23	Positive	279	169	16
Fenthion	6.23	Positive	279	105	21
Fenthion	6.23	Positive	279	247	11
Fenthion-oxon	4.94	Positive	263	216	20
Fenthion-oxon	4.94	Positive	263	231	30
Fenthion-oxon-sulfone	2.75	Positive	295	217	20
Fenthion-oxon-sulfone	2.75	Positive	295	104.1	33
Fenthion-oxon-sulfoxide	2.63	Positive	279.1	264	15
Fenthion-oxon-sulfoxide	2.63	Positive	279.1	104	20
Fenthion-sulfone	4.25	Positive	328	311	7
Fenthion-sulfone	4.25	Positive	328	125.1	22
Fenthion-sulfone	4.25	Positive	328	279	22
Fenthion-sulfoxide	4.05	Positive	295	280	17
Fenthion-sulfoxide	4.05	Positive	295	109.2	26
Fenthion-sulfoxide	4.05	Positive	295	125	30
Fipronil	6.09	Negative	435.2	330.2	13
Fipronil	6.09	Negative	435.2	250.1	42
Fipronil-desulfinyl	6.01	Negative	387	351	10
Fipronil-desulfinyl	6.01	Negative	389	353	15
Fipronil-desulfinyl	6.01	Negative	387	282	35
Fipronil-sulfide	6.23	Negative	419	383	10
Fipronil-sulfide	6.23	Negative	419	262	30
Fipronil-sulfide	6.23	Negative	421	385	10
Fipronil-sulfone	6.38	Negative	451	415	15
Fipronil-sulfone	6.38	Negative	451	282	25
Fipronil-sulfone	6.38	Negative	453	417	15
Fluazifop-p-butyl	7.00	Positive	384	282	18
Fluazifop-p-butyl	7.00	Positive	384	254	18

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Fluazifop-p-butyl	7.00	Positive	384	328	14
Fluoxastrobin	5.77	Positive	459.2	427.1	17
Fluoxastrobin	5.77	Positive	459.2	188	45
Fluquinconazole	5.72	Positive	376	307	22
Fluquinconazole	5.72	Positive	376	349	18
Flusilazole	6.05	Positive	316	247	17
Flusilazole	6.05	Positive	316	165	24
Fosthiazate	4.31	Positive	284.4	104.1	25
Fosthiazate	4.31	Positive	284.4	228.2	30
Heptenophos	4.80	Positive	251	127.1	14
Heptenophos	4.80	Positive	251	125	12
Hexaconazole	6.49	Positive	314	70	13
Hexaconazole	6.49	Positive	314	159.7	25
Hexythiazox	7.34	Positive	353	228	15
Hexythiazox	7.34	Positive	353	168	24
Imazalil	3.82	Positive	297	159	17
Imazalil	3.82	Positive	297	201	13
Imidacloprid	2.45	Positive	256	209	14
Imidacloprid	2.45	Positive	256	175	17
Indoxacarb	6.75	Positive	528	293	12
Indoxacarb	6.75	Positive	528	150	24
Indoxacarb	6.75	Positive	528	203	33
Iodosulfuron-methyl-sodium	4.95	Positive	530.1	163.1	13
Iodosulfuron-methyl-sodium	4.95	Positive	530.1	390	21
Iprodione	6.05	Positive	330	245	12
Iprodione	6.05	Positive	332	247	16
Iprovalicarb	5.72	Positive	321	119	14
Iprovalicarb	5.72	Positive	321	203	7
Isoprothiolane	5.47	Positive	291	231	10
Isoprothiolane	5.47	Positive	291	189	20
Isoproturon	4.66	Positive	207	72	12
Isoproturon	4.66	Positive	207	165	12
Linuron	5.21	Positive	249	160	16
Linuron	5.21	Positive	249	182	14
Lufenuron	7.20	Negative	511	158.1	10
Lufenuron	7.20	Negative	511	141	30
Malaoxon	3.90	Positive	315	127	10
Malaoxon	3.90	Positive	315	99	20
Malathion	5.44	Positive	331	127	10
Malathion	5.44	Positive	331	99	18
Mecarbam	5.79	Positive	330	227	8
Mecarbam	5.79	Positive	330	97	45
Mecarbam	5.79	Positive	330	199	14

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Mepanipyrim	5.67	Positive	224	106	20
Mepanipyrim	5.67	Positive	224	77	49
Metaflumizone	7.15	Negative	505.1	302.1	25
Metaflumizone	7.15	Negative	505.1	328	15
Metalaxyl	4.67	Positive	280	192	17
Metalaxyl	4.67	Positive	280	220	13
Metconazole	6.50	Positive	320	70	15
Metconazole	6.50	Positive	320	125	33
Methamidophos	1.66	Positive	142	94	12
Methamidophos	1.66	Positive	142	125	12
Methiocarb	5.28	Positive	243.4	121.2	30
Methiocarb	5.28	Positive	243.4	169.3	13
Methiocarb-sulfone	2.76	Positive	275	122	15
Methiocarb-sulfone	2.76	Positive	275	107	34
Methiocarb-sulfone	2.76	Positive	275	201	7
Methiocarb-sulfoxide	2.51	Positive	242	185	20
Methiocarb-sulfoxide	2.51	Positive	242	122	23
Methomyl	2.11	Positive	163	106	10
Methomyl	2.11	Positive	163	88	8
Methoxyfenozide	5.50	Positive	369	149	12
Methoxyfenozide	5.50	Positive	369	313	5
Metsulfuron-methyl	3.80	Positive	382	167	14
Metsulfuron-methyl	3.80	Positive	382	199	27
Mevinphos	2.63	Positive	225	127	13
Mevinphos	2.63	Positive	225	193	6
Monocrotophos	2.65	Positive	224	127	13
Monocrotophos	2.65	Positive	224	193	8
Monolinuron	4.24	Positive	215	126	17
Monolinuron	4.24	Positive	215	148	13
Ofurace	3.87	Positive	282	160	22
Ofurace	3.87	Positive	282	236	14
Omethoate	1.84	Positive	214	183	10
Omethoate	1.84	Positive	214	125	20
Omethoate	1.84	Positive	214	155	15
Oxamyl	2.00	Positive	237	72	10
Oxamyl	2.00	Positive	237	90	7
Oxycarboxin	2.93	Positive	268	175	13
Oxycarboxin	2.93	Positive	268	147	24
Oxydemeton-methyl	2.03	Positive	247	169	12
Oxydemeton-methyl	2.03	Positive	247	109	26
Paraoxon-methyl	3.39	Positive	265	202	35
Paraoxon-methyl	3.39	Positive	265	127	40
Penconazole	6.27	Positive	284	159	28
Penconazole	6.27	Positive	284	173	17

