



## **Validation of the MRM pesticides from the EU-MACP in chia seeds and Goji berries**

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## 1. Aim and scope

This document reports the validation data for 178 pesticides included in the European Union Multi Annual Control Program (EU-MACP) [1] using a multiresidue method by LC-MS/MS and GC-MS/MS in chia seeds and Goji berries.

## 2. Short description

Homogenous samples were extracted using three different methods (QuEChERS, ethyl acetate, Mini Luke), which were adapted in the case of chia extractions. The obtained extracts were then analyzed by GC-MS/MS and LC-MS/MS.

These extracts were also analyzed by LC-TOF-MS. Matrix compounds were retrieved and counted using the Molecular Feature Extractor (MFE) algorithm in the MassHunter Workstation Software. The MFE creates a compound list of all the peaks in the data file that represent real molecules. At the end of the data process, a list with the mass, retention time and intensity of all matrix components was obtained. The resulting data was evaluated to get information of the complexity of the matrices through the number and distribution of matrix components.

## 3. Apparatus and consumables

- Automatic pipettes, suitable for handling volumes from 10  $\mu$ L to 5000  $\mu$ L and from 1 mL to 5 mL.
- Graduated 10 mL pipette.
- 50 mL and 15 mL PTFE centrifuge tubes.
- Homogenizer Polytron<sup>TM</sup> PT 10/35 GT.
- Vortex Shaker IKATM 4 Basic.
- Axial shaker Agytax SR1 CP57.
- Centrifuge Orto Alresa Consul 21, suitable for the centrifuge tubes employed in the procedure and capable of achieving at least 4000 rpm.
- Concentration workstation.
- Injection vials, 2 mL, suitable for LC and GC auto-sampler.

## 4. Chemicals

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

- Anhydrous magnesium sulphate
- Primary secondary amine (PSA)
- Petroleum ether
- Acetone
- Dichloromethane
- Supel QuE Z-Sep
- Ammonium formate
- Ultra-pure water
- Methanol HPLC grade
- Formic acid
- Ethyl acetate
- Pesticide standards

## **5. Procedure**

### 5.1. Sample preparation

Following Document No. SANTE/2017/11813 [2], water was added to the chia seeds and Goji berries samples to improve the extraction efficiency of these low moisture commodities. In the case of chia seeds, water should not be added prior to the extraction procedure in order to avoid the appearance of a mucilage around the seeds [3]. Therefore, the extraction procedures were modified so that water could be added after the organic solvent. A grinding step was also included after water addition in order to open the seeds.

Goji berries can be hydrated prior to the extraction procedure. They were mixed with water and grinded to obtain a homogenate with a high water content. The weight proportion matrix-water (in grams) was 5:7.5.

### 5.2. Recovery experiments for method validation

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

For spiking, representative portions of the previously homogenised sample were spiked homogeneously with the appropriate amount of the working standard solution in acetonitrile. The validation methods were performed at two fortification levels (0.01 and 0.05 mg/kg). Three replicates were analysed at each level.

### 5.3. Extraction methods

### 5.3.1. Chia seeds

#### *QuEChERS*

1. Weigh 2 g of chia sample in a 50-mL PTFE centrifuge tube.
2. Add 10 mL acetonitrile and 10 mL H<sub>2</sub>O.
3. Mill the sample using a Polytron homogenizer for 30 sec.
4. Add 4 g anhydrous magnesium sulphate, 1 g sodium chloride, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate.
5. Shake the sample in the Polytron homogenizer for 1 min.
6. Centrifuge the tubes at 4000 rpm for 5 min.
7. Transfer a 5-mL aliquot of the supernatant to a 15 mL PTFE tube containing 750 mg anhydrous magnesium sulphate and 175 mg Z-Sep.
8. Vortex the tubes for 30 sec.
9. Centrifuge at 4000 rpm for 5 min.

#### *Mini Luke*

1. Weigh 1.5 g of chia sample in a 50-mL PTFE centrifuge tube.
2. Add 10 mL acetone and 7.5 mL H<sub>2</sub>O.
3. Mill the sample using a Polytron homogenizer at 1500 rpm for 30 sec.
4. Add 3 g anhydrous magnesium sulphate.
5. Shake the sample in the Polytron homogenizer at 1500 rpm for 30 sec.
6. Add 10 mL petroleum ether and 5 mL dichloromethane.
7. Shake the sample in the Polytron homogenizer at 1500 rpm for 30 sec.
8. Centrifuge at 4000 rpm for 5 min.

#### *Ethyl acetate method*

1. Weigh 2 g of chia sample in a 50-mL PTFE centrifuge tube.
2. Add 10 mL ethyl acetate and 10 mL H<sub>2</sub>O.
3. Mill the sample using a Polytron homogenizer at 1500 rpm for 30 sec.
4. Add 8 g anhydrous magnesium sulphate and 1.5 g sodium chloride.
5. Shake the samples in an Agitax axial extractor for 15 min.
6. Centrifuge the tubes at 4000 rpm for 5 min.

### 5.3.2. Goji berries

#### *QuEChERS*

1. Weigh 12.5 g of Goji homogenate in a 50-mL PTFE centrifuge tube.
2. Add 10 mL acetonitrile.
3. Shake the samples in an Agitax axial extractor for 4 min.

4. Add 4 g anhydrous magnesium sulphate, 1 g sodium chloride, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate.
5. Shake the samples in an Agitax axial extractor for 4 min.
6. Centrifuge at 4000 rpm for 5 min.
7. Transfer a 5-mL aliquot of the supernatant to a 15 mL PTFE tube containing 750 mg anhydrous magnesium sulphate and 125 mg PSA.
8. Vortex the tubes for 30 sec.
9. Centrifuge at 4000 rpm for 5 min.
10. Acidify with 10 µL formic acid 5 % per mL of extract.

#### *Mini Luke*

1. Weigh 9.4 g of Goji homogenate in a 50-mL PTFE centrifuge tube.
2. Add 10 mL acetone and 3 g anhydrous magnesium sulphate.
3. Shake the sample using a Polytron homogenizer at 1500 rpm for 30 sec.
4. Add 10 mL petroleum ether and 5 mL dichloromethane.
5. Shake the sample using a Polytron homogenizer at 1500 rpm for 30 sec.
6. Centrifuge at 4000 rpm for 3 min.

#### *EtAc*

1. Weigh 12.5 g of Goji homogenate in a 50-mL PTFE centrifuge tube.
2. Add 10 mL of ethyl acetate.
3. Shake the tubes manually for 3 sec.
4. Add 8 g anhydrous magnesium sulphate and 1.5 g sodium chloride.
5. Shake the samples in an Agitax axial extractor for 15 min.
6. Centrifuge the tubes at 4000 rpm for 5 min.

#### 5.4. Vial preparation

Extraction method	LC-QqQ-MS/MS	GC-QqQ-MS/MS
QuEChERS	5-fold dilution (ultrapure water)	Solvent change to ethyl acetate
Ethyl acetate	Solvent change to acetonitrile and 5-fold dilution (ultrapure water)	Direct injection
Mini Luke	3.3-fold concentration, solvent change to acetonitrile and 5-fold dilution (ultrapure water)	3.3-fold concentration and solvent change to ethyl acetate

During the vial preparation, dimethoate-d<sub>6</sub> (LC) or lindane-d<sub>6</sub> (GC) were added as internal standards.

#### 5.5. Methodology

Both LC and GC systems were operated in multiple reaction monitoring mode (MRM). Selected reaction monitoring (SRM) experiments were carried out to obtain the maximum sensitivity for the detection of the target molecules. For confirmation of the studied compounds, two SRM transitions and a correct ratio between the abundances of the two optimised SRM transitions (SRM2/SRM1) were used, along with retention time matching. The mass transitions used are presented in Appendix I (Table 1 for LC-MS/MS and Table 2 for GC-MS/MS parameters).

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

### 5.6.1. 1290 UHPLC (Agilent)

- Column: Zorbax Eclipse Plus C8 2.1x100 mm and 1.8 µm particle size (Agilent)
- Mobile phase A: Water (0.1 % formic acid, 5 mM ammonium formate, 2 % MeOH)
- Mobile phase B: Methanol (0.1 % formic acid, 5 mM ammonium formate, 2 % water)
- Column temperature: 35 °C
- Flow rate: 0.3 mL/min
- Injection volume: 5 µL

Mobile phase gradient for pesticides analysis

Time [min]	Mobile phase A	Mobile phase B
0	100 %	0 %
2	80 %	20 %
15	0 %	100 %
18	0 %	100 %

Re-equilibration with initial mobile phase: 2.5 minutes.

### 5.6.2. 6490 triple quadrupole system (Agilent)

- Ionisation mode: Positive mode and negative mode
- Capillary (positive and negative): 3000 V
- Nebulizer: 45 psi
- Nozzle: 400 V
- Drying gas flow: 13 L/min

- Drying gas temperature: 120 °C
- Sheath gas flow: 10 L/min
- Sheath gas temperature: 375 °C
- High Pressure RF (positive): 150 V
- High Pressure RF (negative): 110 V
- Low Pressure RF (positive): 60 V
- Low Pressure RF (negative): 60 V

## 5.7. Instrumentation and analytical conditions for the GC- MS/MS system

### 5.7.1. Intuvo 9000 GC system (Agilent)

- Column: 2 Planar columns HP-5MS UI (15 m long × 0.25 mm i.d. × 0.25 µm film thickness)
- Injection mode: Splitless
- Ultra-inert inlet liner with a glass wool frit from Agilent
- Injection volume: 1 µl
- Injector temperature: 80 °C hold for 0.1 min, then up to 300 °C at 600 °C/min, hold for 5 min and then to 250 °C at 100 °C/min.
- Carrier gas: Helium at constant flow = 1.28 mL/min column 1, 1.48 mL/min column 2.
- Carrier gas purity: 99.999 %
- Oven temperature: 60 °C for 0.5 min, up to 170 °C at 80 °C/min, and up to 310 °C at 20 °C/min.

### 5.7.2. 7410 triple quadrupole system (Agilent)

- Ionisation mode: electron impact ionisation
- Temperature of the transfer line: 280 °C
- Temperature of ion source: 280 °C
- Collision gas: nitrogen
- Collision gas purity: 99.999 %
- Solvent delay: 2.6 minutes

## 5.8. Instrumentation and analytical conditions for the LC-TOF-MS



### 5.8.1. Agilent 1290 HPLC

- Column: Agilent Eclipse Plus Rapid Resolution HD C18, 2.1 mm x 50 mm x 1.8 µm
- Mobile phase A: Methanol 0.1 % Formic Acid, 2 % ultrapure water, 5 mM ammonium formate
- Mobile phase B: 0.1 % Formic acid in ultrapure water, 2 % methanol, 5 mM ammonium formate
- Flow rate: 0.3 mL/min
- Injection volume: 4 µL

#### Mobile phase gradient

Time [min]	Mobile phase A	Mobile phase B
0	20 %	80 %
2	20 %	80 %
15	100 %	0 %
17	100 %	0 %

Re-equilibration with initial mobile phase: 2.5 minutes.

### 5.8.2. Agilent 6550 LC-QTOF-MS

- 4 GHz High Resolution Mode
- ESI source gas temperature: 160 °C
- Gas flow: 14 L/min
- Nebuliser gas and collision gas: nitrogen
- Nebuliser gas pressure: 30 psi
- Sheath gas flow: 12 L/min
- Sheath gas temperature: 350 °C
- Ionisation mode: positive
- Capillary voltage: 4000 V
- OctapoleRFPeak 750V
- Fragmentor 360 V

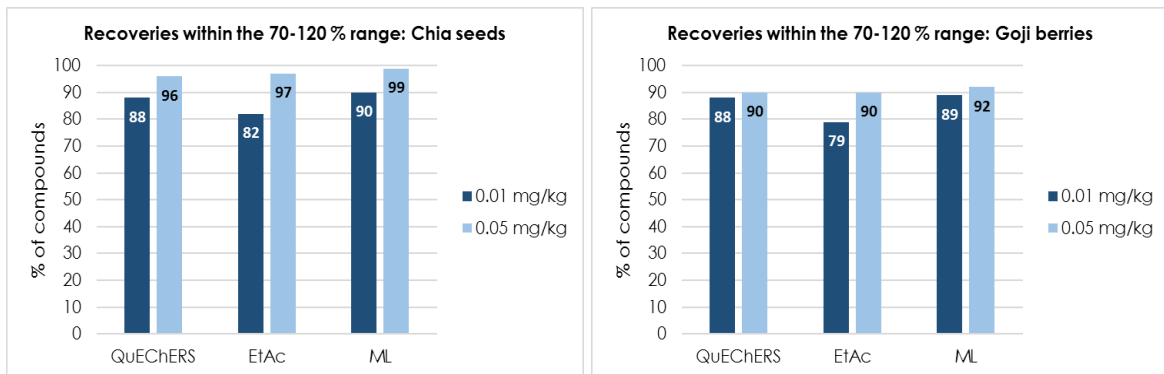
## 6. Results

## 6.1. Validation of the methods

### 6.1.1. Recoveries and within-laboratory reproducibility

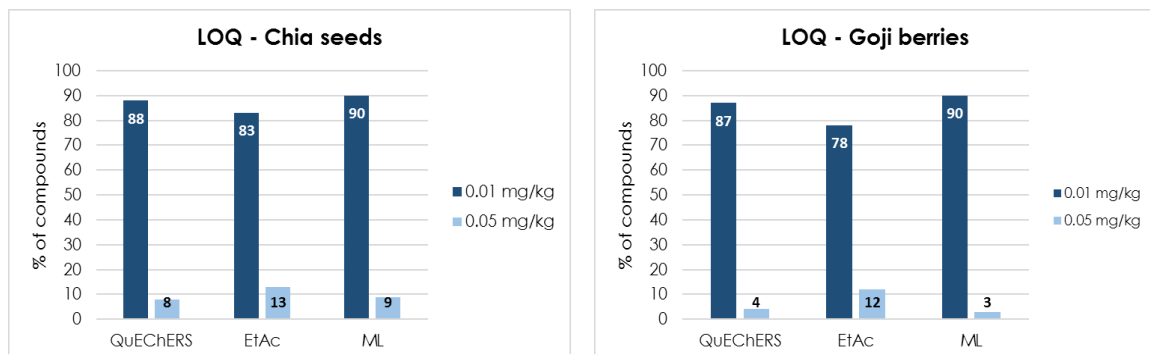
The results corresponding to the mean recovery (n=5) and within-laboratory reproducibility in terms of relative standard deviation (RSD<sub>r</sub>) at two fortification levels (0.01 and 0.05 mg/kg) are summarized in Appendix II, Table 3, Table 4 and Table 5.

