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Method of Test for Pesticide Residues in Livestock and Poultry Products- Multiresidue Analysis

1. Scope

This method is applicable for the determination of 129 pesticide residues in muscle, viscera and eggs of poultry and livestock products.

2. Method

After preparation of the sample solution by the QuEChERS method (Quick, Easy, Cheap, Effective, Rugged, Safe), pesticides are determined by liguid chromatography/tandem mass spectrometry (LC-MS/MS) and gas chromatography/tandem mass spectrometry (GC-MS/MS).

2.1. Equipment

2.1.1. Liquid chromatograph/tandem mass spectrometer.

2.1.1.1. Ion source: electrospray ionization, ESI.

2.1.1.2. Column: CORTECS UPLC, C18, 1.6 μm, 2.1 mm i.d. × 10 cm, or an equivalent product.

2.1.1.3. Guard column: CORTECS UPLC, C18, 1.6 μm, 2.1 mm i.d. × 5 mm, or an equivalent product.

2.1.2. Gas chromatograph/tandem mass spectrometer.

2.1.2.1. Ion source: electron ionization, EI.

2.1.2.2. Column: HP-5MS UI capillary column, 0.25 μm, 0.25 mm × 30 m, or an equivalent product.

2.1.3. Blender.

2.1.4. Grinder.

2.1.5. High speed dispersing device: SPEX SamplePrep 2010 GenoGrinder® ,1000 rpm, or an equivalent product.

2.1.6. Centrifuge: centrifugal force > 4000 ×g, temperature control <15°C.

2.1.7. Nitrogen evaporator.

2.2. Chemicals

Glacial acetic acid, reagent grade;

Formic acid, reagent grade;

Ammonium acetate, reagent grade;

n-Hexane, residue grade;

Acetone, residue grade;

Acetonitrile, HPLC grade;

Methanol, HPLC grade;

Sodium acetate anhydrous, AR grade;
Magnesium sulfate anhydrous, AR grade;
Primary secondary amine (PSA), AR grade;
Octadecysilane end-capped (C18 EC), AR grade;
Graphitized carbon black (GCB), AR grade;
Deionized water, resistivity ≥ 18 MΩ·cm (at 25°C);
Acephate and other pesticides listed in the attached tables, reference standards;
Triphenylphosphate, internal standard.

2.3. Apparatus

2.3.1. Centrifuge tube: 15 mL and 50 mL, PP.

2.3.2. Membrane filter: 0.22-μm, PTFE.

2.3.3. Volumetric flask: 25 mL and 50 mL, amber.

2.3.4. Ceramic homogenizer(note 1): Bond Elut QuEChERS P/N 5982-9313, or an equivalent product.

2.3.5. Extraction powder(note 2): containing 4 g of magnesium sulfate anhydrous and 1 g of sodium acetate anhydrous.

2.3.6. Clean-up centrifuge tube(note 2): containing 375 mg of PSA, 750 g of magnesium sulfate anhydrous, 250 mg of C18EC and 45 mg of GCB, 5 mL.

Note 1: Ceramic homogenizer can be used depending on the viscosity of the sample.

Note 2: Commercial extraction/clean-up kit can be used as needed.

2.4. Mobile phase

2.4.1. Solvent A

Dissolve and dilute 0.4 g of ammonium acetate with deionized water to 1000 mL. Add 1 mL of formic acid, mix well, and filter with a membrane filter.

2.4.2. Solvent B

Dissolve and dilute 0.4 g of ammonium acetate with methanol to 1000 mL, and filter with a membrane filter.

2.5. Internal standard solution preparation

Transfer about 40 mg of triphenylphosphate internal standard accurately weighed into a 50-mL volumetric flask, dissolve and dilute with methanol to volume as the internal standard stock solutions. Store at -18℃ in the

dark.

2.5.1. Dilute appropriate volume of the internal standard stock solution with methanol to 50 μg/mL as the internal standard solution for sample solution preparation in section 2.8.

2.5.2. Dilute appropriate volume of the internal standard stock solution with methanol to 5 μg/mL as the internal standard solution for analysis of LC-MS/MS in section 2.9.1.