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Pencycuron	6.64	Positive	329	125	20
Pencycuron	6.64	Positive	329	218	14
Pendimethalin	7.36	Positive	282.1	212	10
Pendimethalin	7.36	Positive	282.1	194	10
Phosmet	4.96	Positive	335	160	17
Phosmet	4.96	Positive	335	133	36
Phosmet-oxon	3.91	Positive	302	160	21
Phosmet-oxon	3.91	Positive	302	133	31
Phosphamidon	3.49	Positive	300	127	22
Phosphamidon	3.49	Positive	300	174.1	13
Phosphamidon	3.49	Positive	300	227	12
Phoxim	6.43	Positive	299	129	9
Phoxim	6.43	Positive	299	153	6
Pirimicarb	3.10	Positive	239	72.1	16
Pirimicarb	3.10	Positive	239	182.3	25
Pirimiphos-methyl	6.41	Positive	306	164	20
Pirimiphos-methyl	6.41	Positive	306	108	20
Prochloraz	6.33	Positive	376	308	11
Prochloraz	6.33	Positive	376	266	17
Propamocarb	1.78	Positive	189	102	13
Propamocarb	1.78	Positive	189	74	23
Propamocarb	1.78	Positive	189	144	8
Propargite	7.46	Positive	368	231	10
Propargite	7.46	Positive	368	175	15
Propiconazole	6.37	Positive	342	159	20
Propiconazole	6.37	Positive	342	69	20
Propoxur	3.80	Positive	210.3	111.2	13
Propoxur	3.80	Positive	210.3	168.3	30
Propyzamide	5.48	Positive	256	190	13
Propyzamide	5.48	Positive	256	145	35
Propyzamide	5.48	Positive	256	173	21
Prosulfocarb	6.88	Positive	252	91	20
Prosulfocarb	6.88	Positive	252	128	10
Prosulfuron	5.27	Positive	420.3	141.1	15
Prosulfuron	5.27	Positive	420.3	167	15
Prothioconazole-desthio	5.92	Positive	312	125	25
Prothioconazole-desthio	5.92	Positive	312	70	18
Pymetrozine	1.69	Positive	218	105	15
Pymetrozine	1.69	Positive	218	79	20
Pyraclostrobin	6.45	Positive	388	163	19
Pyraclostrobin	6.45	Positive	388	194	8
Pyridate	8.05	Positive	379	207	17
Pyridate	8.05	Positive	379	351	9
Pyrimethanil	4.71	Positive	200	107	20

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Pyrimethanil	4.71	Positive	200	82	21
Pyriproxyfen	7.23	Positive	322	96.2	14
Pyriproxyfen	7.23	Positive	322	184.9	22
Pyriproxyfen	7.23	Positive	322	227	13
Quinoxyfen	7.31	Positive	308	161.9	47
Quinoxyfen	7.31	Positive	308	197	31
Simazine	3.85	Positive	202	124	17
Simazine	3.85	Positive	202	132	32
Spinosad_A	5.71	Positive	733	142	22
Spinosad_A	5.71	Positive	733	189	30
Spinosad_A	5.71	Positive	733	98	20
Spinosad_D	5.97	Positive	747	142	22
Spinosad_D	5.97	Positive	747	189	28
Spirodiclofen	7.61	Positive	411	313	20
Spirodiclofen	7.61	Positive	411	71	20
Spiroxamine	4.63	Positive	298	144	15
Spiroxamine	4.63	Positive	298	100	23
Tebufenozide	6.08	Positive	353	133	17
Tebufenozide	6.08	Positive	353	297	8
Tebufenpyrad	7.08	Positive	334	145	24
Tebufenpyrad	7.08	Positive	334	117	31
Teflubenzuron	7.23	Negative	379	339	9
Teflubenzuron	7.23	Negative	379	195	21
Teflubenzuron	7.23	Negative	379	359	6
Terbufos	7.06	Positive	289	57	18
Terbufos	7.06	Positive	289	103	18
Terbufos	7.06	Positive	289	233	8
Thiabendazole	2.14	Positive	202	175	20
Thiabendazole	2.14	Positive	202	131	27
Thiacloprid	2.95	Positive	253	126	17
Thiacloprid	2.95	Positive	253	90	30
Thiacloprid	2.95	Positive	253	99	37
Thiamethoxam	2.13	Positive	292	211	12
Thiamethoxam	2.13	Positive	292	132	19
Thiamethoxam	2.13	Positive	292	181	18
Thiodicarb	4.22	Positive	355	88	9
Thiodicarb	4.22	Positive	355	108	12
Thiodicarb	4.22	Positive	355	149	9
Thiometon	4.43	Positive	247	89	10
Thiometon	4.43	Positive	247	61	25
Thiophanate-methyl	3.76	Positive	342.8	151.1	20
Thiophanate-methyl	3.76	Positive	342.8	93.2	50
Thiophanate-methyl	3.76	Positive	343	311	8
Tolylfluanid	6.27	Positive	364	238	14

Name	RT	ESI mode	Parent Mass	Product Mass	Collision Energy
Tolylfluanid	6.27	Positive	364	137	29
Triadimenol	5.75	Positive	296	70	7
Triadimenol	5.75	Positive	296	99	12
Triallate	6.96	Positive	306	144.9	23
Triallate	6.96	Positive	306	86	24
Triazophos	5.66	Positive	314	162	17
Triazophos	5.66	Positive	314	119	30
Trichlorfon	2.71	Positive	274	109	20
Trichlorfon	2.71	Positive	274	127	18
Tricyclazole	3.10	Positive	190	136	26
Tricyclazole	3.10	Positive	190	109	32
Trifloxystrobin	6.77	Positive	409	186	11
Trifloxystrobin	6.77	Positive	409	145	36
Triflumuron	6.51	Positive	359.1	156.1	25
Triflumuron	6.51	Positive	359.1	138.8	20
Triticonazole	5.85	Positive	318	70	12
Triticonazole	5.85	Positive	318	125	31
Vamidothion	2.60	Positive	288	146	20
Vamidothion	2.60	Positive	288	118	30
Zoxamide	6.38	Positive	336	187	17
Zoxamide	6.38	Positive	336	132	11
Zoxamide	6.38	Positive	336	159	37

Appendix 2. Recoveries, repeatability (RSD_r), internal reproducibility (RSDR), expanded uncertainty (U) and Limit of Quantification (LOQ) for pesticides validated on four cereal commodities, oat, rice, rye and wheat using QuEChERS.

Red numbers indicate that the recovery is not 70-120% recovery or that RSD is above 20% RSD.