Most recovery results are within the range 70-120%. The following graphs summarize the results obtained:



### 6.1.2. Limits of quantification

Document N° SANTE/2017/11813 defines limit of quantitation as the lowest validated spike level meeting the method performance acceptability criteria. LOQs are detailed in Appendix II, Table 6 and Table 7. The results were as follows:

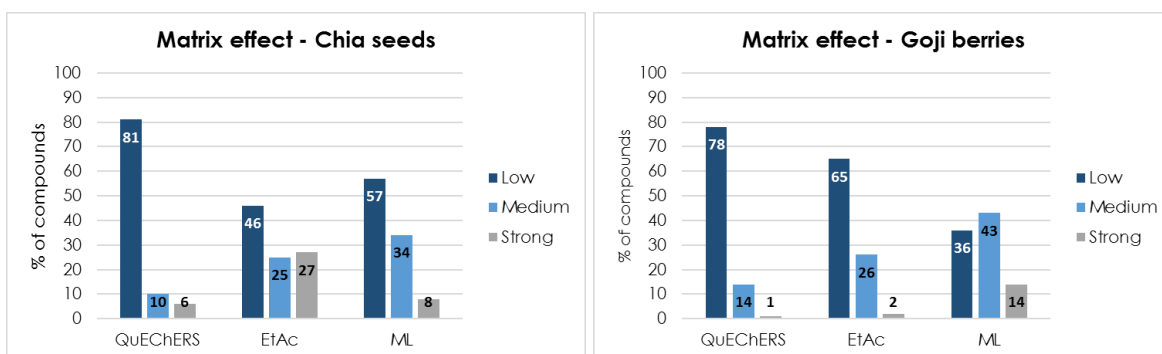


### 6.1.3. Linearity

Linearity of the MS/MS systems was evaluated by assessing the signal responses of the target analytes from matrix-matched calibration solutions prepared by spiking blank extracts at six concentration levels, from 0.001 to 0.100 mg/L. In all cases, the coefficient of determination ( $R^2$ ) was higher than 0.99, except for dithianon. Linearity ranges for all pesticides are summarized in Appendix II, Table 6 and Table 7.

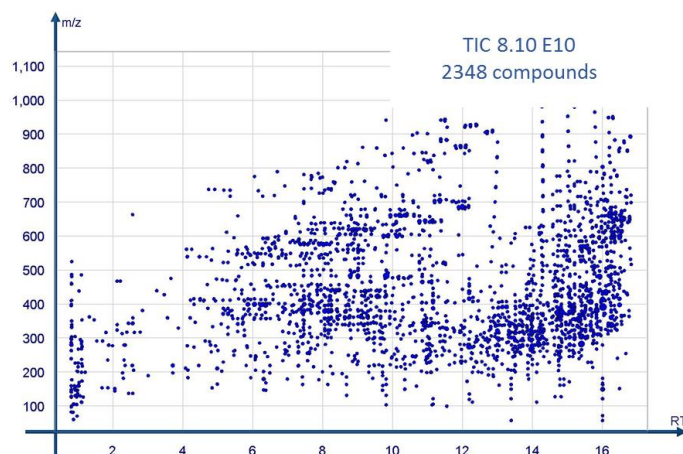
### 6.1.4. Matrix effects

Matrix effects were assessed by comparison of the slopes of six-point matrix-matched calibration curves with the slopes of the calibration curves in solvent (LC) or in tomato (GC). For values (in absolute terms) between 0 and 20 %, there is a low matrix effect; between 20 % and 50 % there is a moderate matrix effect, and for compounds with a value over 50 % there is a strong matrix effect. These values of matrix effects are summarized in Appendix II, Table 6 and Table 7, and they are also represented in the following graphs:

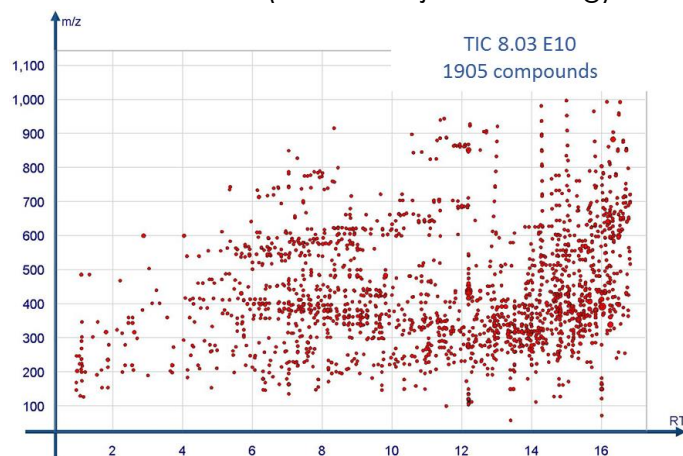


## 6.2. LC-TOF-MS background distribution

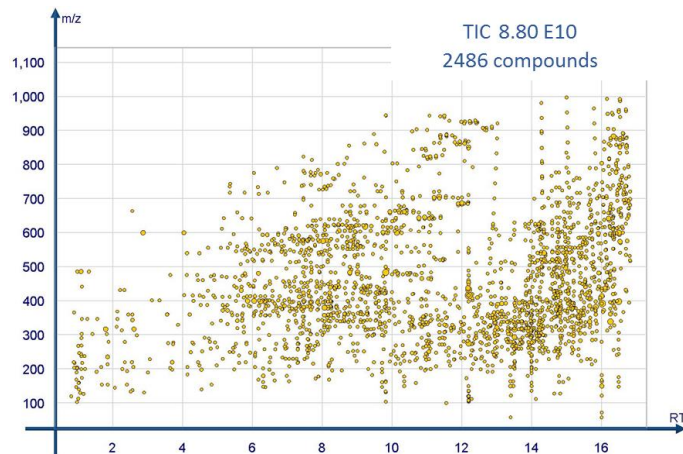
- a) Number and distribution of co-extracted matrix components of chia seeds using QuEChERS method (amount injected: 1 mg)



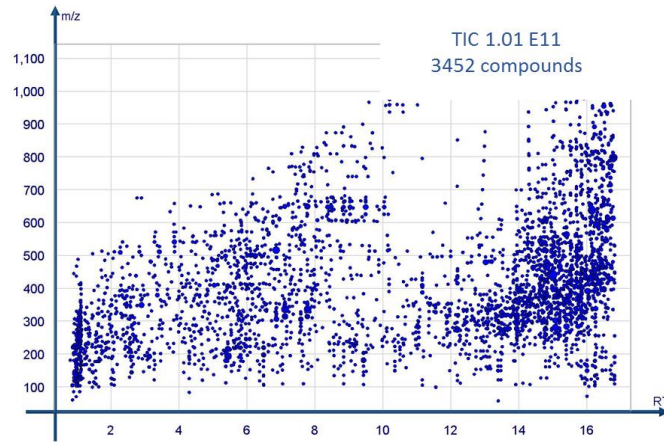
b) Number and distribution of co-extracted matrix components of chia seeds using ethyl acetate method (amount injected: 1 mg)



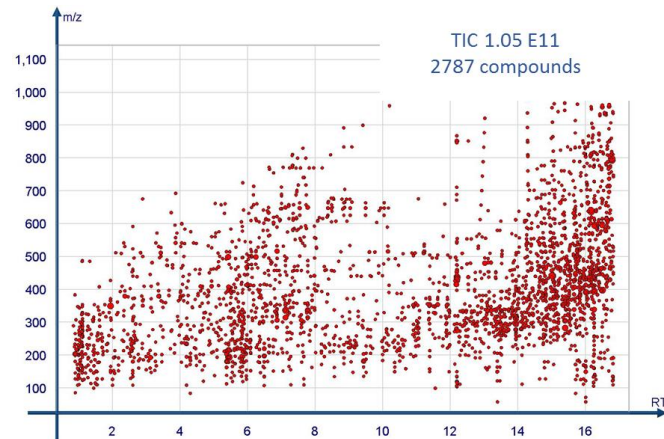
c) Number and distribution of co-extracted matrix components of chia seeds using Mini Luke method (amount injected: 1 mg)



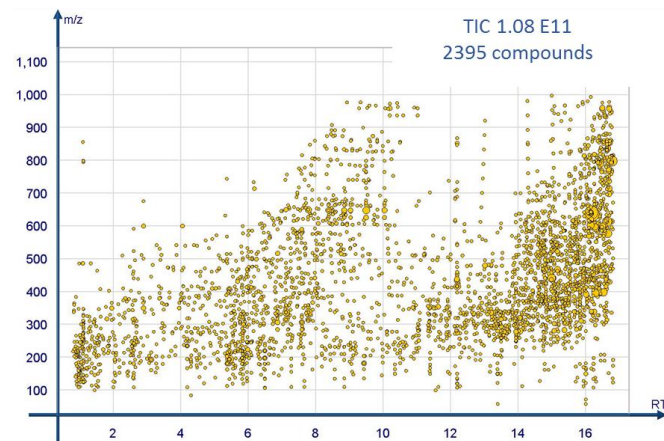
d) Number and distribution of co-extracted matrix components of Goji berries seeds using QuEChERS method (amount injected: 1 mg)



e) Number and distribution of co-extracted matrix components of Goji berries seeds using ethyl acetate method (amount injected: 1 mg)



f) Number and distribution of co-extracted matrix components of Goji berries seeds using Mini Luke method (amount injected: 1 mg)



## 7. References

[1] COMMISSION IMPLEMENTING REGULATION (EU) 2019/533 of 28 March 2019 concerning a coordinated multiannual control programme of the Union for 2020, 2021 and 2022 to ensure compliance with maximum residue levels of pesticides and to assess the consumer exposure to pesticide residues in and on food of plant and animal origin.

[2] Analytical quality control and method validation procedures for pesticide residues analysis in food and feed. Document N° SANTE/2017/11813.

[3] Capitani, M., Ixtaina, V., Nolasco, S. and Tomás, M. (2013). Microstructure, chemical composition and mucilage exudation of chia (*Salvia hispanica*L.) nutlets from Argentina. *Journal of the Science of Food and Agriculture*, 93(15), pp.3856-3862.

**APPENDIX I: MASS TRANSITIONS**
**Table 1.** Detection and chromatographic parameters for the selected compounds analysed by LC-MS/MS.

No.	Name	t <sub>R</sub> (min)	Cone voltaje (V)	Precursor ion (m/z)	Product ion 1 (m/z)	CE 1 (eV)	Product ion 2 (m/z)	CE 2 (EV)	Polarity
1	2,4-D	9.47	380	219.0	161.0	15	163.0	15	Negative
2	Acephate	2.69	380	184.0	143.0	5	125.0	15	Positive
3	Acetamiprid	5.91	380	223.0	126.0	20	56.0	15	Positive
4	Aldicarb	7.40	380	213.0	116.0	10	89.0	15	Positive
5	Aldicarb-sulfone	3.53	380	239.9	223.0	5	86.0	20	Positive
6	Aldicarb-sulfoxide	3.22	380	207.0	132.0	5	89.0	10	Positive
7	Ametoctradin	12.86	380	276.2	176.1	35	149.0	35	Positive
8	Avermectin B1a	14.56	380	890.3	567.1	10	305.1	15	Positive
9	Avermectin B1b	14.33	380	876.5	553.0	15	291.0	15	Positive
10	Azinphos-methyl	10.24	380	318.0	261.0	0	132.1	8	Positive
11	Azoxystrobin	10.62	380	404.0	372.0	10	344.0	20	Positive
12	Benomyl	12.10	380	291.0	191.7	20	160.0	30	Positive
13	Bitertanol	12.58	380	338.2	269.2	5	99.1	10	Positive
14	Bupirimate	11.61	380	317.0	272.0	20	166.0	20	Positive
15	Buprofezin	13.52	380	306.0	201.0	10	116.0	15	Positive
16	Carbaryl	8.90	380	202.0	145.0	10	127.0	20	Positive
17	Carbendazim	4.04	380	192.0	160.0	15	132.0	20	Positive
18	Carbofuran	8.59	380	222.0	165.0	10	123.0	15	Positive
19	Chlorantraniliprole	10.29	380	483.9	452.9	16	285.9	8	Positive
20	Clofentezine	12.35	380	303.0	138.0	12	102.0	40	Positive
21	Clothianidin	5.30	380	250.0	169.0	8	131.9	8	Positive
22	Cyazofamid	11.79	380	325.0	261.2	10	108.1	15	Positive
23	Cymoxanil	6.31	380	199.1	128.0	4	110.9	12	Positive
24	Cyproconazole	11.47	380	292.1	125.0	32	70.0	16	Positive
26	Cyromazine	1.88	380	167.0	125.0	15	59.9	20	Positive
27	Diethofencarb	10.58	380	268.0	226.0	5	180.0	15	Positive
28	Difenoconazole	12.81	380	406.0	337.0	15	251.0	20	Positive
29	Diflubenzuron	11.81	380	311.0	158.0	8	141.0	32	Positive
30	Dimethoate	5.94	380	230.0	199.0	5	171.0	10	Positive
31	Dimethomorph	11.06	380	388.0	301.0	20	165.0	20	Positive
32	Diniconazole	12.92	380	326.1	159.0	28	70.0	28	Positive
33	Dithianon	10.78	380	295.9	264.0	20	163.6	30	Negative
34	DMF	7.81	380	150.0	132.0	15	107.0	20	Positive
35	DMPF	4.60	380	163.0	131.9	15	122.0	15	Positive
36	Dodine	12.49	380	228.2	60.1	20	57.2	20	Positive
37	Emamectin B1a benzoate	13.33	380	886.5	302.2	35	158.1	40	Positive
38	Epoconazole	11.68	380	330.1	121.0	16	101.2	52	Positive
39	Ethion	13.67	380	385.1	199.0	5	171.0	10	Positive