2.5.3. Dilute appropriate volume of the internal standard stock solution with acetone to 5 μg/mL as the internal standard solution for analysis of GC-MS/MS in section 2.9.2.

2.6. Reagents

2.6.1. 1% Acetic acid in acetonitrile

Mix 10 mL of glacial acetic acid and 990 mL of acetonitrile.

2.6.2. Acetone: *n*-hexane (1:1, v/v)

Mix acetone and *n*-hexane at the ratio of 1:1 (v/v).

2.7. Standard solution preparation

2.7.1. Accurately weigh about 25 mg of pesticide reference standards to each 25-mL volumetric flask, dissolve and dilute to volume with acetonitrile as the standard stock solutions. Store at -18°C in the dark. Mix appropriate volume of each standard stock solution, and dilute with methanol to 1 μg/mL as the standard solution for analysis of LC-MS/MS in section 2.9.1.

2.7.2. Accurately weigh about 25 mg of pesticide reference standards to each 25-mL volumetric flask, dissolve and dilute to volume with acetone or *n*-hexane as the standard stock solutions. Store at -18°C in the dark. Mix appropriate volume of each standard stock solution, and dilute with acetone: *n*-hexane (1:1, v/v) to 1 μg/mL as the standard solution for analysis of GC-MS/MS in section 2.9.2.

2.8. Sample solution preparation

Transfer about 10 g of the homogenized muscle or visceral sample accurately weighed; remove eggs' shells and transfer about 10 g of the mixed egg white and yolk sample accurately weighed into a 50-mL centrifuge tube. Add 10 mL of 1% acetic acid in acetonitrile and 10 μL of 50 μg/mL internal standard solution after freezing. Add 1 granule of a ceramic homogenizer and the extraction powder, cap the centrifuge tube, shake

vigorously several times by hands to prevent coagulation of salt, and then shake at 1000 rpm by the high speed dispersing device or shake vigorously by hands for 3 min. Centrifuge at 4000 ×g for 5 min at 15°C, and transfer 5 mL of the supernatant to a clean-up centrifuge tube (avoid getting the oil layer). Shake at 1000 rpm by the high speed dispersing device or shake vigorously by hands for 1 min, and centrifuge at 4000 ×g for 5 min at 15°C. Take 1 mL of the supernatant, and evaporate to near dryness by gently flushing with a stream of nitrogen. Dissolve the residue with 1 mL of methanol, mix well, and filter with a membrane filter. Take the filtrate as the sample solution I for analysis of LC-MS/MS. Take another 1 mL of the supernatant, and evaporate to near dryness by gently flushing with a stream of nitrogen. Dissolve the residue with 1 mL of acetone: *n*-hexane (1:1, v/v), mix well, and filter with a membrane filter. Take the filtrate as the sample solution II for analysis of GC-MS/MS.

2.9. Matrix-matched calibration curve

2.9.1. LC-MS/MS

Take a blank sample without adding the internal standard, and follow the procedure described in section 2.8 to obtain the supernatant after the clean-up procedure. Take several 1 mL of the supernatant and evaporate to near dryness by gently flushing with a stream of nitrogen. Separately add appropriate volume of methanol, 5 - 200 μL of 1 μg/mL the standard solution (prepare 2 - 200 μL for fipronil and its metabolites) and 10 μL of 5 μg/mL the internal standard solution to achieve a final volume of 1 mL, and mix well as the matrix-matched standard solutions I. Operate LC-MS/MS according to the following conditions. Establish the matrix-matched calibration curve of each pesticide by the ratios of the peak area of each pesticide to that of the internal standard vs. the added concentrations (0.005 - 0.2 μg/mL; 0.002 - 0.2 μg/mL for fipronil and its metabolite).

LC-MS/MS operating conditions(note 3):

Column: CORTECS UPLC, C18, 1.6 μm, 2.1 mm i.d. × 10 cm.

Guard column: CORTECS UPLC, C18, 1.6 μm, 2.1 mm i.d. × 5 mm.

Oven temperature: 30°C.

Mobile phase: gradient program of solvent A and solvent B is as follows.