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		Recovery %	RSD_r %	RSD_R %	U %	Cu %	Recovery %	RSD_r %	RSD_R, %	U %	Cu %	Recovery %	RSD_r, %	RSD_R, %	U %	Cu %	Recovery %	RSDr %	RSD_R %	U %	Cu %	LOQ
GC	2-phenylphenol		83	20	20	53	20	92	8	19	41	19	113	8	8	31	8	119	5	9	42	10	0.001
LC	3-hydroxycarbofuran		102	9	20	42	21	103	9	15	31	15	99	6	11	22	11	94	9	15	34	16	0.001
LC	Acephate		91	9	11	28	11	91	7	6	22	6	88	8	13	37	14	87	6	10	34	10	0.001
LC	Acetamiprid		102	9	10	21	10	102	11	12	24	12	97	6	9	19	9	93	9	14	32	14	0.001
GC	Acrinathrin		92	6	16	36	17	91	20	35	74	36	87	9	19	48	20	75	12	20	65	21	0.001
LC	Aldicarb																						NV
LC	Aldicarb-sulfone		106	10	11	26	12	110	8	8	26	9	103	7	13	28	14	103	8	10	21	10	0.001
LC	Aldicarb-sulfoxide		97	16	20	42	21	100	16	20	40	20	99	10	19	40	20	97	10	17	35	17	0.001
GC	Aldrin	-	78	20	21	61	21	76	8	15	58	16	73	14	20	68	21	54	11	11	95	11	0.001
LC	Amitraz												112	12	18	44	18	106	8	10	23	10	0.005
GC	Atrazine							103	14	20	43	21	110	12	12	32	13	103	7	7	15	7	0.002
LC	Atrazine		96	5	5	13	6	102	7	8	18	9	99	4	8	16	8	98	6	9	20	10	0.001
LC	Azinphos-ethyl		101	13	21	43	21	98	7	12	25	12	94	7	11	25	11	94	7	9	21	9	0.001
LC	Azinphos-methyl		95	10	11	25	12	99	6	9	18	9	101	6	8	17	8	102	7	8	18	9	0.001
GC	Azoxystrobin		109	11	11	28	11	108	15	18	41	19	105	9	9	20	9	99	4	5	10	5	0.001
LC	Azoxystrobin		101	8	8	16	8	102	6	7	15	7	100	5	7	15	8	98	6	8	16	8	0.001
LC	Benfuracarb																						NV
GC	Bifenthrin		84	10	16	46	16	89	8	21	49	22	88	9	17	44	18	83	5	15	46	16	0.001
LC	Bifenthrin		95	5	17	36	17	90	8	18	43	19	90	11	19	44	20	82	6	9	40	9	0.001
GC	Bitertanol		85	10	16	45	17	87	16	18	46	19	100	9	16	33	16	95	5	7	18	7	0.001
LC	Bitertanol		105	14	14	29	14	104	10	10	22	10	101	7	8	17	8	97	7	7	16	7	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Bixafen		94	7	18	38	18	94	7	20	44	21	91	4	19	43	19	90	4	17	40	18	0.001
LC	Bixafen		96	7	7	16	7	103	9	13	27	13	102	6	10	21	10	101	6	11	22	11	0.001
GC	Boscalid		93	6	13	30	13	97	9	16	35	17	93	6	20	43	20	89	3	17	41	17	0.001
LC	Boscalid		100	8	14	28	14	102	8	8	17	8	104	4	8	18	8	102	7	9	18	9	0.001
GC	Bromophos-ethyl		82	9	20	55	20	90	12	20	45	20	81	9	17	53	18	70	5	19	72	20	0.001
GC	Bromopropylate		93	7	15	34	15	94	10	19	41	20	92	8	20	43	20	85	4	13	40	13	0.001
LC	Bromoxynil		108	16	19	43	20	105	20	20	42	20	107	17	17	38	18	105	18	20	42	21	0.001
GC	Bromuconazole		104	8	10	23	11	107	12	17	38	18	100	8	12	25	12	95	4	8	19	8	0.001
LC	Bromuconazole		114	9	21	52	22	105	8	9	21	10	100	7	11	22	11	98	6	9	20	10	0.001
GC	Bupirimate		96	11	17	37	18	101	12	19	40	20	100	7	19	40	20	97	5	19	39	19	0.001
LC	Bupirimate		98	7	10	21	10	100	8	8	17	9	99	5	8	17	8	97	7	10	20	10	0.001
LC	Buprofezin		98	8	9	19	9	100	10	10	20	10	95	11	15	32	15	94	11	13	29	13	0.001
GC	Cadusafos	+	99	9	10	21	11	107	11	13	30	13	100	9	12	24	12	98	3	7	15	7	0.001
LC	Cadusafos	+	102	5	7	15	7	102	9	9	19	9	101	4	10	20	10	94	13	14	30	14	0.001
LC	Carbaryl		100	10	11	24	12	109	10	17	39	17	102	10	16	33	16	101	11	17	35	18	0.001
LC	Carbendazim		102	4	7	15	7	103	8	8	18	9	98	7	12	24	12	96	8	9	20	9	0.001
LC	Carbofuran	-	100	4	14	28	14	105	10	18	38	19	103	8	17	36	18	102	12	19	39	19	0.001
GC	Carbosulfan																	68	15	18	74	18	0.01
GC	Carboxin		85	20	19	49	19	97	11	20	41	20	91	13	15	36	16	98	6	6	13	6	0.001
LC	Carboxin		95	8	11	25	12	99	8	19	38	19	93	9	18	40	19	92	11	18	39	18	0.001
GC	Chlordane	+	91	10	11	28	11	99	16	19	39	20	87	12	20	48	21	76	4	19	61	19	0.001
GC	Chlorfenapyr												97	19	20	42	21	93	12	20	45	21	0.005
GC	Chlorfenson		105	9	9	21	9	106	11	17	37	18	98	8	17	36	18	90	4	17	40	17	0.001
GC	Chlorfenvinphos	-																72	9	14	63	15	0.01
GC	Chlormephos																	80	8	20	58	21	0.01
GC	Chlorobenzilate		98	6	13	28	14	99	10	18	37	18	96	8	19	40	20	89	5	15	37	15	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Chlorpropham		101	7	6	13	6	107	14	12	29	13	107	8	13	31	14	100	4	8	17	9	0.001
GC	Chlorpyrifos	+	94	11	16	35	16	98	9	16	34	17	90	9	18	42	18	83	4	18	50	19	0.001
LC	Chlorpyrifos	+	102	12	16	33	16	93	10	9	24	10	93	16	19	41	20	92	6	11	27	11	0.001
GC	Chlorpyrifos-methyl		99	13	16	35	17	96	9	20	43	21	91	10	20	46	21	81	4	20	56	21	0.001
LC	Clethodim							108	11	14	33	14	104	11	15	32	15	99	16	17	34	17	0.002
GC	Clofentezine							91	24	23	52	24	81	12	11	44	12	74	14	14	59	15	0.002
LC	Clofentezine		90	11	20	47	21	92	16	19	42	20	99	6	13	27	14	97	7	13	27	13	0.001
GC	Clomazone		98	6	11	23	11	98	11	17	36	18	93	7	20	44	21	93	3	14	33	15	0.001
LC	Clothianidin		104	10	12	25	12	108	12	14	33	14	105	6	11	24	11	100	11	17	35	17	0.001
LC	Cyazofamid		101	6	11	23	11	101	9	9	18	9	100	5	8	17	8	100	7	11	23	12	0.001
GC	Cyflutrin		98	10	10	21	11	99	16	20	41	21	86	11	20	51	21	77	10	20	61	21	0.001
GC	Cyhalothrin-lambda												86	16	19	49	20	80	8	12	47	13	0.005
GC	Cypermethrin							105	15	15	33	16	89	12	18	44	19	76	10	20	63	21	0.002
LC	Cypermethrin		98	18	20	41	20	89	8	12	32	12	92	8	14	32	14	95	9	17	37	18	0.001
GC	Cyproconazole		97	14	21	45	22	107	12	14	32	15	98	7	9	19	10	92	7	9	23	9	0.001
GC	Cyprodinil		95	8	15	32	15	108	8	18	40	18	101	10	12	26	13	94	4	8	21	8	0.001
GC	DDTs	+	91	7	7	23	7	100	19	19	39	20	89	9	13	35	14	82	3	16	49	16	0.001
GC	DEET		100	13	16	33	16	102	11	15	32	16	102	9	10	21	10	97	3	3	9	3	0.001
LC	Deltamethrin_cis		87	9	8	31	9	92	10	13	32	14	96	8	12	27	13	96	6	16	34	16	0.001
GC	Demeton-S-methyl	+	95	16	15	33	15	96	13	17	37	18	94	9	13	29	13	92	3	5	18	5	0.001
LC	Demeton-S-methyl	+	99	10	16	33	16	102	10	17	35	17	96	10	19	40	19	97	12	20	42	21	0.001
LC	Demeton-S-methylsulfone		100	9	13	26	13	101	15	16	33	16	96	8	11	24	11	94	8	10	24	10	0.001
GC	Diazinon	-																86	6	18	46	19	0.01
LC	Diazinon	-	97	7	8	18	9	100	9	8	17	9	99	5	6	12	6	97	8	8	17	8	0.001
GC	Dichlofluanid																						NV
LC	Dichlofluanid																						NV