40	Ethirimol	7.22	380	210.2	140.1	20	43.1	52	Positive
41	Etoxazole	13.99	380	360.0	304.0	20	140.9	30	Positive
42	Famoxadone	12.41	380	392.0	331.0	10	238.0	20	Positive
43	Fenamiphos	11.99	380	304.1	234.0	12	217.1	20	Positive
44	Fenbuconazole	11.84	380	337.1	125.1	40	70.0	33	Positive
45	Fenhexamid	11.57	380	302.0	97.0	25	55.0	30	Positive
46	Fenoxycarb	11.95	380	302.2	116.2	5	88.2	20	Positive
47	Fenpropidin	10.25	380	274.3	147.1	30	85.8	25	Positive
48	Fenpropimorph	10.53	380	304.3	147.1	30	130.0	25	Positive
49	Fenpyroximate (E)	14.12	380	422.2	366.2	12	107.0	64	Positive
50	Fenpyroximate (Z)	13.61	380	422.2	366.2	12	107.0	64	Positive
51	Fenthion	12.20	380	279.0	247.1	8	169.1	12	Positive
52	Fenthion sulfone	9.16	380	310.7	125.0	15	108.8	15	Positive
53	Fenthion sulfoxide	8.86	380	295.0	280.0	16	109.0	32	Positive
54	Fipronil	12.15	380	434.9	329.9	12	249.9	28	Negative
55	Fipronil sulfone	12.59	380	451.0	414.8	15	281.9	20	Negative
56	Fonicamid	4.25	380	230.1	202.6	10	173.9	10	Positive
57	Fluazifop	10.84	380	328.2	282.2	15	254.2	20	Positive
58	Flubendiamide	12.33	380	680.9	273.9	15	254.0	20	Negative
59	Fludioxonil	11.00	380	265.9	228.9	5	158.0	20	Positive
60	Flufenoxuron	13.94	380	489.1	158.0	20	140.9	56	Positive
61	Flusilazol	12.03	380	316.1	247.1	12	165.0	24	Positive
62	Flutriafol	9.77	380	302.1	95.0	56	70.1	16	Positive
63	Fluxapyrosad	11.18	380	381.9	362.0	10	342.0	15	Positive
64	Formetanate Hydrochloride	2.76	380	222.1	165.1	8	65.1	52	Positive
65	Fosthiazate	9.39	380	284.0	227.8	10	103.8	20	Positive
66	Haloxypop	12.09	380	362.1	316.2	12	288.1	24	Positive
67	Hexaconazole	12.65	380	314.1	159.0	30	70.1	20	Positive
68	Hexythiazox	13.85	380	353.1	228.2	10	168.2	20	Positive
69	Imazalil	9.38	380	297.0	255.0	15	159.0	20	Positive
70	Imidacloprid	5.16	380	256.0	209.0	15	175.0	15	Positive
71	Indoxacarb	13.00	380	528.1	218.0	20	203.0	45	Positive
72	Iprovalicarb	11.76	380	321.2	202.9	0	119.0	16	Positive
73	Isocarbophos	10.02	380	290.1	121.0	28	65.2	60	Positive
74	Isoprothiolane	11.12	380	291.0	230.7	10	189.1	15	Positive
75	Kresoxim-methyl	12.13	380	314.1	267.0	0	222.1	10	Positive
76	Linuron	10.66	380	249.0	160.1	20	133.0	36	Positive
77	Lufenuron	13.62	380	508.9	339.0	10	325.9	10	Negative
78	Mandipropamid	11.03	380	412.1	356.1	4	328.1	8	Positive
79	Metalaxyl	9.98	380	280.3	220.0	5	192.4	10	Positive
80	Methamidophos	2.26	380	142.1	125.0	10	94.1	10	Positive
81	Methidathion	10.17	380	302.9	145.0	0	85.1	15	Positive
82	Methiocarb	10.83	380	226.1	121.1	12	169.0	5	Positive
83	Methiocarb sulfone	6.23	380	275.0	201.1	5	122.0	15	Positive



84	Methiocarb sulfoxide	5.59	380	242.0	185.0	10	170.0	20	Positive
85	Methomyl	3.99	380	163.1	106.0	4	88.0	0	Positive
86	Methoxyfenozide	11.44	380	369.3	149.0	15	133.0	20	Positive
87	Metrafenone	12.70	380	409.1	226.9	16	209.1	8	Positive
88	Monocrotophos	4.60	380	224.2	193.1	5	127.0	10	Positive
89	Myclobutanil	11.40	380	289.2	125.1	20	70.2	15	Positive
90	Oxamyl	2.89	380	237.0	90.0	5	72.0	10	Positive
91	Paclobutrazol	11.20	380	294.1	125.2	36	70.1	16	Positive
92	Penconazole	12.31	380	284.0	159.0	20	70.0	15	Positive
93	Pencycuron	12.85	380	329.1	125.1	24	89.1	60	Positive
94	Pendimethalin	13.76	380	282.1	212.1	4	194.1	16	Positive
95	Phosmet	10.33	380	318.0	160.0	8	133.0	36	Positive
96	Pirimicarb	7.49	380	239.2	182.1	15	72.2	20	Positive
97	Pirimiphos-methyl	12.54	380	306.2	164.2	20	108.2	20	Positive
98	Profenofos	13.29	380	374.9	347.0	5	304.9	15	Positive
99	Propamocarb	3.14	380	189.2	144.1	10	102.1	15	Positive
100	Propargite	14.00	380	368.1	231.2	0	175.2	8	Positive
101	Propiconazole	12.43	380	342.1	159.0	32	69.1	16	Positive
102	Propyzamide	11.18	380	256.0	190.0	10	173.0	20	Positive
103	Prosulfocarb	13.19	380	252.1	128.0	10	90.9	20	Positive
104	Prothioconazole	12.48	380	341.9	306.1	15	99.8	20	Negative
105	Pymetrozine	2.71	380	218.1	105.0	20	51.0	60	Positive
106	Pyraclostrobin	12.41	380	388.1	193.8	8	163.1	20	Positive
107	Pyriproxyfen	13.51	380	322.0	185.0	20	96.0	10	Positive
108	Quinoxifen	13.58	380	308.1	271.9	25	196.9	35	Positive
109	Spinosyn A	12.46	380	732.5	142.1	30	98.1	40	Positive
110	Spinosyn D	12.82	380	746.5	142.0	25	98.0	40	Positive
111	Spiromesifen	14.09	380	371.0	273.0	5	255.0	20	Positive
112	Spirotetramat	11.60	380	374.2	330.3	15	270.1	20	Positive
113	Spiroxamine	10.89	380	298.0	144.0	20	100.0	20	Positive
114	Tebuconazole	12.32	380	308.0	125.0	20	70.0	20	Positive
115	Tebufenozide	12.22	380	353.2	296.9	5	133.1	15	Positive
116	Teflubenzuron	13.45	380	379.0	359.0	0	339.0	4	Negative
117	Terbutylazine	10.94	380	230.0	174.0	15	146.0	20	Positive
118	Tetraconazole	11.79	380	372.0	159.0	36	70.0	20	Positive
119	Thiabendazol	4.70	380	202.0	175.0	30	131.0	40	Positive
120	Thiacloprid	6.63	380	253.0	186.0	10	126.0	20	Positive
121	Thiamethoxam	4.25	380	292.0	211.0	10	181.0	20	Positive
122	Thiodicarb	9.06	380	355.1	108.1	8	88.1	8	Positive
123	Thiophanate-methyl	8.18	380	343	151	20	93	56	Positive
124	Triadimenol	11.60	380	296.0	227.0	5	70.0	10	Positive
125	Triazophos	11.4	380	314.1	286.2	10	162.2	20	Positive
126	Triflumuron	12.58	380	359.0	156.0	8	139.0	32	Positive

**Table 2.** Acquisition and chromatographic parameters for the selected compounds analysed by GC-MS/MS.

No.	Name	t <sub>R</sub> (min)	Precursor ion 1 (m/z)	Product ion 1 (m/z)	CE 1 (eV)	Precursor ion 2 (m/z)	Product ion 2 (m/z)	CE 2 (eV)
1	2-Phenylphenol	4.42	170	141	30	170	115	40
2	Acrinathrin	9.19	289	93	5	208	181	5
3	Azoxystrobin	11.65	344	329	10	344	156	40
4	Bifenthrin	8.59	181	166	10	181	115	50
5	Biphenyl	3.90	154	126	40	154	102	40
6	Boscalid	10.23	140	112	10	140	76	25
7	Bromopropylate	8.62	341	185	20	341	155	20
8	Bupirimate	7.49	273	193	5	273	108	15
9	Buprofezin	7.49	305	172	5	172	57	15
10	Captan	6.99	264	79	25	151	80	5
11	Chlorfenapyr	7.61	247	227	15	247	200	25
12	Chlorothalonil	5.83	266	231	20	266	133	40
13	Chlorpropham	4.92	213	171	5	213	127	5
14	Chlorpyrifos	6.51	314	286	5	314	258	15
15	Chlorpyrifos-methyl	6.07	288	93	26	286	271	16
16	Cyfluthrin	9.90	226	206	10	163	127	5
17	Cypermethrin	10.06	165	127	5	163	127	5
18	Cyproconazole	7.71	222	125	18	139	111	14
19	Cyprodinil	6.76	224	208	20	224	197	21
20	Deltamethrin	11.34	253	172	5	181	152.1	25
21	Diazinon	5.58	304	179	15	137	84	15
22	Dicloran	5.39	206	176	5	206	148	20
23	Diclorvos	3.39	185	109	15	185	93	15
24	Diclorvos-d6	3.38	191	115	20	191	99	15
25	Dieldrin	7.49	345	263	8	279	243	8
26	Endosulfan sulfate	8.22	387	289	5	272	237	15
27	Endosulfan-alpha	7.25	239	204	15	195	160	5
28	Endosulfan-beta	7.79	207	172	15	195	159	10
29	EPN	8.64	157	110	15	157	77	25
30	Epoxiconazole	8.45	192	138	10	192	111	35
31	Ethion	7.83	231	175	5	231	129	25
32	Etofenprox	10.26	163	135	5	163	107	15
33	Fenamidone	8.74	268	180	20	238	103	20
34	Fenarimol	9.26	219	107	10	139	111	15
35	Fenazaquin	8.77	160	145	5	160	117	20
36	Fenbuconazole	10.00	198	129	5	129	102	15
37	Fenhexamid	8.21	177	113	10	177	78	20
38	Fenitrothion	6.31	277	260	5	277	109	20
39	Fenpropathrin	8.67	265	210	10	265	89	30
40	Fenpropidin	6.26	273	98	3	98	55	12