Time (min)	A (%)	B (%)
0.0 → 2.0	99 → 50	1 → 50
2.0 → 8.0	50 → 30	50 → 70
8.0 → 10.0	30 → 1	70 → 99
10.0 → 13.0	1 → 1	99 → 99
13.0 → 13.5	1 → 99	99 → 1
13.5 → 15.0	99 → 99	1 → 1

Flow rate: 0.3 mL/min.

Injection volume: 5 µL.

Capillary voltage: ESI^+, 3.5 kV; ESI^-, 1.6 kV.

Ion source temperature: 150°C.

Desolvation temperature: 500°C.

Detection mode: multiple reaction monitoring (MRM). Detection ion pair, cone voltage and collision energy are shown in **Table 1** and **Table 2**.

Note 3: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

2.9.2. GC-MS/MS

Take a blank sample without adding the internal standard, and follow the procedure described in section 2.8 to obtain the supernatant after the clean-up procedure. Take several 1 mL of the supernatant and evaporate to near dryness by gently flushing with a stream of nitrogen. Separately add appropriate volume of acetone: *n*-hexane (1:1, v/v), 5 - 200 µL of 1 µg/mL the standard solution and 10 µL of 5 µg/mL the internal standard solution to achieve a final volume of 1 mL, and mix well as the matrix-matched standard solutions II. Operate GC-MS/MS according to the following conditions. Establish the matrix-matched calibration curve of each pesticide by the ratios of the peak area of each pesticide to that of the internal standard vs. the added concentrations (0.005 - 0.2 µg/mL).

GC-MS/MS operating conditions(note 3):

Column: HP-5MS UI capillary column, 0.25 µm, 0.25 mm × 30 m.

Oven temperature:

Initial temperature: 60°C, 1 min;

Temperature rising rate: 40°C /min;

Middle temperature: 170°C;
Temperature rising rate: 10°C /min;
Final temperature: 310°C, 2.25 min.
Gas flow rate: helium, 1 mL/min.
Injection volume: 1 μL.
Injector temperature: 280°C.
Injection mode: splitless.
Ion source: electron ionization, 70 eV.
Ion source temperature: 280°C.
Detection mode: multiple reaction monitoring, (MRM).Detection ion pair and collision energy are shown in **Table 3**.

2.10. Identification and quantification

2.10.1. LC-MS/MS

Accurately inject 5 μL of the sample solution I and the matrix-matched standard solutions I into LC-MS/MS separately, and operate according to the conditions in section 2.9.1. Identify each pesticide based on the retention time and the relative ion intensities(note 4). Calculate the amount of each pesticide in the sample using the following formula:

$$\text{The amount of each pesticide in the sample (ppm)} = \frac{C \times V}{M}$$

Where,

C: the concentration of each pesticide in the sample solution calculated by the matrix-matched calibration curve (μg/mL)

V: the volume of 1% acetic acid in acetonitrile for sample extraction (10 mL)

M: the weight of the sample (g)

Note 4: Relative ion intensities are calculated by peak areas of quantitative ions divided by peak areas of qualitative ions (≤100%). Maximum permitted tolerances of relative ion intensities are as the following:

Relative ion intersity (%)	Tolerance (%)
> 50	± 20
> 20～50	± 25

> 10～20	± 30
≤ 10	± 50

2.10.2. GC-MS/MS

Accurately inject 1 μL of the sample solution II and the matrix-matched standard solutions II into GC-MS/MS separately, and operate according to the conditions in section 2.9.2. Identify each pesticide based on the retention time and the relative ion intensities(note5). Calculate the amount of each pesticide in the sample using the following formula:

$$\text{The amount of each pesticide in the sample (ppm)} = \frac{C \times V}{M}$$

Where,

C: the concentration of each pesticide in the sample solution calculated by the matrix-matched calibration curve (μg/mL)

V: the volume of 1% acetic acid in acetonitrile for sample extraction (10 mL)

M: the weight of the sample (g)

Remark

1. Limit of quantification (LOQ) for each pesticide is listed in the attached table.
2. Since the internal standard, triphenylphosphate (TPP), used in the procedure may not represent the physicochemical properties of all items of pesticides, it is optional for applying it in the formula to calculate the amount of pesticides in the sample. The TPP is recommended to serve as a quality control factor to confirm the operating procedure.
3. Whether pesticide items should be analyzed by LC-MS/MS or GC-MS/MS can be reviewed as needed.
4. Because the spinosad standard contains two compounds (spinosad A and spinosad D), the lowest concentration level of the matrix-matched calibration curve for each compound should be calculated by its LOQ multiplied by the proportion of each compound in the pesticide standard.
5. Further validation should be performed when interfering compounds are found in the samples.