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
LC	Dichlorprop		124	13	17	61	18	112	12	15	39	15	106	12	13	30	14	103	12	11	24	11	0.001
GC	Dichlorvos	-	97	19	17	37	18	95	12	11	26	12	96	8	20	42	20	98	7	16	33	16	0.001
LC	Dichlorvos	-						105	17	18	37	18	101	10	12	24	12	103	12	16	33	16	0.002
GC	Dicloran							86	18	17	45	18	103	8	12	26	12	104	4	15	33	16	0.002
GC	Dicofol	+	96	9	11	24	12	104	13	15	32	15	96	7	20	42	20	97	5	16	34	17	0.001
GC	Dieldrin	-											93	15	14	33	15	84	12	20	53	21	0.005
GC	Difenoconazole		100	9	15	31	15	103	9	15	31	15	95	6	19	40	19	92	4	16	36	16	0.001
LC	Difenoconazole		106	8	11	26	12	105	9	13	28	13	108	7	14	32	14	100	14	15	30	15	0.001
LC	Diflubenzuron							112	18	18	45	19	99	11	11	22	11	96	9	9	20	9	0.002
LC	Dimethoate	+	99	5	8	18	9	101	8	10	21	10	98	6	10	22	11	94	8	15	32	15	0.001
GC	Dimethomorph		92	8	10	26	11	97	9	15	32	16	94	7	20	42	20	90	2	19	44	20	0.001
LC	Dinoterb							94	13	14	32	15	109	16	16	37	16	111	14	13	34	13	0.002
GC	Diphenylamine							68	14	15	71	15	88	13	12	35	12	97	6	7	16	7	0.002
GC	Disulfoton	-																79	8	20	59	21	0.01
LC	Disulfoton	-						107	16	19	42	20	94	15	19	41	20	106	15	18	39	18	0.002
LC	Disulfoton sulfone		102	12	12	24	12	108	13	14	32	14	104	6	14	30	14	103	10	16	34	17	0.001
LC	Disulfoton sulfoxide		101	6	6	12	6	108	7	9	25	10	106	7	13	29	13	102	7	11	23	11	0.001
LC	Ditalimphos		91	7	7	23	7	91	7	13	32	13	90	5	10	30	11	89	5	11	31	11	0.001
LC	DMF		107	7	11	26	11	110	5	8	27	8	108	4	11	28	12	106	7	14	31	14	0.001
LC	DMPF							111	16	20	47	21	96	13	26	54	27	101	12	15	30	15	0.002
LC	DMST		106	8	13	30	14	114	12	20	50	21	108	11	19	42	19	106	13	20	42	20	0.001
LC	DNOC																	103	12	20	41	20	0.01
GC	Endosulfan-alpha																						NV
GC	Endosulfan-beta		95	21	19	40	20	101	16	16	34	17	98	14	20	41	20	89	8	17	41	17	0.001
GC	Endosulfan-sulfate																						NV
GC	Endrin	-	99	18	16	34	17	93	17	23	49	24	81	13	27	67	27	77	7	16	56	16	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	EPN												90	11	11	29	11	89	4	12	33	13	0.005
GC	Epoxiconazole		98	10	12	25	12	105	11	15	33	16	99	7	20	42	21	97	3	15	32	16	0.001
LC	Epoxiconazole		107	9	10	25	11	94	10	11	26	11	99	6	11	22	11	97	7	8	17	8	0.001
LC	Ethiofencarb		100	8	11	23	11	103	8	18	37	18	98	6	16	32	16	97	9	15	32	16	0.001
GC	Ethion	+	87	8	13	38	14	94	10	17	37	17	87	8	13	38	14	82	4	14	46	14	0.001
LC	Ethion	+	98	9	13	26	13	98	9	10	21	10	97	11	16	34	17	98	8	15	31	15	0.001
GC	Ethoprophos	+	109	11	11	30	12	108	12	15	36	16	105	6	6	16	6	101	4	4	8	4	0.001
LC	Ethoprophos	+	93	9	8	21	8	93	9	10	25	10	97	7	7	16	7	96	7	6	15	7	0.001
GC	Ethoxyquin							36	19	24	138	25	29	20	33	157	34	28	12	20	150	20	0.002
GC	Etofenprox		82	11	15	47	15	88	12	22	53	23	82	12	22	58	23	76	4	18	61	19	0.001
LC	Etofenprox		83	11	11	41	12	89	8	14	36	14	88	6	15	40	16	88	8	20	47	20	0.001
LC	Fenamiphos	+	100	7	7	14	7	99	5	6	12	6	98	7	8	16	8	97	6	8	19	9	0.001
LC	Fenamiphos-sulfone		104	7	9	20	9	112	11	18	43	18	105	13	18	39	19	105	14	20	42	20	0.001
LC	Fenamiphos-sulfoxide												103	19	20	41	20	100	13	14	28	14	0.005
GC	Fenarimol		98	12	17	35	18	103	8	15	31	15	95	7	20	42	20	94	3	14	31	14	0.001
GC	Fenazaquin		98	8	12	25	12	92	12	14	34	15	77	9	18	59	19	76	3	13	55	14	0.001
LC	Fenazaquin		90	7	10	28	10	91	7	12	29	12	89	7	13	35	14	88	5	13	36	14	0.001
GC	Fenbuconazole		99	7	13	27	14	104	9	15	31	15	100	7	20	40	20	96	3	17	37	18	0.001
LC	Fenbuconazole		102	8	12	24	12	102	6	7	14	7	100	6	9	19	10	101	8	11	22	11	0.001
LC	Fenhexamid							101	16	15	31	15	92	5	12	29	12	97	10	11	24	12	0.002
GC	Fenitrothion																	80	8	19	56	20	0.01
GC	Fenoxycarb		92	17	16	37	17	109	8	8	24	8	110	6	6	24	6	104	4	4	12	4	0.001
LC	Fenoxycarb		100	6	7	14	7	100	8	8	17	8	99	6	6	13	7	97	6	7	15	7	0.001
GC	Fenpropathrin												95	12	14	32	15	88	8	11	34	11	0.005
LC	Fenpropidin		89	10	10	31	10	95	10	15	33	16	89	6	14	36	14	87	9	16	42	17	0.001
GC	Fenpropimorph		81	13	14	48	15	89	11	18	43	19	92	11	16	37	17	81	6	18	53	19	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Fenson		100	8	16	33	16	105	11	18	39	19	97	9	20	41	20	91	4	17	39	18	0.001
GC	Fensulfothion	+	97	16	20	42	21	91	18	21	47	22	84	11	30	69	31	83	11	32	74	33	0.001
LC	Fensulfothion	+	98	7	7	15	7	102	8	11	23	11	100	7	9	19	10	100	7	7	15	7	0.001
GC	Fenthion		103	9	13	27	13	102	11	17	35	18	97	8	14	30	15	90	4	12	32	12	0.001
LC	Fenthion		102	9	10	22	11	98	9	9	19	9	99	4	8	16	8	98	5	7	14	7	0.001
LC	Fenthion-oxon		95	7	6	16	7	103	7	11	22	11	102	5	7	15	7	102	9	9	19	10	0.001
LC	Fenthion-oxon-sulfone		102	9	14	28	14	101	9	9	19	10	98	6	8	18	9	95	7	11	24	11	0.001
LC	Fenthion-oxon-sulfoxide		98	6	9	19	9	101	10	11	22	11	100	6	9	18	9	95	11	15	31	15	0.001
LC	Fenthion-sulfone		105	11	10	24	11	112	14	16	40	16	103	10	16	32	16	101	11	17	34	17	0.001
LC	Fenthion-sulfoxide		105	10	12	26	12	109	10	15	35	15	105	10	15	33	16	102	12	17	35	17	0.001
GC	Fenvalerate																						NV
LC	Fipronil	-	104	20	19	39	19	105	14	14	30	14	112	16	15	39	15	111	12	11	32	11	0.001
LC	Fipronil-desulfinyl		98	8	7	16	8	100	8	8	16	8	100	6	7	15	7	101	9	9	19	9	0.001
LC	Fipronil-sulfide																	92	17	19	43	20	0.01
LC	Fipronil-sulfone																						NV
GC	Fluazifop-P-butyl		96	10	11	25	12	106	13	20	44	21	96	10	12	27	13	91	6	12	31	13	0.001
LC	Fluazifop-p-butyl		98	8	9	18	9	99	9	8	17	9	94	9	12	27	12	95	9	11	25	11	0.001
GC	Fludioxonil		86	15	16	42	16	83	14	21	55	22	80	13	19	57	20	80	16	20	58	21	0.001
GC	Flufenoxuron																						NV
LC	Fluoxastrobin		98	7	7	14	7	99	6	7	14	7	96	3	5	13	5	96	6	7	16	7	0.001
GC	Fluquinconazole	+	98	8	10	22	11	96	10	18	39	19	94	7	20	42	20	91	4	19	42	19	0.001
LC	Fluquinconazole	+	91	10	10	26	10	95	6	9	21	9	99	5	9	18	9	98	6	8	17	8	0.001
GC	Flusilazole	+	101	10	18	37	18	104	10	11	24	11	98	8	20	42	21	93	6	15	35	16	0.001
LC	Flusilazole	+	99	5	6	12	6	99	6	5	11	6	98	5	6	14	6	98	5	5	11	5	0.001
GC	Flutriafol		91	13	18	41	19	100	10	18	36	18	104	7	22	46	23	103	6	20	42	21	0.001
GC	Fluvalinate-tau																						NV