41	Fenpropimorph	6.47	128	110	10	128	70	12
42	Fenthion	6.49	278	169	20	278	109	20
43	Fenvalerate	10.80	167	125	12	125	89	20
44	Fludioxonil	7.45	248	154	25	248	127	30
45	Fluopicolide	8.17	209	182	20	173	109	25
46	Fluopyram	6.86	223	196	15	173	145	15
47	Fluquinconazole	9.69	340	298	20	340	286	30
48	Flusilazole	7.48	233	165	20	233	152	20
49	Flutriafol	7.25	219	123	12	219	95	20
50	Fluvalinate-tau	10.89	250	200	20	250	55	15
51	Folpet	7.01	260	130	15	147	103	5
52	Fosthiazate	6.69	195	139	5	195	103	5
53	Hexaconazole	7.33	214	172	20	214	159	20
54	Indoxacarb	11.29	264	148	25	203	134	10
55	Iprodione	8.41	244	187	5	187	124	25
56	Iprovalicarb	7.45	158	116	5	158	98	10
57	Isoprothiolane	7.33	162	134	5	162	85	15
58	Kresoxim-methyl	7.48	206	131	10	206	116	5
59	Lambda-Cyhalothrin	9.10	197	161	5	197	141	10
60	Lindane-d6	5.51	224	187	5	224	150	20
61	Malathion	6.38	173	99	15	158	125	8
62	Malathion-d10	6.34	183	151	3	183	132	5
63	Mepanipyrim	7.17	222	207	30	222	158	30
64	Methidathion	7.08	145	85	5	145	58	15
65	Methiocarb	6.31	168	153	10	153	109	10
66	Myclobutanil	7.46	179	152	5	179	125	10
67	Oxadixyl	7.85	163	132	15	163	117	25
68	Paclobutrazol	7.16	236	132	15	236	125	10
69	Parathion-methyl	6.08	263	109	10	233	124	10
70	Penconazole	6.84	248	192	15	248	157	25
71	Pendimethalin	6.82	252	191	10	252	162	10
72	Permethrin	9.52	183	153	15	163	127	5
73	Phosmet	8.69	160	133	15	160	77	30
74	Phthalimide	4.17	147	76	30	104	76	10
75	Procymidone	6.99	283	255	8	283	96	8
76	Profenofos	7.37	337	309	5	337	267	15
77	Propiconazole	8.17	259	191	8	259	173	10
78	Propyzamide	5.53	173	145	15	173	109	30
79	Prosulfocarb	6.21	251	128	5	128	86	3
80	Pyridaben	9.63	147	132	10	147	117	20
81	Pyrimethanil	5.60	198	156	25	198	118	25
82	Pyriproxyfen	8.98	136	96	10	136	78	20
83	Quinoxifen	8.11	307	272	5	307	237	25
84	Spirodiclofen	9.58	312	259	10	312	109	20

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85	Spiromesifen	8.51	272	254	3	272	209	12
86	Tebuconazole	8.29	250	153	12	250	125	20
87	Tefluthrin	5.64	177	137	15	177	127	15
88	Tetraconazole	6.55	336	218	30	336	204	30
89	Tetradifon	8.89	356	229	10	356	159	10
90	Tetrahydrophthalimide	4.30	151	122	8	151	80	5
91	Tolclofos-methyl	6.13	265	250	15	265	220	25
92	Triadimefon	6.54	208	181	5	208	127	15
93	Triazophos	7.94	161	134	5	161	106	10
94	Trifloxystrobin	8.07	222	190	3	222	130	15
95	Vinclozolin	6.05	212	172	15	212	109	40

**APPENDIX II: VALIDATION RESULTS**
**Table 3.** Accuracy data (as % recovery) and precision data (as repeatability RSD<sub>r</sub>, n=5) at 0.01 and 0.05 mg/kg for chia seeds and QuEChERS and ethyl acetate extraction methods.

No.	Compound	Chia				Method
		QuEChERS		Ethyl acetate		
		0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	
1	2,4-D	25 (9)	37 (7)	86 (7)	103 (7)	LC
2	2-Phenylphenol	81 (11)	78 (3)	97 (11)	85 (3)	GC
3	Acephate	86 (3)	106 (12)	45 (4)	111 (12)	LC
4	Acetamiprid	109 (2)	70 (10)	87 (3)	118 (12)	LC
5	Acrinathrin	86 (5)	86 (2)	94 (8)	103 (2)	GC
6	Aldicarb	112 (2)	72 (3)	72 (4)	88 (2)	LC
7	Aldicarb-sulfone	108 (2)	111 (3)	84 (3)	87 (10)	LC
8	Aldicarb-sulfoxide	81 (2)	75 (4)	71 (3)	74 (3)	LC
9	Ametoctradin	90 (3)	111 (10)	93 (4)	80 (11)	LC
10	Avermectin B1a	59 (9)	79 (3)	115 (13)	94 (6)	LC
11	Avermectin B1b	61 (12)	100 (5)	112 (14)	117 (11)	LC
12	Azinphos-methyl	104 (2)	79 (9)	95 (4)	74 (6)	LC
13	Azoxystrobin	112 (2)	77 (11)	99 (9)	86 (10)	LC
14	Carbendazim (Benomyl)	110 (3)	75 (12)	79 (2)	99 (2)	LC
15	Bifenthrin	75 (4)	92 (10)	88 (11)	85 (10)	GC
16	Biphenyl	28 (10)	42 (3)	91 (2)	100 (12)	GC
17	Bitertanol	98 (3)	83 (3)	100 (39)	81 (8)	LC
18	Boscalid	93 (4)	107 (11)	88 (2)	77 (5)	GC
19	Bromopropylate	72 (2)	89 (2)	100 (12)	101 (13)	GC
20	Bupirimate	109 (5)	112 (9)	106 (8)	116 (2)	LC
21	Buprofezin	86 (3)	76 (5)	102 (7)	70 (9)	LC
22	Tetrahydrophthalimide (Captan)	< LOQ	96 (11)	< LOQ	105 (13)	GC
23	Carbaryl	109 (2)	116 (8)	95 (3)	98 (7)	LC
24	Carbofuran	88 (5)	94 (5)	92 (7)	102 (4)	LC
25	Chlorantraniliprole	108 (2)	101 (12)	100 (5)	116 (8)	LC
26	Chlorfenapyr	90 (8)	93 (6)	71 (10)	114 (5)	GC
27	Chlorothalonil	73 (12)	80 (5)	88 (3)	94 (8)	GC
28	Chlorpropham	78 (3)	90 (2)	109 (3)	89 (12)	GC
29	Chlorpyrifos	71 (2)	101 (3)	89 (13)	95 (4)	GC
30	Chlorpyrifos-methyl	70 (5)	79 (4)	94 (11)	80 (11)	GC
31	Clofentezine	89 (6)	72 (12)	89 (7)	97 (10)	LC
32	Clothianidin	< LOQ	< LOQ	< LOQ	< LOQ	LC
33	Cyazofamid	102 (4)	84 (9)	109 (5)	97 (6)	LC
34	Cyfluthrin	89 (4)	118 (7)	92 (3)	116 (3)	GC
35	Cymoxanil	108 (3)	104 (4)	82 (5)	81 (6)	LC
36	Cypermethrin	76 (16)	88 (9)	93 (12)	78 (13)	GC
37	Cyproconazole	101 (3)	97 (8)	97 (2)	95 (8)	LC
38	Cyprodinil	84 (13)	115 (2)	110 (12)	85 (3)	GC
39	Cyromazine	36 (3)	106 (9)	14 (3)	89 (5)	LC
40	Deltamethrin	82 (9)	80 (3)	86 (8)	74 (3)	GC
41	Diazinon	75 (2)	102 (6)	100 (9)	86 (10)	GC
42	Dicloran	84 (16)	115 (11)	96 (5)	105 (2)	GC
43	Diclorvos	54 (15)	42 (12)	92 (2)	81 (6)	GC
44	Dieldrin	< LOQ	99 (6)	< LOQ	100 (2)	GC
45	Diethofencarb	107 (3)	86 (3)	99 (4)	86 (3)	LC
46	Difenoconazole	94 (11)	118 (11)	104 (6)	105 (7)	LC
47	Diflubenzuron	110 (5)	94 (8)	104 (5)	70 (12)	LC

48	Dimethoate	109 (2)	118 (3)	88 (3)	82 (8)	LC
49	Dimethomorph	103 (2)	80 (10)	94 (3)	115 (5)	LC
50	Diniconazole	93 (5)	112 (12)	100 (5)	74 (7)	LC
51	Dithianon	< LOQ	< LOQ	< LOQ	< LOQ	LC
52	DMF	103 (3)	92 (8)	84 (2)	81 (8)	LC
53	DMPF	92 (2)	109 (4)	28 (4)	98 (12)	LC
54	Dodine	109 (6)	119 (2)	80 (2)	74 (5)	LC
55	Emamectin B1a benzoate	81 (8)	106 (10)	60 (6)	70 (6)	LC
56	Endosulfan sulfate	86 (11)	94 (7)	81 (13)	75 (8)	GC
57	Endosulfan-alpha	70 (3)	78 (3)	< LOQ	81 (7)	GC
58	Endosulfan-beta	75 (16)	75 (11)	< LOQ	89 (11)	GC
59	EPN	79 (4)	109 (9)	96 (5)	96 (5)	GC
60	Epoxiconazole	102 (6)	86 (6)	99 (3)	93 (2)	LC
61	Ethion	90 (7)	79 (10)	83 (2)	75 (2)	LC
62	Ethirimol	30 (3)	114 (9)	79 (2)	119 (11)	LC
63	Etofenprox	72 (2)	109 (6)	98 (6)	77 (2)	GC
64	Etoxazole	87 (9)	104 (4)	90 (2)	103 (10)	LC
65	Famoxadone	95 (8)	82 (12)	116 (6)	100 (6)	LC
66	Fenamidone	79 (4)	118 (2)	78 (3)	102 (12)	GC
67	Fenamiphos	106 (4)	76 (3)	68 (4)	79 (7)	LC
68	Fenarimol	100 (4)	80 (10)	89 (5)	108 (10)	GC
69	Fenazaquin	119 (13)	82 (4)	86 (7)	83 (2)	GC
70	Fenbuconazole	105 (3)	99 (9)	99 (3)	72 (9)	LC
71	Fenhexamid	88 (8)	118 (12)	106 (3)	101 (3)	LC
72	Fenitrothion	76 (13)	96 (10)	103 (5)	108 (10)	GC
73	Fenoxycarb	90 (8)	78 (3)	99 (12)	90 (3)	LC
74	Fenpropathrin	83 (13)	114 (5)	93 (10)	78 (12)	GC
75	Fenpropidin	100 (2)	97 (3)	48 (3)	79 (10)	LC
76	Fenpropimorph	104 (3)	89 (9)	87 (9)	99 (4)	LC
77	Fenpyroximate	84 (7)	83 (3)	84 (9)	96 (3)	LC
78	Fenthion	113 (6)	87 (9)	10 (18)	92 (9)	LC
79	Fenthion sulfone	112 (4)	108 (4)	102 (5)	109 (3)	LC
80	Fenthion sulfoxide	101 (2)	106 (2)	113 (3)	83 (6)	LC
81	Fenvalerate	82 (16)	103 (3)	91 (9)	75 (6)	GC
82	Fipronil	106 (4)	99 (2)	101 (9)	110 (3)	LC
83	Fipronil sulfone	102 (3)	115 (11)	114 (2)	73 (12)	LC
84	Flonicamid	111 (3)	87 (9)	86 (3)	92 (10)	LC
85	Fluazifop	39 (5)	92 (9)	110 (16)	101 (3)	LC
86	Flubendiamide	110 (5)	79 (9)	119 (7)	92 (4)	LC
87	Fludioxonil	116 (3)	107 (11)	133 (7)	92 (4)	LC
88	Flufenoxuron	< LOQ	< LOQ	73 (5)	95 (7)	LC
89	Fluopicolide	90 (13)	77 (5)	95 (10)	98 (2)	GC
90	Fluopyram	92 (12)	84 (9)	84 (4)	95 (12)	GC
91	Fluquinconazole	90 (16)	100 (11)	92 (11)	118 (2)	GC
92	Flusilazol	101 (7)	116 (3)	112 (4)	100 (9)	LC
93	Flutriafol	101 (4)	87 (9)	95 (3)	108 (9)	LC
94	Fluvalinate-tau	86 (2)	76 (10)	94 (5)	91 (7)	GC
95	Fluxapyrosad	109 (3)	115 (12)	99 (3)	97 (6)	LC
96	Phthalimide (Folpet)	105 (2)	105 (5)	95 (6)	80 (3)	GC
97	Formetanate Hydrochloride	83 (3)	119 (6)	< LOQ	< LOQ	LC
98	Fosthiazate	111 (2)	92 (6)	96 (2)	116 (7)	LC
99	Haloxyfop	NA	NA	NA	NA	LC
100	Hexaconazole	99 (8)	87 (9)	100 (7)	113 (3)	LC
101	Hexythiazox	82 (9)	117 (6)	83 (10)	117 (9)	LC
102	Imazail	112 (2)	74 (3)	115 (4)	99 (6)	LC
103	Imidacloprid	107 (3)	117 (10)	89 (2)	89 (3)	LC
104	Indoxacarb	98 (3)	96 (2)	100 (5)	111 (7)	LC
105	Iprodione	< LOQ	87 (10)	< LOQ	79 (4)	GC
106	Iprovalicarb	114 (3)	73 (3)	99 (2)	104 (7)	LC