Reference

1. Lehotay, S. J., Maštovská, K. and Yun, S. J. 2005. Evaluation of two fast and easy methods for pesticide residue analysis in fatty food matrixes. J. AOAC Int. 88: 630-638.
2. European Committee for Standardization. 2017. Food 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. DIN EN 15662: 2017 (English version).

Table 1. MRM parameters and LOQs of 65 pesticides including acephate etc. and the internal standard (LC-MS/MS positive ion mode)

No	Analyte	Quantitative ion pair			Qualitative ion pair			LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Muscle	Viscera	Egg
1	Acephate	184 > 143	11	8	184 > 125	11	18	0.01	0.01	0.01
2	Acetamiprid	223 > 126	26	21	223 > 90	26	35	0.01	0.01	0.01
3	Aldicarb	208 > 89	10	20	208 > 116	10	8	0.01	0.01	0.01
4	Amisulbrom	468 > 229	20	16	468 > 148	20	50	0.01	0.01	0.01
5	Azoxystrobin	404 > 372	22	15	404 > 329	22	30	0.01	0.01	0.01
6	Bendiocarb	224 > 167	20	8	224 > 109	20	18	0.01	0.05	0.01
7	Boscalid	343 > 307	35	20	343 > 140	35	20	0.01	0.01	0.01
8	Buprofezin	306 > 201	20	12	306 > 116	20	16	0.01	0.01	0.01
9	Carbaryl	202 > 145	22	22	202 > 117	22	28	0.01	0.01	0.01
10	Carbendazim	192 > 160	27	18	192 > 132	27	28	0.01	0.01	0.01
11	Carbofuran	222 > 165	34	16	222 > 123	34	16	0.01	0.05	0.01
12	Chlorantraniliprole	484 > 453	24	22	484 > 286	24	14	0.01	0.01	0.01
13	Chlorfluazuron	540 > 383	42	20	540 > 158	42	20	0.05	0.01	0.01
14	Clofentezine	303 > 138	22	22	303 > 102	22	35	0.05	0.05	0.01
15	Clothianidin	250 > 169	18	12	250 > 132	18	18	0.01	0.01	0.01
16	Cyazofamid	325 > 108	20	20	325 > 261	20	10	0.01	0.01	0.01
17	Dimethoate	230 > 125	14	20	230 > 199	14	11	0.01	0.01	0.01
18	Dimethomorph	388 > 301	35	20	388 > 165	35	30	0.01	0.01	0.01
19	Dinotefuran	203 > 157	20	8	203 > 129	20	14	0.01	0.01	0.01
20	Etoxazole	360 > 141	35	35	360 > 304	35	17	0.01	0.01	0.01

Table 1. MRM parameters and LOQs of 65 pesticides including acephate etc. and the internal standard (LC-MS/MS positive ion mode) (continued)