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Formothion							94	6	7	20	8	98	11	14	30	15	94	8	19	41	19	0.002
LC	Fosthiazate		99	7	7	15	8	105	8	12	25	12	100	5	10	21	11	99	9	12	24	12	0.001
GC	HCH-alpha							87	12	17	44	18	94	9	9	22	9	101	7	14	29	14	0.002
GC	HCH-beta							103	9	16	33	16	109	8	19	44	20	106	4	23	50	24	0.002
GC	Heptachlorepoxide-cis		96	11	13	28	14	102	11	17	35	17	94	14	14	30	14	89	8	20	48	21	0.001
GC	Heptachlorepoxide-trans												87	10	21	50	21	88	10	20	48	21	0.005
GC	Heptenophos		89	8	10	29	10	83	9	18	50	18	84	9	10	38	11	80	8	7	43	8	0.001
LC	Heptenophos		99	7	8	16	8	103	9	9	18	9	99	7	7	14	7	100	7	7	15	7	0.001
GC	Hexachlorobenzene	-																					NV
GC	Hexaconazole																	97	11	16	34	17	0.01
LC	Hexaconazole		100	8	20	41	21	102	17	16	33	16	101	9	12	25	13	105	7	7	18	7	0.001
GC	Hexythiazox							96	14	16	34	16	96	10	10	22	10	96	7	10	22	10	0.002
LC	Hexythiazox		93	7	9	24	10	95	6	9	21	9	96	9	14	30	15	96	4	11	24	11	0.001
LC	Imazalil		99	11	12	24	12	102	9	10	22	11	100	8	13	27	14	97	11	18	37	18	0.001
LC	Imidacloprid		107	9	13	31	14	109	12	13	31	13	109	7	9	26	10	101	11	14	30	15	0.001
GC	Indoxacarb																						NV
LC	Indoxacarb		100	8	8	17	8	119	8	15	49	16	110	10	16	38	16	101	14	14	29	14	0.001
LC	Iodosulfuron-methyl-sodium		101	5	5	11	5	111	8	8	27	8	112	5	6	28	6	112	7	7	28	7	0.001
GC	Iprodione																	61	19	17	86	17	0.01
LC	Iprodione		106	16	20	42	20	104	16	19	40	20	98	10	12	24	12	101	9	14	28	14	0.001
GC	Iprovalicarb		115	12	19	50	20	122	16	17	56	18	118	12	14	46	15	107	7	11	26	11	0.001
LC	Iprovalicarb		97	8	8	18	8	97	8	9	20	9	96	7	9	21	9	94	7	10	23	10	0.001
GC	Isofenphos-methyl		109	5	7	23	7	111	11	13	36	14	108	9	10	26	11	100	3	6	13	6	0.001
GC	Isoprothiolane		106	12	14	31	14	110	8	10	29	11	109	6	5	20	6	104	4	5	13	5	0.001
LC	Isoprothiolane		98	5	5	10	5	100	5	6	13	6	97	5	6	13	6	98	5	7	14	7	0.001
LC	Isoproturon		96	6	6	14	6	100	9	9	18	9	98	5	7	15	7	98	7	8	16	8	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Jodofenfos		92	14	16	38	17	93	13	28	59	29	83	5	20	54	21	77	4	24	68	25	0.001
GC	Kresoxim-methyl		106	11	13	28	13	101	9	15	31	15	95	9	20	43	21	89	4	17	41	17	0.001
GC	Lindane		85	10	10	37	11	95	12	11	24	11	97	9	12	25	12	95	7	17	36	17	0.001
GC	Linuron												103	19	18	38	19	97	12	11	24	12	0.005
LC	Linuron							88	14	21	49	21	99	7	15	30	15	98	8	9	18	9	0.002
LC	Lufenuron																						NV
LC	Malaoxon		103	6	14	30	15	107	11	18	39	18	103	9	17	36	18	100	13	17	36	18	0.001
LC	Malathion		99	8	8	16	8	101	7	8	16	8	99	6	7	14	7	99	6	7	15	7	0.001
LC	Mecarbam	+	99	5	5	11	6	101	6	6	13	6	97	4	6	13	6	97	5	7	16	7	0.001
LC	Mepanipyrim							94	15	20	42	20	95	9	13	28	13	92	7	9	25	9	0.002
LC	Metaflumizone		100	18	17	34	17	101	19	18	38	19	116	19	18	48	19	112	17	18	43	18	0.001
GC	Metalaxyl							107	11	14	33	15	113	9	9	32	9	109	3	6	22	6	0.002
LC	Metalaxyl		96	7	7	16	7	101	8	8	16	8	99	6	7	14	7	98	6	6	13	6	0.001
LC	Metconazole		105	10	11	25	11	103	11	12	24	12	102	5	7	14	7	99	4	4	8	4	0.001
GC	Methacrifos							98	20	19	40	20	98	13	14	29	15	105	9	14	31	15	0.002
LC	Methamidophos	+	84	8	9	37	9	84	16	16	45	16	81	14	24	63	25	87	5	6	30	6	0.001
GC	Methidathion	+	90	11	20	46	21	92	13	26	56	27	86	12	19	48	20	83	7	14	44	14	0.001
LC	Methiocarb		111	11	16	40	17	107	8	8	21	8	103	8	10	22	10	105	7	9	21	9	0.001
LC	Methiocarb-sulfone		95	9	11	26	12	97	11	14	29	14	93	8	13	31	14	90	7	14	35	14	0.001
LC	Methiocarb-sulfoxide		100	7	9	18	9	102	10	12	25	13	101	5	8	17	9	97	9	14	29	14	0.001
LC	Methomyl	+	115	15	14	41	15	108	12	14	33	15	98	7	8	16	8	96	10	9	21	10	0.001
LC	Methoxyfenozide		97	7	7	16	7	100	7	10	20	10	95	8	11	24	11	95	7	8	19	8	0.001
GC	Metribuzin		96	11	13	28	13	101	11	15	31	15	98	7	17	35	17	99	4	14	30	15	0.001
LC	Metsulfuron-methyl		106	6	14	30	14	113	7	15	39	15	108	7	15	36	16	105	9	18	38	18	0.001
GC	Mevinphos		80	11	15	51	16	84	15	18	49	19	86	14	13	39	14	81	7	15	50	16	0.001
LC	Mevinphos		98	5	8	16	8	100	10	12	24	12	99	6	9	20	10	94	9	13	30	14	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
LC	Monocrotophos	-																					NV
LC	Monolinuron		101	7	8	16	8	106	8	16	35	17	101	7	16	33	16	102	10	18	37	18	0.001
GC	Myclobutanil		98	13	17	34	17	106	9	16	34	16	100	5	21	43	22	98	4	18	38	19	0.001
GC	Nitrofen	+	79	16	21	60	22	75	20	19	63	19	87	15	20	48	21	88	7	23	54	24	0.001
GC	Nuarimol		105	11	19	42	20	107	13	17	38	18	110	10	10	29	10	110	8	11	31	11	0.001
LC	Ofurace		105	5	12	27	13	112	12	17	42	17	104	11	16	33	16	102	12	17	35	17	0.001
LC	Omethoate	+	95	10	12	26	12	98	13	12	26	13	94	7	19	41	19	93	9	14	32	14	0.001
GC	Oxadixyl		99	6	6	13	6	109	9	14	34	15	111	6	6	25	6	104	3	3	9	3	0.001
LC	Oxamyl	+	101	8	8	16	8	106	9	9	23	10	99	8	11	22	11	99	9	9	19	9	0.001
LC	Oxycarboxin		100	6	8	16	8	103	7	8	18	8	101	5	8	17	8	99	6	9	19	9	0.001
GC	Oxychlordane																						NV
LC	Oxydemeton-methyl	+	96	6	8	18	8	96	9	9	21	10	91	8	12	30	12	89	8	8	27	8	0.001
GC	Paclobutrazol																	97	5	17	35	17	0.01
LC	Paraoxon-methyl																	100	17	19	40	20	0.01
GC	Parathion	-											90	11	13	34	13	90	6	6	23	7	0.005
GC	Parathion-methyl		103	11	20	42	21	117	16	37	85	39	126	7	46	110	48	125	4	52	118	54	0.001
GC	Penconazole		94	14	17	37	18	101	15	18	38	19	93	9	19	41	20	91	4	15	36	16	0.001
LC	Penconazole		100	14	14	29	15	106	9	9	22	9	103	8	9	21	10	103	7	7	16	7	0.001
GC	Pencycuron		100	6	11	24	12	98	10	17	36	18	93	7	20	44	21	93	3	15	34	15	0.001
LC	Pencycuron		101	8	8	18	9	105	8	8	18	8	104	4	6	15	6	102	9	8	18	9	0.001
GC	Pendimethalin																	66	14	14	74	14	0.01
LC	Pendimethalin		100	9	10	21	10	96	11	11	25	12	94	13	16	35	16	94	5	12	26	12	0.001
GC	Permethrin																						NV
GC	Phenthoate		96	10	17	37	18	94	12	21	45	22	94	8	17	37	17	87	5	15	41	16	0.001
GC	Phosalone																						NV
GC	Phosmet		116	8	7	35	7	100	20	28	58	29	79	15	24	66	25	68	14	20	76	21	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
LC	Phosmet		100	10	12	25	13	105	14	16	35	16	106	8	12	27	12	107	17	17	37	17	0.001
LC	Phosmet-oxon																						NV
LC	Phosphamidon	+	98	7	10	21	10	104	8	9	21	10	103	6	13	28	14	104	6	14	29	14	0.001
LC	Phoxim		107	9	10	26	11	105	7	9	22	9	107	7	7	20	7	103	6	6	14	6	0.001
GC	Pirimicarb		96	9	10	22	10	95	9	17	36	17	93	7	20	44	21	90	4	17	40	17	0.001
LC	Pirimicarb		101	4	7	13	7	101	5	6	13	6	101	4	8	16	8	97	6	11	24	11	0.001
GC	Pirimicarb-desmethyl												76	8	20	64	21	83	6	21	55	21	0.005
GC	Pirimiphos-ethyl		100	16	17	36	18	101	9	16	33	16	95	8	16	35	16	88	4	15	39	15	0.001
GC	Pirimiphos-methyl		106	14	13	30	14	103	11	15	31	15	96	5	11	24	11	89	4	13	34	13	0.001
LC	Pirimiphos-methyl		106	5	8	20	8	106	8	10	25	11	104	5	10	22	10	102	6	9	20	10	0.001
GC	Prochloraz							117	7	18	51	19	96	9	9	20	9	86	7	7	32	7	0.002
LC	Prochloraz		102	7	6	14	7	103	6	6	14	6	101	6	7	14	7	99	6	7	14	7	0.001
GC	Procymidone		96	11	11	25	12	98	11	22	45	22	89	12	27	61	28	88	3	16	41	17	0.001
GC	Profenofos																						NV
LC	Propamocarb		91	6	6	22	6	95	9	9	20	9	91	9	10	26	10	91	5	7	23	7	0.001
LC	Propargite		94	6	9	22	9	97	6	8	18	9	96	8	13	28	13	93	6	13	29	13	0.001
LC	Propiconazole		89	11	10	30	10	96	8	8	17	8	100	6	6	13	6	99	8	8	16	8	0.001
GC	Propoxur		84	13	19	50	19	74	22	27	76	28	71	24	22	74	23	62	18	18	85	19	0.001
LC	Propoxur		104	5	12	26	12	108	10	16	36	16	106	7	16	35	16	104	10	16	34	17	0.001
GC	Propyzamide		100	5	7	14	7	101	9	15	31	16	93	7	20	43	21	92	3	16	36	16	0.001
LC	Propyzamide		97	6	6	14	6	98	6	8	18	9	98	4	7	15	7	96	5	7	17	8	0.001
LC	Prosulfocarb		114	18	20	50	21	108	16	17	39	18	109	16	19	44	20	102	16	18	38	19	0.001
LC	Prosulfuron		109	10	10	27	10	105	9	10	23	11	102	7	13	28	14	103	6	12	25	12	0.001
LC	Prothioconazole-desthio		115	13	20	51	21	103	11	12	25	12	103	7	6	15	7	102	7	10	20	10	0.001
GC	Prothiofos		99	10	10	20	10	89	15	19	44	19	81	16	17	52	18	79	6	16	54	17	0.001
LC	Pymetrozine		65	8	13	75	13	66	10	12	71	12	68	8	11	68	11	66	11	14	75	15	0.001