107	Isocarbophos	96 (15)	104 (12)	< LOQ	< LOQ	LC
108	Isoprothiolane	101 (4)	98 (4)	111 (3)	84 (12)	LC
109	Kresoxim-methyl	106 (3)	91 (8)	105 (3)	71 (6)	LC
110	Lambda-Cyhalothrin	88 (4)	81 (3)	91 (3)	103 (6)	GC
111	Linuron	99 (3)	92 (11)	104 (5)	71 (11)	LC
112	Lufenuron	38 (20)	105 (4)	76 (6)	76 (11)	LC
113	Malathion	84 (11)	87 (6)	108 (8)	90 (8)	GC
114	Mandipropamid	109 (4)	87 (10)	99 (4)	119 (11)	LC
115	Meproniprim	84 (3)	99 (4)	< LOQ	103 (2)	GC
116	Metalaxyl	109 (5)	82 (6)	96 (2)	77 (10)	LC
117	Methamidophos	48 (2)	78 (2)	35 (3)	71 (7)	LC
118	Methidathion	108 (3)	76 (11)	98 (5)	81 (8)	LC
119	Methiocarb	104 (2)	92 (7)	98 (4)	100 (6)	LC
120	Methiocarb sulfone	78 (3)	95 (9)	62 (10)	112 (8)	LC
121	Methiocarb sulfoxide	88 (2)	82 (10)	67 (13)	100 (9)	LC
122	Methomyl	115 (3)	81 (8)	85 (3)	70 (3)	LC
123	Methoxyfenozide	110 (2)	73 (4)	102 (2)	109 (3)	LC
124	Metrafenone	103 (3)	76 (6)	107 (5)	117 (8)	LC
125	Monocrotophos	98 (2)	92 (12)	80 (2)	102 (10)	LC
126	Myclobutanil	106 (5)	73 (10)	94 (4)	80 (9)	LC
127	Oxadixyl	101 (10)	86 (3)	< LOQ	110 (7)	GC
128	Oxamyl	98 (8)	79 (8)	79 (4)	108 (9)	LC
129	Paclobutrazol	113 (5)	108 (10)	95 (3)	72 (8)	LC
130	Parathion	78 (3)	90 (5)	102 (13)	107 (4)	GC
131	Parathion-methyl	85 (4)	88 (2)	98 (11)	96 (2)	GC
132	Penconazole	104 (3)	93 (7)	113 (8)	101 (4)	LC
133	Pencycuron	96 (3)	117 (8)	98 (5)	111 (3)	LC
134	Pendimethalin	77 (9)	114 (6)	71 (3)	93 (5)	LC
135	Permethrin	74 (15)	114 (2)	94 (11)	118 (10)	GC
136	Phosmet	111 (3)	82 (7)	101 (3)	91 (10)	LC
137	Pirimicarb	106 (3)	86 (12)	97 (3)	88 (9)	LC
138	Pirimiphos-methyl	NA	NA	NA	NA	LC
139	Procyridone	85 (11)	93 (7)	94 (5)	100 (6)	GC
140	Profenofos	93 (2)	111 (10)	109 (3)	96 (9)	LC
141	Propamocarb	78 (3)	86 (12)	12 (32)	104 (3)	LC
142	Propargite	90 (8)	72 (3)	40 (5)	93 (6)	LC
143	Propiconazole	103 (5)	108 (11)	104 (7)	107 (7)	LC
144	Propyzamide	98 (6)	111 (6)	105 (2)	83 (5)	LC
145	Prosulfocarb	88 (5)	82 (12)	101 (4)	114 (3)	LC
146	Prothioconazole	193 (11)	104 (10)	10 (18)	112 (8)	LC
147	Pymetrozine	51 (3)	89 (10)	15 (2)	74 (12)	LC
148	Pyraclostrobin	103 (10)	101 (5)	114 (2)	102 (3)	LC
149	Pyridaben	72 (2)	88 (3)	95 (6)	112 (8)	GC
150	Pyrimethanil	91 (3)	92 (5)	102 (3)	110 (6)	GC
151	Pyriproxyfen	83 (6)	114 (11)	83 (5)	85 (3)	LC
152	Quinoxifen	73 (6)	94 (12)	93 (4)	85 (7)	LC
153	Spinosyn A	97 (8)	88 (2)	68 (8)	109 (6)	LC
154	Spinosyn D	94 (3)	83 (10)	74 (4)	99 (3)	LC
155	Spirodiclofen	80 (2)	100 (11)	90 (11)	104 (3)	GC
156	Spiromesifen	92 (4)	101 (6)	87 (7)	72 (2)	LC
157	Spirotetramat	111 (2)	76 (6)	93 (3)	117 (8)	LC
158	Spiroxamine	101 (2)	109 (9)	42 (9)	100 (7)	LC
159	Tebuconazole	103 (2)	96 (2)	106 (3)	115 (5)	LC
160	Tebufenozide	109 (2)	73 (12)	106 (3)	82 (12)	LC
161	Tebufenpyrad	77 (15)	96 (9)	94 (3)	99 (3)	GC
162	Teflubenzuron	91 (7)	73 (8)	110 (2)	75 (4)	LC
163	Tefluthrin	76 (8)	113 (11)	99 (3)	107 (14)	GC
164	Terbutylazine	100 (3)	119 (11)	101 (3)	86 (11)	LC
165	Tetraconazole	99 (5)	105 (11)	99 (6)	106 (5)	LC

166	Tetradifon	72 (6)	115 (8)	94 (2)	101 (2)	GC
167	Thiabendazol	51 (3)	91 (2)	55 (3)	97 (5)	LC
168	Thiacloprid	107 (3)	118 (8)	85 (2)	100 (2)	LC
169	Thiamethoxam	106 (3)	97 (3)	79 (3)	106 (3)	LC
170	Thiodicarb	84 (10)	94 (5)	87 (5)	102 (2)	LC
171	Thiophanate-methyl	< LOQ	< LOQ	< LOQ	< LOQ	LC
172	Tolclofos-methyl	74 (4)	94 (6)	95 (2)	94 (5)	GC
173	Triadimefon	96 (6)	110 (5)	99 (2)	89 (2)	GC
174	Triadimenol	102 (5)	118 (3)	109 (4)	105 (10)	LC
175	Triazophos	101 (3)	83 (9)	109 (2)	90 (11)	LC
176	Trifloxystrobin	89 (2)	109 (3)	82 (3)	74 (3)	GC
177	Triflumuron	103 (10)	92 (3)	110 (4)	89 (9)	LC
178	Vinclozolin	88 (17)	96 (9)	96 (11)	101 (14)	GC

NA: Not analysed (present in the sample)

< LOQ: below the limit of quantification

**Table 4.** Accuracy data (as % recovery) and precision data (as repeatability RSD<sub>r</sub>, n=5) at 0.01 and 0.05 mg/kg for Goji berries and QuEChERS and ethyl acetate extraction methods.

No.	Compound	Goji				Method
		QuEChERS		Ethyl acetate		
		0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	
1	2,4-D	< LOQ	35 (12)	86 (3)	116 (12)	LC
2	2-Phenylphenol	85 (11)	92 (3)	92 (6)	118 (7)	GC
3	Acephate	82 (2)	78 (6)	49 (3)	114 (10)	LC
4	Acetamiprid	NA	NA	NA	NA	LC
5	Acrinathrin	103 (3)	97 (5)	98 (3)	112 (9)	GC
6	Aldicarb	103 (2)	113 (6)	73 (3)	88 (9)	LC
7	Aldicarb-sulfone	100 (2)	73 (6)	85 (2)	99 (3)	LC
8	Aldicarb-sulfoxide	89 (3)	77 (5)	66 (4)	72 (12)	LC
9	Ametoctradin	103 (3)	71 (6)	72 (5)	112 (8)	LC
10	Avermectin B1a	119 (5)	82 (6)	93 (16)	89 (9)	LC
11	Avermectin B1b	117 (6)	99 (2)	96 (17)	73 (9)	LC
12	Azinphos-methyl	108 (7)	76 (3)	79 (4)	95 (7)	LC
13	Azoxystrobin	97 (3)	102 (4)	74 (3)	114 (12)	LC
14	Carbendazim (Benomyl)	NA	NA	NA	NA	LC
15	Bifenthrin	96 (3)	114 (7)	102 (10)	93 (8)	GC
16	Biphenyl	71 (5)	109 (4)	95 (11)	115 (6)	GC
17	Bitertanol	114 (5)	76 (7)	75 (9)	118 (4)	LC
18	Boscalid	93 (2)	106 (3)	86 (8)	93 (11)	GC
19	Bromopropylate	101 (7)	108 (11)	97 (8)	98 (2)	GC
20	Bupirimate	110 (5)	79 (10)	72 (10)	78 (12)	LC
21	Buprofezin	102 (3)	105 (9)	72 (7)	112 (9)	LC
22	Tetrahydrophthalimide (Captan)	106 (9)	83 (3)	112 (2)	103 (2)	GC
23	Carbaryl	105 (2)	81 (10)	82 (5)	95 (8)	LC
24	Carbofuran	NA	NA	NA	NA	LC
25	Chlorantraniliprole	102 (2)	98 (5)	83 (4)	87 (3)	LC
26	Chlorfenapyr	100 (7)	107 (9)	87 (3)	100 (10)	GC
27	Chlorothalonil	70 (6)	83 (10)	92 (3)	107 (7)	GC
28	Chlorpropham	99 (10)	83 (7)	100 (4)	95 (4)	GC
29	Chlorpyrifos	87 (7)	100 (2)	105 (3)	106 (8)	GC



30	Chlorpyrifos-methyl	100 (2)	76 (7)	91 (2)	93 (9)	GC
31	Clofentezine	NA	NA	NA	NA	LC
32	Clothianidin	< LOQ	< LOQ	< LOQ	< LOQ	LC
33	Cyazofamid	101 (5)	103 (5)	80 (3)	93 (2)	LC
34	Cyfluthrin	95 (2)	107 (8)	94 (7)	108 (3)	GC
35	Cymoxanil	117 (3)	102 (4)	83 (2)	80 (7)	LC
36	Cypermethrin	NA	NA	NA	NA	GC
37	Cyproconazole	108 (2)	81 (11)	78 (8)	112 (5)	LC
38	Cyprodinil	95 (2)	107 (11)	101 (6)	97 (5)	GC
39	Cyromazine	23 (2)	79 (4)	5 (4)	71 (2)	LC
40	Deltamethrin	96 (4)	83 (11)	75 (12)	99 (7)	GC
41	Diazinon	100 (11)	104 (6)	88 (5)	84 (5)	GC
42	Dicloran	86 (2)	96 (5)	97 (9)	88 (8)	GC
43	Diclorvos	63 (14)	59 (22)	97 (3)	89 (3)	GC
44	Dieldrin	105 (9)	109 (4)	79 (11)	104 (6)	GC
45	Diethofencarb	108 (4)	70 (8)	74 (6)	93 (4)	LC
46	Difenoconazole	117 (5)	82 (7)	76 (10)	115 (9)	LC
47	Diflubenzuron	106 (5)	101 (8)	79 (6)	78 (10)	LC
48	Dimethoate	101 (3)	118 (12)	89 (3)	110 (7)	LC
49	Dimethomorph	98 (2)	96 (6)	84 (5)	101 (11)	LC
50	Diniconazole	96 (5)	90 (3)	74 (7)	105 (5)	LC
51	Dithianon	< LOQ	< LOQ	< LOQ	< LOQ	LC
52	DMF	94 (3)	104 (2)	85 (3)	82 (6)	LC
53	DMPF	93 (3)	114 (3)	11 (2)	83 (7)	LC
54	Dodine	115 (6)	77 (8)	< LOQ	87 (12)	LC
55	Emamectin B1a benzoate	119 (3)	115 (2)	78 (10)	117 (6)	LC
56	Endosulfan sulfate	109 (2)	115 (2)	85 (5)	88 (3)	GC
57	Endosulfan-alpha	106 (10)	106 (2)	82 (8)	118 (3)	GC
58	Endosulfan-beta	111 (2)	101 (4)	82 (2)	100 (4)	GC
59	EPN	94 (6)	96 (3)	94 (3)	104 (11)	GC
60	Epoxiconazole	104 (3)	103 (4)	72 (8)	92 (11)	LC
61	Ethion	109 (2)	72 (7)	98 (3)	113 (12)	LC
62	Ethirimol	81 (3)	79 (10)	51 (3)	113 (9)	LC
63	Etofenprox	105 (3)	116 (3)	103 (6)	79 (4)	GC
64	Etozazole	86 (5)	78 (4)	86 (6)	109 (2)	LC
65	Famoxadone	113 (3)	113 (6)	94 (6)	74 (8)	LC
66	Fenamidone	95 (5)	93 (7)	89 (11)	95 (3)	GC
67	Fenamiphos	104 (3)	86 (4)	65 (8)	103 (4)	LC
68	Fenarimol	95 (4)	98 (4)	87 (3)	99 (8)	GC
69	Fenazaquin	98 (3)	109 (9)	94 (3)	94 (9)	GC
70	Fenbuconazole	109 (7)	104 (7)	80 (7)	74 (12)	LC
71	Fenhexamid	103 (5)	114 (2)	78 (3)	105 (6)	LC
72	Fenitrothion	93 (4)	97 (5)	99 (12)	99 (9)	GC
73	Fenoxycarb	117 (12)	73 (3)	80 (18)	91 (5)	LC
74	Fenpropathrin	97 (2)	80 (3)	100 (3)	80 (4)	GC
75	Fenpropidin	109 (3)	78 (3)	57 (3)	84 (5)	LC
76	Fenpropimorph	110 (3)	85 (3)	70 (3)	110 (3)	LC
77	Fenpyroximate	119 (3)	73 (12)	96 (3)	95 (12)	LC
78	Fenthion	106 (3)	113 (11)	13 (18)	80 (10)	LC
79	Fenthion sulfone	104 (2)	78 (2)	78 (7)	94 (2)	LC
80	Fenthion sulfoxide	111 (2)	75 (11)	98 (3)	115 (12)	LC
81	Fenvalerate	NA	NA	NA	NA	GC
82	Fipronil	111 (5)	95 (11)	75 (6)	87 (4)	LC
83	Fipronil sulfone	108 (4)	118 (2)	82 (2)	90 (6)	LC
84	Fonicamid	97 (2)	112 (3)	90 (3)	105 (4)	LC
85	Fluazifop	19 (12)	87 (9)	101 (4)	115 (9)	LC
86	Flubendiamide	116 (2)	82 (3)	77 (12)	80 (4)	LC
87	Fludioxonil	102 (4)	115 (5)	65 (4)	114 (11)	LC
88	Flufenoxuron	117 (3)	88 (3)	99 (3)	92 (12)	LC