No	Analyte	Quantitative ion pair			Qualitative ion pair			LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Muscle	Viscera	Egg
21	Famoxadone	392 > 331	20	11	392 > 238	20	20	0.01	0.01	0.01
22	Fenazaquin	307 > 57	30	25	307 > 161	30	19	0.01	0.05	0.01
23	Fenobucarb	208 > 95	20	10	208 > 152	20	10	0.01	0.01	0.01
24	Fenpyroximate	422 > 366	26	15	422 > 138	26	32	0.01	0.01	0.01
25	Fenthion	279 > 247	30	12	279 > 169	30	18	0.01	0.01	0.01
26	Flonicamid	230 > 203	32	18	230 > 174	32	18	0.01	0.01	0.01
27	Fludioxonil	266 > 158	14	32	266 > 185	14	24	0.01	0.01	0.01
28	Flufenoxuron	489 > 158	40	22	489 > 141	40	46	0.01	0.01	0.01
29	Fluopicolide	385 > 175	29	23	385 > 147	29	49	0.01	0.01	0.01
30	Flusilazole	316 > 165	32	26	316 > 247	32	16	0.01	0.01	0.01
31	Flutriafol	302 > 70	26	18	302 > 123	26	29	0.01	0.01	0.01
32	Hexaconazole	314 > 70	26	22	314 > 159	26	40	0.01	0.01	0.01
33	Hexythiazox	353 > 228	24	14	353 > 168	24	26	0.01	0.01	0.01
34	Imidacloprid	256 > 175	28	20	256 > 209	28	15	0.01	0.01	0.01
35	Isoprocarb	194 > 95	18	14	194 > 137	18	8	0.01	0.01	0.01
36	Mecarbam	330 > 227	21	8	330 > 97	21	35	0.01	0.01	0.01
37	Metalaxyl	280 > 220	20	13	280 > 192	20	17	0.01	0.01	0.01
38	Methiocarb	226 > 169	22	10	226 > 121	22	22	0.01	0.01	0.01
39	Methoprene	279 > 81	24	24	279 > 95	24	22	0.01	0.01	0.05
40	Metrafenone	409 > 209	20	17	409 > 227	20	29	0.01	0.01	0.01

Table 1. MRM parameters and LOQs of 65 pesticides including acephate etc. and the internal standard (LC-MS/MS positive ion mode) (continued)

No	Analyte	Quantitative ion pair			Qualitative ion pair			LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Muscle	Viscera	Egg
41	Monocrotophos	224 > 127	20	16	224 > 98	20	12	0.01	0.01	0.01
42	Nitenpyram	271 > 126	30	15	271 > 237	30	15	0.01	0.01	0.01
43	Omethoate	214 > 183	20	10	214 > 155	20	15	0.01	0.01	0.01
44	Oxycarboxin	268 > 175	20	16	268 > 147	20	25	0.01	0.01	0.01
45	Pencycuron	329 > 125	40	22	329 > 218	40	20	0.01	0.01	0.01
46	Phoxim	299 > 129	20	11	299 > 153	20	7	0.01	0.01	0.01
47	Pirimicarb	239 > 72	28	18	239 > 182	28	15	0.01	0.01	0.01
48	Prochloraz	376 > 308	17	11	376 > 266	17	14	0.01	0.01	0.01
49	Profenophos	373 > 303	36	20	373 > 128	36	40	0.01	0.05	0.01
50	Propargite	368 > 231	15	11	368 > 175	15	17	0.01	0.01	0.01
51	Propoxur	210 > 111	15	16	210 > 168	15	10	0.01	0.01	0.01
52	Pyraclostrobin	388 > 163	25	25	388 > 194	25	12	0.01	0.01	0.01
53	Pyridaben	365 > 147	22	24	365 > 309	22	12	0.01	0.01	0.01
54	Spinosad A	733 > 142	50	31	733 > 98	50	59	0.01	0.01	0.01
55	Spinosad D	747 > 142	51	31	747 > 98	51	53			
56	Spirodiclofen	411 > 313	25	11	411 > 71	25	14	0.01	0.01	0.01
57	Tebufenozide	353 > 133	13	20	353 > 297	13	8	0.01	0.01	0.01
58	Tebufenpyrad	334 > 117	52	34	334 > 145	52	28	0.01	0.01	0.01
59	Thiabendazole	202 > 175	45	25	202 > 131	45	30	0.05	0.01	0.01
60	Thiacloprid	253 > 126	35	20	253 > 90	35	40	0.01	0.01	0.01

Table 1. MRM parameters and LOQs of 65 pesticides including acephate etc. and the internal standard (LC-MS/MS positive ion mode) (continued)