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
LC	Pyraclostrobin		103	7	6	15	7	106	8	8	21	8	105	6	8	19	8	104	6	8	17	8	0.001
GC	Pyrazophos		99	11	20	42	21	91	9	17	40	18	88	11	20	48	21	81	7	15	49	15	0.001
GC	Pyridaben		64	15	14	78	15	73	12	30	84	32	69	11	14	69	14	68	4	8	65	9	0.001
GC	Pyridaphenthion		96	16	15	31	15	90	15	18	42	19	93	8	14	32	15	90	9	11	30	11	0.001
LC	Pyridate		105	9	11	24	11	111	10	20	46	20	115	12	14	40	14	120	7	19	56	19	0.001
GC	Pyrimethanil		98	7	14	30	15	97	8	19	40	20	92	7	20	44	21	85	3	15	43	16	0.001
LC	Pyrimethanil		106	8	8	20	8	109	8	13	31	13	101	6	9	18	9	100	7	9	19	9	0.001
GC	Pyriproxyfen		86	12	16	43	17	95	10	18	39	18	88	7	20	48	21	84	4	17	47	18	0.001
LC	Pyriproxyfen		92	7	10	26	10	95	7	8	19	9	92	10	16	38	17	93	8	13	30	13	0.001
GC	Quinoxyfen		98	3	17	36	18	97	10	16	33	16	92	10	16	37	17	85	4	16	45	17	0.001
LC	Quinoxyfen		93	10	11	27	12	96	8	9	21	10	96	11	12	25	12	95	6	8	20	9	0.001
GC	Simazine							95	9	14	30	14	100	8	20	41	21	93	4	17	38	18	0.002
LC	Simazine		106	8	10	23	10	105	12	17	35	17	101	8	16	33	17	102	10	19	39	20	0.001
LC	Spinosad_A		94	11	10	24	10	90	12	14	36	15	92	10	16	37	17	93	9	13	30	13	0.001
LC	Spinosad_D		101	19	18	37	19	99	14	16	33	16	103	17	19	39	19	95	13	16	34	16	0.001
LC	Spirodiclofen		93	7	6	19	6	93	8	9	24	10	92	8	12	28	12	94	7	15	34	16	0.001
LC	Spiroxamine		81	11	11	44	11	89	10	12	34	13	87	5	12	36	13	85	9	17	45	17	0.001
GC	Tebuconazole		100	13	18	37	19	109	9	14	33	14	103	8	20	42	21	99	5	18	38	19	0.001
LC	Tebufenozide		101	11	11	23	11	99	10	10	20	10	100	10	10	20	10	96	10	11	24	11	0.001
GC	Tebufenpyrad		97	9	11	23	11	98	9	16	33	16	87	8	19	46	19	82	4	16	49	17	0.001
LC	Tebufenpyrad		100	10	10	20	10	102	12	12	25	12	99	13	15	30	15	100	11	11	23	11	0.001
GC	Tecnazene																						NV
LC	Teflubenzuron		100	18	20	40	20	100	11	12	25	13	102	13	14	30	15	99	12	11	23	12	0.001
GC	Tefluthrin		85	8	18	47	18	87	11	27	62	28	76	12	31	80	32	77	4	20	61	20	0.001
GC	TEPP																						NV
GC	Terbufos	-											85	16	20	52	21	84	9	21	55	22	0.005

			Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound		**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
LC	Terbufos	-																87	12	12	35	12	0.01
GC	Tetraconazole		100	12	15	31	15	108	9	14	33	14	99	13	20	41	20	97	5	14	30	15	0.001
GC	Tetradifon		102	14	13	27	13	99	13	18	37	18	93	8	12	30	13	86	6	20	51	21	0.001
LC	Thiabendazole		97	6	10	22	11	98	11	11	23	11	97	8	11	23	11	92	10	12	29	12	0.001
LC	Thiacloprid		97	6	9	19	9	102	8	8	18	8	99	6	11	22	11	96	7	11	23	11	0.001
LC	Thiamethoxam		104	6	8	19	9	102	12	14	28	14	99	7	10	20	10	95	10	10	22	10	0.001
LC	Thiodicarb		96	8	10	23	11	108	10	18	42	19	102	7	20	42	21	101	12	28	59	30	0.001
GC	Thiometon							68	17	19	76	20	82	10	14	47	15	96	5	13	28	14	0.002
LC	Thiometon							118	20	20	56	21	101	13	16	34	17	98	11	14	30	15	0.002
LC	Thiophanate-methyl																						NV
GC	Tolclofos-methyl		100	7	9	18	9	99	8	17	36	18	92	5	18	42	19	89	3	21	48	22	0.001
GC	Tolyfluanid																						NV
LC	Tolylfluanid																						NV
GC	Triadimefon							111	12	15	39	16	108	10	10	27	10	105	5	9	21	9	0.002
LC	Triadimenol		99	8	8	17	8	102	8	8	17	8	100	4	5	10	5	96	4	5	14	6	0.001
GC	Triallate		86	14	13	39	13	91	13	24	54	25	86	9	21	52	22	84	4	28	66	29	0.001
LC	Triallate		102	17	19	39	20	101	11	10	21	11	98	11	16	32	16	95	12	15	33	16	0.001
GC	Triazophos	-											93	12	21	46	22	86	8	13	39	13	0.005
LC	Triazophos	+	96	4	4	12	4	97	7	6	15	7	97	5	6	13	6	95	5	6	16	6	0.001
GC	Trichlorfon							96	12	11	24	11	100	9	18	37	18	101	5	15	30	15	0.002
LC	Trichlorfon							112	8	19	45	19	102	9	12	24	12	96	9	11	24	11	0.002
GC	Tricyclazole		97	14	13	29	14	108	12	14	33	14	107	8	10	24	10	101	5	7	14	7	0.001
LC	Tricyclazole		95	7	9	20	9	102	6	8	16	8	99	5	10	20	10	97	6	12	25	12	0.001
GC	Trifloxystrobin		113	20	19	48	20	103	18	19	40	20	110	7	6	24	6	100	5	5	11	6	0.001
LC	Trifloxystrobin		106	5	10	23	10	109	7	10	29	11	109	10	15	35	15	100	10	11	23	12	0.001
LC	Triflumuron		100	6	6	12	6	102	7	7	14	7	102	5	7	15	7	99	6	7	14	7	0.001