89	Fluopicolide	99 (5)	114 (11)	86 (10)	82 (10)	GC
90	Fluopyram	97 (7)	115 (2)	96 (3)	107 (2)	GC
91	Fluquinconazole	94 (6)	111 (2)	86 (5)	105 (3)	GC
92	Flusilazol	107 (4)	115 (7)	75 (3)	99 (12)	LC
93	Flutriafol	102 (7)	103 (6)	84 (7)	96 (4)	LC
94	Fluvalinate-tau	96 (7)	109 (2)	98 (5)	86 (5)	GC
95	Fluxapyrosad	109 (3)	119 (11)	71 (3)	70 (9)	LC
96	Phthalimide (Folpet)	84 (3)	109 (5)	116 (5)	95 (3)	GC
97	Formetanate Hydrochloride	117 (3)	77 (3)	< LOQ	< LOQ	LC
98	Fosthiazate	105 (4)	71 (7)	94 (2)	92 (7)	LC
99	Haloxyfop	< LOQ	68 (9)	< LOQ	32 (16)	LC
100	Hexaconazole	103 (3)	77 (3)	79 (4)	104 (8)	LC
101	Hexythiazox	103 (6)	93 (2)	111 (4)	88 (12)	LC
102	Imazalil	107 (3)	104 (9)	48 (3)	89 (12)	LC
103	Imidacloprid	98 (3)	76 (5)	105 (5)	77 (4)	LC
104	Indoxacarb	111 (3)	113 (6)	95 (5)	108 (11)	LC
105	Iprodione	< LOQ	< LOQ	< LOQ	< LOQ	GC
106	Iprovalicarb	94 (6)	79 (3)	81 (7)	95 (3)	LC
107	Isocarbophos	105 (10)	103 (4)	< LOQ	99 (8)	LC
108	Isoprothiolane	102 (5)	114 (4)	78 (2)	101 (5)	LC
109	Kresoxim-methyl	105 (4)	84 (10)	72 (7)	70 (3)	LC
110	Lambda-Cyhalothrin	NA	NA	NA	NA	GC
111	Linuron	104 (2)	83 (3)	77 (4)	97 (9)	LC
112	Lufenuron	115 (2)	112 (4)	110 (3)	85 (2)	LC
113	Malathion	99 (10)	104 (6)	97 (4)	106 (3)	GC
114	Mandipropamid	112 (3)	72 (3)	71 (3)	84 (5)	LC
115	Mepanipyrim	90 (11)	87 (5)	92 (2)	111 (2)	GC
116	Metalaxyl	104 (2)	80 (12)	94 (4)	81 (3)	LC
117	Methamidophos	75 (3)	101 (12)	35 (3)	114 (2)	LC
118	Methidathion	106 (2)	113 (11)	78 (4)	102 (3)	LC
119	Methiocarb	107 (5)	118 (4)	79 (3)	78 (12)	LC
120	Methiocarb sulfone	81 (8)	84 (8)	75 (4)	96 (12)	LC
121	Methiocarb sulfoxide	72 (2)	103 (12)	79 (3)	70 (3)	LC
122	Methomyl	94 (3)	98 (8)	84 (3)	112 (7)	LC
123	Methoxyfenozide	106 (4)	95 (3)	80 (3)	116 (8)	LC
124	Metrafenone	116 (2)	101 (6)	76 (8)	82 (11)	LC
125	Monocrotophos	95 (2)	91 (9)	84 (3)	90 (2)	LC
126	Myclobutanil	102 (3)	117 (6)	83 (6)	113 (3)	LC
127	Oxadixyl	71 (8)	118 (5)	92 (11)	98 (11)	GC
128	Oxamyl	93 (5)	107 (6)	81 (2)	111 (11)	LC
129	Paclobutrazol	116 (7)	112 (8)	86 (6)	79 (7)	LC
130	Parathion	89 (10)	101 (3)	90 (2)	86 (10)	GC
131	Parathion-methyl	92 (2)	83 (2)	92 (13)	79 (9)	GC
132	Penconazole	103 (2)	95 (4)	78 (5)	118 (10)	LC
133	Pencycuron	104 (7)	88 (8)	65 (5)	98 (3)	LC
134	Pendimethalin	110 (2)	92 (3)	95 (5)	76 (3)	LC
135	Permethrin	97 (5)	75 (7)	97 (8)	75 (6)	GC
136	Phosmet	100 (2)	91 (5)	75 (5)	95 (2)	LC
137	Pirimicarb	102 (2)	106 (3)	88 (2)	78 (4)	LC
138	Pirimiphos-methyl	99 (2)	106 (11)	68 (3)	83 (3)	LC
139	Procymidone	97 (9)	88 (3)	83 (5)	95 (11)	GC
140	Profenofos	110 (3)	83 (11)	81 (7)	111 (2)	LC
141	Propamocarb	83 (2)	99 (11)	< LOQ	80 (7)	LC
142	Propargite	NA	NA	NA	NA	LC
143	Propiconazole	106 (5)	75 (6)	77 (7)	119 (5)	LC
144	Propyzamide	107 (3)	84 (3)	77 (9)	75 (3)	LC
145	Prosulfocarb	110 (5)	104 (2)	71 (3)	104 (5)	LC
146	Prothioconazole	13 (14)	91 (2)	< LOQ	99 (6)	LC
147	Pymetrozine	46 (3)	99 (7)	< LOQ	72 (6)	LC

148	Pyraclostrobin	108 (3)	115 (6)	82 (6)	76 (5)	LC
149	Pyridaben	NA	NA	NA	NA	GC
150	Pyrimethanil	94 (6)	96 (5)	98 (2)	77 (3)	GC
151	Pyriproxyfen	112 (3)	91 (5)	111 (5)	78 (7)	LC
152	Quinoxifen	105 (7)	117 (4)	71 (3)	79 (6)	LC
153	Spinosyn A	113 (2)	112 (10)	80 (9)	89 (9)	LC
154	Spinosyn D	111 (2)	84 (3)	66 (10)	82 (8)	LC
155	Spirodiclofen	NA	NA	NA	NA	GC
156	Spiromesifen	112 (4)	107 (4)	101 (3)	105 (6)	LC
157	Spirotetramat	107 (5)	105 (9)	85 (10)	98 (9)	LC
158	Spiroxamine	113 (3)	115 (5)	59 (5)	80 (8)	LC
159	Tebuconazole	NA	NA	NA	NA	LC
160	Tebufenozide	111 (3)	87 (10)	72 (5)	90 (7)	LC
161	Tebufenpyrad	95 (10)	115 (5)	94 (13)	84 (3)	GC
162	Teflubenzuron	115 (2)	86 (11)	92 (7)	79 (12)	LC
163	Tefluthrin	98 (10)	92 (7)	104 (5)	84 (4)	GC
164	Terbutylazine	102 (4)	97 (4)	74 (3)	89 (2)	LC
165	Tetraconazole	106 (5)	118 (5)	74 (10)	113 (12)	LC
166	Tetradifon	103 (5)	97 (7)	83 (2)	82 (3)	GC
167	Thiabendazol	79 (4)	73 (12)	65 (3)	86 (11)	LC
168	Thiacloprid	102 (2)	78 (12)	89 (3)	94 (11)	LC
169	Thiamethoxam	95 (3)	75 (3)	82 (2)	76 (12)	LC
170	Thiodicarb	78 (12)	92 (6)	85 (13)	105 (4)	LC
171	Thiophanate-methyl	< LOQ	< LOQ	< LOQ	< LOQ	LC
172	Tolclofos-methyl	97 (2)	76 (3)	93 (5)	103 (11)	GC
173	Triadimefon	94 (5)	116 (7)	97 (2)	94 (2)	GC
174	Triadimenol	NA	NA	NA	NA	LC
175	Triazophos	102 (5)	95 (2)	75 (4)	75 (2)	LC
176	Trifloxystrobin	105 (2)	86 (8)	84 (5)	106 (3)	GC
177	Triflumuron	113 (5)	112 (12)	75 (5)	109 (5)	LC
178	Vinclozolin	103 (5)	80 (2)	89 (2)	111 (3)	GC

NA: Not analysed (present in the sample)

< LOQ: below the limit of quantification

**Table 5.** Accuracy data (as % recovery) and precision data (as repeatability RSD<sub>r</sub>, n=5) at 0.01 and 0.05 mg/kg for chia and Goji berries and Mini Luke extraction method.

No.	Compound	Mini Luke				Method
		Chia seeds		Goji berries		
		0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	0.01 mg/kg Recov. (RSD) (%)	0.05 mg/kg Recov. (RSD) (%)	
1	2-Phenylphenol	91 (11)	92 (2)	81 (3)	114 (9)	GC
2	Acrinathrin	90 (7)	79 (11)	109 (6)	93 (7)	GC
3	Azoxystrobin	84 (15)	114 (8)	91 (2)	115 (3)	GC
4	Bifenthrin	77 (13)	106 (8)	96 (10)	87 (9)	GC
5	Biphenyl	84 (10)	85 (7)	54 (11)	99 (10)	GC
6	Boscalid	89 (6)	114 (5)	97 (4)	82 (10)	GC
7	Bromopropylate	88 (3)	84 (10)	91 (13)	94 (3)	GC
8	Bupirimate	120 (4)	90 (9)	107 (4)	108 (10)	GC
9	Buprofezin	101 (7)	83 (4)	97 (5)	115 (3)	GC
10	Tetrahydrophthalimide (Captan)	79 (14)	78 (11)	88 (12)	75 (10)	GC

11	Chlorfenapyr	71 (10)	115 (5)	95 (2)	87 (8)	GC
12	Chlorothalonil	72 (9)	96 (2)	82 (3)	85 (2)	GC
13	Chlorpropham	84 (7)	77 (3)	91 (10)	76 (5)	GC
14	Chlorpyrifos	83 (12)	96 (3)	95 (3)	117 (10)	GC
15	Chlorpyrifos-methyl	91 (12)	93 (3)	96 (12)	85 (6)	GC
16	Cyfluthrin	87 (5)	108 (2)	94 (8)	106 (9)	GC
17	Cypermethrin	87 (14)	86 (7)	NA	NA	GC
18	Cyproconazole	98 (16)	107 (3)	99 (4)	118 (4)	GC
19	Cyprodinil	99 (11)	86 (10)	99 (6)	94 (4)	GC
20	Deltamethrin	84 (5)	81 (3)	99 (11)	108 (3)	GC
21	Diazinon	92 (5)	97 (8)	93 (8)	111 (10)	GC
22	Dicloran	82 (3)	86 (9)	88 (3)	113 (5)	GC
23	Diclorvos	46 (13)	60 (15)	28 (20)	62 (16)	GC
24	Dieldrin	< LOQ	103 (4)	105 (7)	118 (3)	GC
25	Endosulfan sulfate	92 (3)	86 (2)	91 (6)	94 (5)	GC
26	Endosulfan-alpha	94 (12)	113 (5)	99 (10)	107 (7)	GC
27	Endosulfan-beta	< LOQ	97 (9)	< LOQ	93 (7)	GC
28	EPN	83 (9)	111 (5)	93 (13)	104 (10)	GC
29	Epoxiconazole	95 (2)	118 (2)	91 (13)	100 (5)	GC
30	Ethion	91 (8)	84 (2)	98 (2)	107 (9)	GC
31	Etofenprox	75 (10)	76 (11)	98 (5)	99 (6)	GC
32	Fenamidone	80 (3)	111 (11)	92 (9)	111 (8)	GC
33	Fenarimol	91 (10)	103 (9)	96 (13)	94 (8)	GC
34	Fenazaquin	75 (13)	74 (3)	98 (9)	97 (4)	GC
35	Fenbuconazole	91 (3)	115 (2)	98 (13)	74 (5)	GC
36	Fenhexamid	< LOQ	78 (9)	100 (4)	118 (3)	GC
37	Fenitrothion	84 (11)	76 (8)	89 (6)	96 (7)	GC
38	Fenpropathrin	80 (14)	116 (10)	96 (13)	105 (7)	GC
39	Fenpropidin	112 (3)	107 (6)	112 (11)	92 (3)	GC
40	Fenpropimorph	103 (13)	110 (4)	100 (9)	101 (2)	GC
41	Fenvalerate	86 (9)	112 (11)	NA	NA	GC
42	Fludioxonil	85 (16)	92 (3)	91 (3)	77 (7)	GC
43	Fluopicolide	94 (6)	94 (3)	96 (3)	90 (9)	GC
44	Fluopyram	85 (11)	87 (9)	97 (9)	112 (6)	GC
45	Fluquinconazole	92 (5)	78 (2)	96 (9)	78 (2)	GC
46	Flusilazole	95 (14)	89 (3)	98 (5)	109 (10)	GC
47	Flutriafol	101 (3)	116 (5)	97 (3)	87 (3)	GC
48	Fluvalinate-tau	87 (3)	85 (3)	97 (4)	117 (2)	GC
49	Phthalimide (Folpet)	87 (2)	82 (3)	102 (2)	83 (4)	GC
50	Fosthiazate	< LOQ	79 (4)	88 (5)	83 (3)	GC
51	Hexaconazole	88 (12)	113 (6)	97 (13)	81 (10)	GC
52	Indoxacarb	79 (12)	114 (3)	99 (5)	109 (3)	GC
53	Iprodione	< LOQ	< LOQ	< LOQ	< LOQ	GC
54	Iprovalicarb	< LOQ	87 (6)	93 (2)	91 (10)	GC