No	Analyte	Quantitative ion pair			Qualitative ion pair			LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Muscle	Viscera	Egg
61	Thiamethoxam	292 > 211	22	12	292 > 132	22	22	0.01	0.01	0.01
62	Thiophanate-methyl	343 > 151	40	27	343 > 268	40	17	0.01	0.01	0.01
63	Trichlorfon	257 > 109	22	18	257 > 79	22	30	0.01	0.01	0.01
64	Tricyclazole	190 > 163	38	20	190 > 136	38	26	0.01	0.01	0.01
65	Trifloxystrobin	409 > 186	28	16	409 > 145	28	40	0.01	0.01	0.01
I.S.	Triphenylphosphate	327 > 77	40	35	-	-	-	-	-	-

Table 2. MRM parameters and LOQs of 3 pesticides including diflubenzuron etc. (LC-MS/MS negative ion mode)

No	Analyte	Quantitative ion pair			Qualitative ion pair			LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Cone voltage (V)	Collision energy (eV)	Muscle	Viscera	Egg
1	Diflubenzuron	309 > 289	-20	10	309 > 156	-20	10	0.01	0.01	0.01
2	Fipronil	435 > 330	-25	20	435 > 250	-25	25	0.005	0.005	0.005
3	Fipronil-sulfone	451 > 415	-29	16	451 > 282	-29	28			

Table 3. MRM parameters and LOQs of 61 pesticides including aldrin etc. and the internal standard (GC-MS/MS)

No	Analyte	Quantitative ion pair		Qualitative ion pair		LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Muscle	Viscera	Egg
1	Aldrin	263 > 193	35	263 > 228	20	0.01	0.01	0.05
2	Bifenthrin	181 > 166	15	165 > 115	30	0.01	0.01	0.01
3	Bitertanol	170 > 141	20	170 > 115	30	0.01	0.01	0.01
4	Bromopropylate	341 > 183	15	341 > 155	25	0.01	0.01	0.01
5	Bupirimate	273 > 193	10	208 > 165	10	0.01	0.01	0.01
6	Chlorfenvinphos	267 > 159	20	323 > 267	15	0.01	0.01	0.01
7	cis-Chlordane	373 > 266	20	373 > 301	10	0.01	0.01	0.01
8	Chlorpyrifos	314 > 258	15	314 > 286	10	0.01	0.01	0.01
9	Chlorpyrifos-methyl	286 > 93	20	286 > 271	15	0.01	0.01	0.01
10	Cyfluthrin	163 > 127	5	206 > 151	20	0.01	0.05	0.01
11	λ-Cyhalothrin	181 > 152	20	197 > 141	15	0.01	0.01	0.01
12	Cypermethrin	163 > 127	5	181 > 152	20	0.01	0.01	0.01
13	o,p'-DDD	235 > 165	25	235 > 199	15	0.01	0.01	0.01
14	o,p'-DDE	246 > 176	30	318 > 246	25	0.01	0.01	0.01
15	*o,p'*-DDT	235 > 165	15	235 > 199	15	0.01	0.01	0.01
16	Deltamethrin	253 > 93	20	253 > 172	10	0.01	0.01	0.01
17	Diazinon	304 > 179	10	179 > 137	15	0.01	0.01	0.01
18	Difenoconazole	323 > 265	15	323 > 202	30	0.01	0.01	0.01
19	Dimethipin	118 > 58	5	124 > 76	5	0.01	0.01	0.01
20	Edifenphos	173 > 109	10	310 > 173	15	0.01	0.01	0.01

Table 3. MRM parameters and LOQs of 61 pesticides including aldrin etc. and the internal standard (GC-MS/MS) (continued)