		Spike level 0.001 mg/kg					Spike level 0.002 mg/kg					Spike level 0.005 mg/kg					Spike level 0.01 mg/kg					
	Compound	**Recovery %**	**RSD_r %**	**RSD_R %**	**U %**	**Cu %**	**Recovery %**	**RSD_r %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSD_r, %**	**RSD_R, %**	**U %**	**Cu %**	**Recovery %**	**RSDr %**	**RSD_R %**	**U %**	**Cu %**	**LOQ**
GC	Trifluralin																					NV
GC	Triticonazole											71	12	19	71	20	64	9	14	77	15	0.005
LC	Triticonazole	91	9	12	31	13	95	6	10	22	10	98	6	8	17	8	97	6	8	17	8	0.001
LC	Vamidothion	103	10	10	21	10	105	13	13	29	13	101	9	11	23	11	97	10	15	32	16	0.001
GC	Vinclozolin	91	15	18	42	19	102	14	15	31	16	97	7	17	36	18	91	4	14	33	14	0.001
LC	Zoxamide	104	6	7	16	7	105	7	9	20	9	104	4	7	16	7	103	5	6	14	6	0.001

Appendix 3 Pesticides on the EFSA list

Pesticide	Calculated MRL in mg/kg to be below an ADI of 0.0026 mg/kg bw	Achieved LOQ, mg/kg	LOQ acceptable?
Aldrin	0.0004	0.001	NO
Cadusafos	0.0015	0.001	YES
Carbofuran	0.0006	0.001	NO
Carbophenothion	0.0019		NA
Chlordane	0.0019	0.001	YES
Chlorfenvinphos	0.0019	0.01	NO
Chlorpyrifos	0.0038	0.001	YES
Cyhalothrin-gamma	0.0046		NA
Cyhalothrin-lambda	0.0096	0.005	YES
DDTs	0.0077	0.001	YES
Demeton-S-methyl	0.0012	0.001	YES
Diazinon	0.0008	0.01	NO
Dichlorvos	0.0003	0.001	NO
Dicofol	0.0077	0.001	YES
Dieldrin	0.0004	0.005	NO
Dimethoate	0.0038	0.001	YES
Dioxathion	0.0058		NA
Disulfoton	0.0012	0.01	NO
Endrin	0.0008	0.001	NO
Ethion	0.0077	0.001	YES
Ethoprophos	0.0015	0.001	YES
Fenamiphos	0.0031	0.001	YES
Fensulfothion	0.0012	0.001	YES
Fipronil	0.0008	0.001	NO
Fipronil desulfinyl	0.0008	0.001	NO
Fipronil-sulfide	0.0008	0.01	NO
Fluometuron	0.0019		NA
Fluquinconazole	0.0077	0.001	YES
Flusilazole	0.0077	0.001	YES
Heptachlor	0.0004		NA
Hexachlorobenzene	0.0023	Not validated	NO
Isofenphos	0.0038		NA
Mecarbam	0.0077	0.001	YES
Methamidophos	0.0038	0.001	YES
Methidathion	0.0038	0.001	YES
Methomyl	0.0096	0.001	YES
Monocrotophos	0.0023	Not Validated	NO
Nitrofen	0.0011	0.001	YES

Pesticide	Calculated MRL in mg/kg to be below an ADI of 0.0026 mg/kg bw	Achieved LOQ, mg/kg	LOQ acceptable?
Omethoate	0.0012	0.001	YES
Oxamyl	0.0038	0.001	YES
Oxydemeton-methyl	0.0012	0.001	YES
Parathion	0.0023	0.005	NO
Phorate	0.0027		NA
Phosphamidon	0.0019	0.001	YES
Quinoclamine	0.0077		NA
Tembotrione	0.0015		NA
terbufos	0.0023	0.005	NO
Topramezone	0.0038		NA
Triazophos	0.0038	0.001	YES
Triazoxide	0.0008		NA
SRM compounds			
1-Methyl-cyclopropene	SRM		
Abamectin	SRM		
Amitrole	SRM		
Chloropicrin	SRM		
Cyanamide	SRM		
Diclofop	SRM		
Diquat	SRM		
Emamectin	SRM		
Fenthiosulf	SRM		
Fentin acetate	SRM		
Fentin hydroxide	SRM		
Haloxyfop	SRM		
Haloxyfop-P	SRM		
Metam	SRM		
Methyl bromide	SRM		
Nicotine	SRM		
Sulcotrione	SRM		
Propineb	SRM		

Appendix 4: Principles of the QuEChERS method for cereal extraction

QuEChERS for cereals (FP417)

Weigh 5 g (±0.05 g) of flour into a 50 ml single use centrifuge tube (red cap). Add internal standard and/or spike standard (maximum 25 µl)

Add a ceramic homogenizer and 10 g of cold water and shake briefly

Add 10 ml acetonitrile and shake vigorously by hand for 1 min. (1. extraction)

Add the prepared mixture of 4 g $MgSO_4$, 1 g NaCl, 1 g Na_3 citrate dihydrate and 0.5 g Na_2H cirate sesquihydrate. Shake for a few seconds after each addition to prevent lumps.

Shake vigorously for 1 min. (2. Extraction with phase separation)

Centrifuge for 10 min at 4500 rpm

Transfer at least 8 ml of the extract to a 15 ml single use centrifuge tube and store in the freezer (-80˚C for 1 hour or over night). When the extract are almost thawed (i.e. About -40 ˚C) centrifugate (should be cold 5 C) for 5 min. at 4500 rpm.

Transfer 6 ml of the cold extract to a 15 ml single use centrifuge tube containing 150 mg PSA and 900 mg $MgSO_4$. Close the tube and shake vigorously for 30 seconds.

Centrifuge for 5 min. at 4500 rpm

Transfer 4 ml of the extract to a 15 ml single use centrifuge tube. Add 40 µl of 5% formic acid solution in acetonitrile (10 µl/ml extract). Dilute the extract 1:1 with acetonitrile

Transfer the final extract into auto sampler vials and analyse by GC and LC.