55	Isoprothiolane	95 (14)	115 (7)	98 (5)	105 (9)	GC
56	Kresoxim-methyl	93 (5)	106 (8)	96 (5)	88 (3)	GC
57	Lambda-Cyhalothrin	88 (16)	85 (8)	NA	NA	GC
58	Malathion	90 (6)	99 (5)	95 (9)	110 (10)	GC
59	Mepanipyrim	< LOQ	116 (11)	96 (7)	86 (2)	GC
60	Methidathion	93 (3)	109 (2)	97 (5)	105 (9)	GC
61	Myclobutanil	99 (4)	75 (9)	98 (12)	87 (9)	GC
62	Oxadixyl	99 (3)	81 (11)	98 (12)	114 (3)	GC
63	Paclobutrazol	93 (9)	104 (3)	96 (8)	79 (8)	GC
64	Parathion	83 (9)	118 (3)	90 (13)	108 (7)	GC
65	Parathion-methyl	86 (3)	112 (7)	90 (12)	99 (2)	GC
66	Penconazole	89 (7)	94 (6)	97 (2)	116 (9)	GC
67	Pendimethalin	77 (2)	84 (10)	89 (7)	76 (4)	GC
68	Permethrin	82 (9)	88 (4)	98 (3)	105 (10)	GC
69	Phosmet	80 (11)	96 (8)	92 (8)	85 (5)	GC
70	Procymidone	91 (15)	96 (5)	96 (13)	105 (5)	GC
71	Profenofos	91 (4)	98 (9)	96 (11)	84 (2)	GC
72	Propiconazole	97 (14)	82 (8)	92 (7)	102 (3)	GC
73	Propyzamide	96 (2)	74 (2)	95 (6)	115 (5)	GC
74	Prosulfocarb	< LOQ	78 (6)	106 (12)	88 (6)	GC
75	Pyridaben	86 (12)	88 (11)	NA	NA	GC
76	Pyrimethanil	94 (2)	102 (3)	94 (9)	117 (4)	GC
77	Pyriproxyfen	97 (2)	77 (8)	99 (5)	89 (6)	GC
78	Quinoxifen	89 (2)	79 (3)	96 (7)	85 (7)	GC
79	Spirodiclofen	86 (4)	77 (6)	NA	NA	GC
80	Spiromesifen	91 (11)	78 (8)	98 (4)	85 (4)	GC
81	Tebuconazole	94 (3)	81 (8)	NA	NA	GC
82	Tebufenpyrad	95 (6)	107 (6)	98 (13)	113 (8)	GC
83	Tefluthrin	88 (9)	75 (8)	93 (4)	96 (9)	GC
84	Tetraconazole	94 (12)	75 (2)	93 (6)	106 (6)	GC
85	Tetradifon	84 (6)	76 (4)	95 (10)	113 (9)	GC
86	Tolclofos-methyl	89 (3)	115 (2)	95 (4)	118 (2)	GC
87	Triadimefon	94 (8)	101 (10)	92 (7)	93 (8)	GC
88	Triazophos	97 (2)	96 (7)	92 (3)	89 (4)	GC
89	Trifloxystrobin	96 (10)	111 (10)	97 (12)	93 (10)	GC
90	Vinclozolin	91 (8)	77 (11)	95 (6)	118 (10)	GC

NA: Not analysed (present in the sample)

< LOQ: below the limit of quantification.

**Table 6.** Limits of quantification, linearity range, coefficient of determination and matrix effects for the selected matrices studied and the QuEChERS and ethyl acetate extraction methods. Negative values of matrix effects mean suppression of the signal, and positives values, enhancement.

No.	Compound	LOQ (mg/kg)				Linear range (mg/kg)				R <sup>2</sup>				Matrix effect (%)				Method
		Chia seeds		Goji berries		Chia seeds		Goji berries		Chia seeds		Goji berries		Chia seeds		Goji berries		
		Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	Q.C.	Ac.	
1	2,4-D	N.F.R.	0.01	N.F.R.	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9947	0.9932	0.9968	0.9904	-12%	-5%	-2%	-8%	LC
2	2-Phenylphenol	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.001-0.1	1.0000	0.9999	0.9993	0.9991	-12%	12%	26%	31%	GC
3	Acephate	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9997	1.0000	0.9991	1%	-3%	5%	24%	LC
4	Acetamiprid	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9998	1.0000	NA	NA	2%	0%	NA	NA	LC
5	Acrinathrin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9999	0.9930	0.9911	64%	49%	25%	17%	GC
6	Aldicarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9999	0.9997	0.9992	3%	-6%	12%	-27%	LC
7	Aldicarb-sulfone	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9998	0.9999	0.9985	0%	-2%	4%	21%	LC
8	Aldicarb-sulfoxide	0.01	0.01	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9993	0.9998	0.9987	-3%	-7%	3%	17%	LC
9	Ametoctradin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9964	0.9993	0.9963	0.9969	4%	29%	11%	10%	LC
10	Avermectin B1a	0.05	0.01	0.01	0.01	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.9968	0.9980	0.9978	0.9953	4%	39%	13%	-6%	LC
11	Avermectin B1b	0.05	0.01	0.01	0.01	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.9991	0.9970	0.9968	0.9943	4%	38%	13%	-6%	LC
12	Azinphos-methyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9990	0.9992	0.9997	0.9981	-8%	43%	-3%	7%	LC
13	Azoxystrobin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9971	0.9969	0.9983	3%	25%	-12%	2%	LC
14	Carbendazim (Benomyl)	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9996	0.9991	NA	NA	0%	-2%	NA	NA	LC
15	Bifenthrin	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.001-0.1	0.9992	0.9999	0.9993	0.9976	13%	6%	24%	20%	GC
16	Biphenyl	N.F.R.	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9978	0.9998	0.9994	0.9982	-8%	1%	8%	3%	GC
17	Bitertanol	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9980	0.9945	0.9954	4%	53%	7%	-14%	LC
18	Boscalid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9999	0.9992	0.9980	-3%	-11%	-11%	-10%	GC
19	Bromopropylate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9992	0.9997	0.9995	0.9975	-21%	0%	8%	9%	GC
20	Bupirimate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9938	0.9973	0.9996	0.9988	14%	61%	1%	-9%	LC

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21	Buprofezin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9975	0.9931	0.9999	0.9971	15%	68%	4%	-6%	LC	
22	Tetrahydrophthalimide (Captan)	0.05	0.05	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9985	0.9908	0.9935	0.9943	27%	4%	38%	70%	GC	
23	Carbaryl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9990	0.9992	0.9989	1%	17%	6%	18%	LC	
24	Carbofuran	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9988	0.9904	NA	NA	56%	58%	NA	NA	LC	
25	Chlorantraniliprole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9989	0.9977	0.9988	0.9993	-9%	12%	-8%	-1%	LC	
26	Chlorfenapyr	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.005-0.1	0.9990	0.9986	0.9988	0.9978	37%	-7%	26%	23%	GC	
27	Chlorothalonil	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9992	0.9997	0.9983	-9%	-1%	-6%	-2%	GC	
28	Chlorpropham	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9999	0.9996	0.9967	-23%	-17%	-12%	-4%	GC	
29	Chlorpyrifos	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9999	0.9999	0.9977	3%	0%	-6%	3%	GC	
30	Chlorpyrifos-methyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9997	0.9999	0.9978	-26%	-17%	-20%	-16%	GC	
31	Clofentezine	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9991	0.9993	NA	NA	8%	72%	NA	NA	LC	
32	Clothianidin	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	LC
33	Cyazofamid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9946	0.9939	0.9992	0.9957	14%	63%	0%	18%	LC	
34	Cyfluthrin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9984	1.0000	0.9997	0.9993	4%	-7%	-21%	-3%	GC	
35	Cymoxanil	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9959	0.9916	0.9983	-34%	-13%	-5%	6%	LC	
36	Cypermethrin	0.01	0.01	NA	NA	0.005-0.1	0.005-0.1	NA	NA	0.9961	1.0000	NA	NA	13%	-2%	NA	NA	GC	
37	Cyproconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9980	0.9997	0.9997	0.9992	7%	28%	6%	10%	LC	
38	Cyprodinil	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.001-0.1	0.9974	0.9999	0.9996	0.9978	-8%	5%	4%	15%	GC	
39	Cyromazine	0.05	0.05	0.05	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9991	0.9998	0.9985	-4%	-9%	2%	9%	LC	
40	Deltamethrin	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	1.0000	0.9998	0.9998	0.9997	8%	-7%	-15%	-24%	GC	
41	Diazinon	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9999	1.0000	0.9981	-51%	4%	7%	10%	GC	
42	Dicloran	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	1.0000	0.9997	0.9994	0.9985	-6%	2%	-5%	5%	GC	
43	Diclorvos	N.F.R.	0.01	N.F.R.	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9998	1.0000	0.9973	-21%	-4%	12%	7%	GC	
44	Dieldrin	0.05	0.05	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	0.9962	0.9902	0.9987	0.9966	6%	-30%	-10%	-13%	GC	
45	Diethofencarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9988	0.9987	0.9983	1%	31%	-2%	4%	LC	
46	Difenoconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9991	0.9944	0.9999	0.9998	18%	61%	13%	19%	LC	
47	Diflubenzuron	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9985	0.9979	0.9991	10%	68%	3%	40%	LC	
48	Dimethoate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9997	1.0000	0.9986	-3%	-1%	6%	22%	LC	

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49	Dimethomorph	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9993	0.9984	0.9958	0.9932	12%	30%	3%	6%	LC
50	Diniconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9911	0.9988	0.9959	14%	58%	14%	24%	LC
51	Dithianon	N.F.R.	N.F.R.	N.F.R.	N.F.R.	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.9937	0.9379	0.8572	0.9659	-121%	68%	100%	46%	LC
52	DMF	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9999	0.9998	0.9990	6%	7%	4%	31%	LC
53	DMPF	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9931	0.9986	0.9987	-9%	19%	1%	-7%	LC
54	Dodine	0.01	0.01	0.01	N.F.R.	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9933	0.9945	0.9984	0.9946	16%	78%	36%	17%	LC
55	Emamectin B1a benzoate	0.01	0.05	0.01	0.01	0.01-0.1	0.01-0.1	0.01-0.1	0.01-0.1	0.9925	0.9900	0.9922	0.9997	2%	38%	26%	9%	LC
56	Endosulfan sulfate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9988	0.9995	0.9988	17%	-20%	-21%	-16%	GC
57	Endosulfan-alpha	0.01	0.05	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.001-0.1	0.9997	0.9991	0.9997	0.9993	5%	-16%	-6%	-2%	GC
58	Endosulfan-beta	0.01	0.05	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	0.9995	0.9978	1.0000	0.9986	3%	-36%	-14%	-13%	GC
59	EPN	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9998	0.9999	0.9989	8%	3%	-16%	-7%	GC
60	Epoxiconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9970	0.9932	0.9996	0.9978	6%	41%	-1%	4%	LC
61	Ethion	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9991	0.9908	1.0000	0.9947	7%	69%	29%	38%	LC
62	Ethirimol	0.05	0.01	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9999	0.9999	0.9984	2%	4%	3%	18%	LC
63	Etofenprox	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9995	0.9972	0.9978	-4%	-19%	-19%	-12%	GC
64	Etozazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9988	0.9961	0.9994	0.9980	20%	74%	35%	37%	LC
65	Famoxadone	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9984	0.9919	0.9971	0.9998	12%	67%	7%	26%	LC
66	Fenamidone	0.01	0.01	0.01	0.01	0.001-0.1	0.005-0.1	0.001-0.1	0.001-0.1	0.9995	0.9999	0.9994	0.9986	-5%	5%	13%	12%	GC
67	Fenamiphos	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9995	0.9985	0.9964	-1%	28%	-11%	-7%	LC
68	Fenarimol	0.01	0.01	0.01	0.01	0.005-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9993	0.9999	0.9996	0.9982	-47%	-15%	-6%	-5%	GC
69	Fenazaquin	0.01	0.01	0.01	0.01	0.01-0.1	0.005-0.1	0.005-0.1	0.001-0.1	0.9983	0.9999	0.9998	0.9979	-86%	-9%	8%	8%	GC
70	Fenbuconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9975	0.9938	0.9934	0.9995	11%	62%	9%	36%	LC
71	Fenhexamid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9962	0.9996	0.9949	10%	36%	11%	9%	LC
72	Fenitrothion	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9989	0.9999	0.9984	-7%	8%	2%	14%	GC
73	Fenoxycarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9933	0.9911	0.9997	0.9923	10%	64%	15%	17%	LC
74	Fenpropathrin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	1.0000	0.9995	0.9980	30%	9%	19%	15%	GC
75	Fenpropidin	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9982	0.9986	0.9992	0.9950	-1%	51%	9%	-18%	LC
76	Fenpropimorph	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9986	0.9927	0.9999	0.9982	3%	71%	8%	1%	LC
77	Fenpyroximate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9990	0.9947	0.9982	0.9933	0%	63%	25%	12%	LC