No	Analyte	Quantitative ion pair		Qualitative ion pair		LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Muscle	Viscera	Egg
21	alpha-Endosulfan	241 > 206	15	241 > 170	25	0.01	0.01	0.01
22	beta-Endosulfan	195 > 159	10	241 > 206	15	0.01	0.01	0.01
23	Endosulfan-sulfate	272 > 237	15	387 > 253	10	0.01	0.01	0.01
24	Endrin	263 > 193	25	263 > 228	20	0.01	0.01	0.01
25	Ethion	231 > 129	25	231 > 175	15	0.01	0.01	0.01
26	Etrimfos	292 > 181	10	292 > 153	20	0.01	0.01	0.01
27	Fenitrothion	277 > 109	20	277 > 260	10	0.01	0.01	0.01
28	Fenpropathrin	181 > 152	20	265 > 210	15	0.01	0.01	0.01
29	Fensulfothion	156 > 141	10	292 > 109	15	0.01	0.01	0.01
30	Fenvalerate	167 > 125	10	125 > 89	20	0.01	0.01	0.01
31	Flucythrinate	199 > 157	10	451 > 199	10	0.01	0.01	0.01
32	Flutolanil	173 > 145	15	281 > 173	15	0.01	0.01	0.01
33	Heptachlor	272 > 237	15	237 > 167	30	0.01	0.01	0.01
34	Iprodione	314 > 245	15	314 > 271	10	0.05	0.05	0.01
35	Isofenphos	213 > 121	15	213 > 185	10	0.01	0.01	0.01
36	Isoprothiolane	290 > 118	15	290 > 204	10	0.01	0.01	0.01
37	Kresoxim-methyl	116 > 89	10	206 > 116	10	0.01	0.01	0.01
38	Lindane	181 > 145	15	219 > 183	10	0.01	0.01	0.01
39	Methacrifos	125 > 79	5	208 > 93	15	0.01	0.01	0.01
40	Methidathion	145 > 85	10	145 > 58	15	0.01	0.01	0.01

Table 3. MRM parameters and LOQs of 61 pesticides including aldrin etc. and the internal standard (GC-MS/MS) (continued)

No	Analyte	Quantitative ion pair		Qualitative ion pair		LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Muscle	Viscera	Egg
41	Myclobutanil	179 > 125	15	179 > 152	10	0.01	0.01	0.01
42	Oxadiazon	258 > 175	10	258 > 112	25	0.01	0.01	0.01
43	Oxadixyl	163 > 132	10	163 > 117	25	0.01	0.01	0.01
44	Penconazole	248 > 157	25	248 > 192	15	0.01	0.01	0.01
45	Pendimethalin	252 > 162	10	252 > 191	10	0.01	0.01	0.01
46	Permethrin	183 > 153	15	163 > 127	5	0.01	0.05	0.01
47	Phenthoate	274 > 121	10	274 > 246	10	0.01	0.01	0.01
48	Phorate	121 > 93	5	260 > 75	10	0.01	0.01	0.01
49	Phosalone	182 > 111	15	182 > 138	10	0.01	0.01	0.01
50	Phosmet	160 > 133	10	160 > 105	15	0.01	0.01	0.01
51	Pirimiphos-methyl	290 > 125	20	290 > 151	20	0.01	0.01	0.01
52	Procymidone	96 > 68	10	283 > 96	10	0.01	0.05	0.01
53	Propiconazole	173 > 145	15	259 > 69	10	0.01	0.01	0.01
54	Prothiofos	267 > 239	10	309 > 239	15	0.01	0.01	0.01
55	Pyriproxyfen	136 > 96	15	136 > 78	25	0.01	0.01	0.01
56	Tebuconazole	250 > 125	20	250 > 153	10	0.01	0.01	0.01
57	Terbufos	231 > 175	15	231 > 157	20	0.01	0.01	0.01
58	Tetraconazole	336 > 218	15	336 > 204	30	0.01	0.01	0.01
59	trans-Chlordane	373 > 266	20	373 > 301	10	0.01	0.01	0.01
60	Triadimefon	208 > 181	10	208 > 111	20	0.01	0.01	0.01

Table 3. MRM parameters and LOQs of 61 pesticides including aldrin etc. and the internal standard (GC-MS/MS) (continued)

No	Analyte	Quantitative ion pair		Qualitative ion pair		LOQ (ppm)		
		Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Precursor ion (*m/z*) > product ion (*m/z*)	Collision energy (eV)	Muscle	Viscera	Egg
61	Vinclozolin	212 > 145	25	285 > 212	10	0.01	0.01	0.01
I.S.	Triphenylphosphate	326 > 169	30	-	-	-	-	-