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78	Fenthion	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9961	0.9958	0.9996	0.9986	17%	70%	11%	57%	LC
79	Fenthion sulfone	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9990	0.9995	0.9994	0.9987	5%	24%	14%	16%	LC
80	Fenthion sulfoxide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9990	0.9998	0.9987	0%	-3%	2%	1%	LC
81	Fenvalerate	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9999	1.0000	NA	NA	8%	-8%	NA	NA	GC
82	Fipronil	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9986	1.0000	0.9989	0%	57%	0%	-12%	LC
83	Fipronil sulfone	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9993	0.9901	0.9975	0.9979	4%	62%	4%	-9%	LC
84	Flonicamid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9999	0.9995	0.9987	-1%	0%	3%	27%	LC
85	Fluazifop	0.05	0.01	0.05	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9945	0.9918	0.9969	0.9985	4%	-5%	-4%	-9%	LC
86	Flubendiamide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9988	0.9946	0.9913	0.9988	-14%	44%	10%	-12%	LC
87	Fludioxonil	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9979	0.9989	0.9988	12%	54%	9%	34%	LC
88	Flufenoxuron	N.F.R.	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9977	0.9985	0.9978	0.9930	-52%	43%	-14%	-29%	LC
89	Fluopicolide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9999	0.9995	0.9983	-3%	10%	30%	29%	GC
90	Fluopyram	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9999	0.9998	0.9978	23%	38%	21%	26%	GC
91	Fluquinconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9974	1.0000	0.9996	0.9989	-36%	-10%	-10%	-6%	GC
92	Flusilazol	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9993	0.9953	0.9997	0.9991	-2%	52%	1%	9%	LC
93	Flutriafol	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9996	0.9997	0.9987	-1%	6%	0%	15%	LC
94	Fluvalinate-tau	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9999	0.9996	0.9992	0%	-3%	-8%	-6%	GC
95	Fluxapyrosad	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9983	0.9994	0.9970	2%	30%	-7%	6%	LC
96	Phthalimide (Folpet)	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9988	0.9931	0.9976	66%	-101%	88%	99%	GC
97	Formetanate Hydrochloride	0.01	N.F.R.	0.01	N.F.R.	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9912	0.9983	0.9975	-56%	4%	2%	-58%	LC
98	Fosthiazate	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9990	0.9999	0.9985	2%	2%	-2%	8%	LC
99	Haloxyfop	NA	NA	N.F.R.	N.F.R.	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9921	0.9962	NA	NA	7%	-1%	LC
100	Hexaconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9981	0.9994	0.9994	15%	55%	9%	15%	LC
101	Hexythiazox	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9969	0.9976	0.9987	0.9981	23%	73%	33%	45%	LC
102	Imazalil	0.01	0.01	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9980	0.9903	0.9999	0.9973	13%	62%	0%	-4%	LC
103	Imidacloprid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9998	0.9999	0.9979	-2%	-4%	0%	4%	LC
104	Indoxacarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9977	0.9967	0.9991	0.9996	17%	69%	16%	29%	LC
105	Iprodione	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	GC

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106	Iprovalicarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9924	0.9995	0.9982	0.9997	-1%	24%	-5%	11%	LC
107	Isocarbophos	0.01	N.F.R.	0.01	0.05	0.001-0.1	0.01-0.1	0.001-0.1	0.01-0.1	0.9981	0.9937	0.9968	0.9960	1%	39%	7%	20%	LC
108	Isoprothiolane	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9993	0.9948	0.9999	0.9993	4%	50%	-3%	18%	LC
109	Kresoxim-methyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9941	0.9986	0.9977	6%	63%	-2%	10%	LC
110	Lambda-Cyhalothrin	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9998	0.9999	NA	NA	30%	15%	NA	NA	GC
111	Linuron	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9990	0.9967	0.9998	0.9993	1%	43%	6%	23%	LC
112	Lufenuron	0.05	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9977	0.9947	0.9984	0.9973	-69%	31%	-30%	-44%	LC
113	Malathion	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9995	1.0000	0.9976	-11%	-106%	18%	22%	GC
114	Mandipropamid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9928	0.9995	0.9961	5%	28%	2%	11%	LC
115	Mepanipyrim	0.01	0.05	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	0.9998	0.9968	0.9995	0.9986	31%	43%	26%	38%	GC
116	Metalaxyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9997	0.9997	0.9993	3%	1%	-6%	12%	LC
117	Methamidophos	0.05	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9994	1.0000	0.9988	0%	-2%	9%	27%	LC
118	Methidathion	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9951	0.9997	0.9990	4%	42%	-1%	12%	LC
119	Methiocarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9962	0.9953	0.9979	1%	37%	8%	12%	LC
120	Methiocarb sulfone	0.01	0.05	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9934	0.9932	0.9977	-45%	3%	-8%	-14%	LC
121	Methiocarb sulfoxide	0.01	0.05	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9902	0.9909	0.9974	-95%	-35%	-25%	-42%	LC
122	Methomyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9996	0.9997	0.9992	5%	1%	14%	32%	LC
123	Methoxyfenozide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9986	0.9999	0.9989	2%	34%	-6%	-8%	LC
124	Metrafenone	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9958	0.9992	0.9991	13%	73%	17%	21%	LC
125	Monocrotophos	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9997	1.0000	0.9988	4%	-1%	4%	21%	LC
126	Myclobutanil	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9995	0.9937	0.9994	3%	29%	2%	-4%	LC
127	Oxadixyl	0.01	0.05	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.001-0.1	0.9997	0.9987	0.9998	0.9983	-19%	-26%	4%	4%	GC
128	Oxamyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9968	0.9997	0.9983	3%	17%	1%	16%	LC
129	Paclobutrazol	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9981	0.9995	0.9993	14%	25%	10%	17%	LC
130	Parathion	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9988	0.9997	0.9977	13%	22%	11%	29%	GC
131	Parathion-methyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9988	0.9999	0.9982	-28%	2%	-3%	10%	GC
132	Penconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9976	0.9981	0.9997	0.9982	-2%	51%	-4%	17%	LC
133	Pencycuron	0.01	0.01	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9989	0.9997	0.9995	16%	72%	11%	27%	LC
134	Pendimethalin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9987	0.9959	0.9989	0.9966	9%	72%	27%	50%	LC

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135	Permethrin	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.005-0.1	0.005-0.1	0.9998	1.0000	0.9994	0.9991	20%	3%	0%	0%	GC
136	Phosmet	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9961	0.9978	0.9966	-5%	48%	-14%	-2%	LC
137	Pirimicarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9992	1.0000	0.9987	0%	1%	4%	0%	LC
138	Pirimiphos-methyl	NA	NA	0.01	0.05	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9988	0.9981	NA	NA	12%	11%	LC
139	Procymidone	0.01	0.01	0.01	0.01	0.005-0.1	0.005-0.1	0.001-0.1	0.001-0.1	1.0000	1.0000	0.9996	0.9983	2%	2%	-3%	5%	GC
140	Profenofos	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9928	0.9996	0.9972	18%	73%	14%	23%	LC
141	Propamocarb	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9971	0.9999	0.9966	-2%	-7%	3%	-6%	LC
142	Propargite	0.01	0.05	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9976	0.9965	NA	NA	16%	68%	NA	NA	LC
143	Propiconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9991	0.9917	0.9995	0.9956	-2%	50%	0%	2%	LC
144	Propyzamide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9996	0.9962	0.9989	0.9970	2%	49%	1%	26%	LC
145	Prosulfocarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9994	0.9952	0.9968	0.9985	15%	74%	15%	37%	LC
146	Prothioconazole	0.05	0.05	0.05	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9915	0.9926	0.9939	0.9912	90%	99%	23%	30%	LC
147	Pymetrozine	0.05	0.05	0.05	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9985	0.9973	0.9993	0.9988	-3%	-6%	6%	21%	LC
148	Pyraclostrobin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9931	0.9997	0.9997	2%	63%	-4%	14%	LC
149	Pyridaben	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9995	1.0000	NA	NA	11%	13%	NA	NA	GC
150	Pyrimethanil	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9972	0.9999	0.9999	0.9984	-20%	3%	8%	14%	GC
151	Pyriproxyfen	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9985	0.9941	0.9994	0.9955	15%	71%	33%	39%	LC
152	Quinoxifen	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9974	0.9961	0.9997	0.9990	12%	72%	20%	41%	LC
153	Spinosyn A	0.01	0.05	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9941	0.9973	0.9994	0.9911	10%	46%	14%	-13%	LC
154	Spinosyn D	0.01	0.01	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9934	0.9966	0.9993	0.9989	4%	48%	14%	-13%	LC
155	Spirodiclofen	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9992	0.9999	NA	NA	-11%	2%	NA	NA	GC
156	Spiromesifen	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9990	0.9984	0.9986	0.9955	12%	70%	32%	28%	LC
157	Spirotetramat	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9998	1.0000	0.9985	3%	10%	-2%	-15%	LC
158	Spiroxamine	0.01	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9984	0.9998	0.9976	4%	52%	7%	-18%	LC
159	Tebuconazole	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9994	0.9973	NA	NA	3%	45%	NA	NA	LC
160	Tebufenozide	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9978	0.9900	0.9973	0.9971	9%	41%	1%	5%	LC
161	Tebufenpyrad	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9998	0.9987	0.9982	40%	26%	27%	23%	GC
162	Teflubenzuron	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9988	0.9961	0.9991	0.9966	15%	67%	18%	29%	LC
163	Tefluthrin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	1.0000	0.9998	0.9976	-8%	5%	9%	13%	GC

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164	Terbutylazine	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9995	0.9995	0.9996	0.9980	4%	49%	4%	15%	LC	
165	Tetraconazole	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9984	0.9999	0.9964	3%	47%	1%	20%	LC	
166	Tetradifon	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	0.9998	0.9998	0.9985	8%	-14%	-8%	-10%	GC	
167	Thiabendazol	0.05	0.05	0.01	0.05	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9998	1.0000	0.9987	3%	2%	3%	13%	LC	
168	Thiacloprid	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9999	1.0000	1.0000	0.9987	-2%	3%	3%	14%	LC	
169	Thiamethoxam	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9998	0.9994	1.0000	0.9986	-3%	-5%	3%	15%	LC	
170	Thiodicarb	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9963	0.9973	0.9994	0.9950	4%	20%	10%	4%	LC	
171	Thiophanate-methyl	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	LC
172	Tolclofos-methyl	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	1.0000	0.9999	0.9978	-22%	-6%	-7%	-2%	GC	
173	Triadimefon	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9997	0.9999	0.9999	0.9981	-16%	5%	8%	15%	GC	
174	Triadimenol	0.01	0.01	NA	NA	0.001-0.1	0.001-0.1	NA	NA	0.9952	0.9986	NA	NA	12%	17%	NA	NA	LC	
175	Triazophos	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9973	0.9955	0.9982	0.9985	2%	53%	-6%	2%	LC	
176	Trifloxystrobin	0.01	0.01	0.01	0.01	0.001-0.1	0.005-0.1	0.005-0.1	0.001-0.1	0.9992	0.9997	0.9996	0.9982	16%	-5%	23%	21%	GC	
177	Triflumuron	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	0.9986	0.9914	0.9991	0.9995	18%	72%	20%	36%	LC	
178	Vinclozolin	0.01	0.01	0.01	0.01	0.001-0.1	0.001-0.1	0.001-0.1	0.001-0.1	1.0000	0.9998	0.9997	0.9989	-14%	4%	7%	15%	GC	

**LOQ:** limit of quantification

**R:** correlation coefficient

**NA:** Not analysed (present in the sample)

**N.F.R.:** Not fulfilling requirements for quantitative method (Recovery < 30 % and/or R.S.D. > 20 %)

**Q.C.:** QuEChERS

**Ac.:** Ethyl acetate

**LC:** Liquid chromatography coupled to tandem mass spectrometry

**GC:** Gas chromatography coupled to tandem mass spectrometry

**Table 7.** Limits of quantification, linearity range, coefficient of determination and matrix effects for the selected matrices studied and the Mini Luke method. Negative values of matrix effects mean suppression of the signal, and positives values, enhancement.

No.	Compound	LOQ (mg/kg)		Linear range (mg/kg)		R <sup>2</sup>		Matrix effect (%)		Method
		Chia seeds	Goji berries	Chia seeds	Goji berries	Chia seeds	Goji berries	Chia seeds	Goji berries	
1	2-Phenylphenol	0.01	0.01	0.005-0.1	0.001-0.1	0.9998	0.9969	17%	-92%	GC
2	Acrinathrin	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9958	32%	41%	GC
3	Azoxystrobin	0.01	0.01	0.005-0.1	0.001-0.1	1.0000	0.9993	-27%	11%	GC
4	Bifenthrin	0.01	0.01	0.005-0.1	0.001-0.1	1.0000	0.9958	0%	30%	GC
5	Biphenyl	0.01	0.05	0.001-0.1	0.001-0.1	0.9997	0.9986	3%	16%	GC
6	Boscalid	0.01	0.01	0.001-0.1	0.001-0.1	0.9993	0.9978	-28%	-6%	GC
7	Bromopropylate	0.01	0.01	0.001-0.1	0.001-0.1	0.9996	0.9978	-9%	20%	GC
8	Bupirimate	0.01	0.01	0.005-0.1	0.001-0.1	0.9976	0.9971	-17%	28%	GC
9	Buprofezin	0.01	0.01	0.001-0.1	0.001-0.1	0.9993	0.9987	-22%	23%	GC
10	Tetrahydrophthalimide (Captan)	0.01	0.01	0.001-0.1	0.001-0.1	0.9977	0.9961	18%	107%	GC
11	Chlorfenapyr	0.01	0.01	0.005-0.1	0.005-0.1	1.0000	0.9990	-24%	22%	GC
12	Chlorothalonil	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9984	-9%	12%	GC
13	Chlorpropham	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9981	-24%	23%	GC
14	Chlorpyrifos	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9976	-5%	21%	GC
15	Chlorpyrifos-methyl	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9980	-20%	-4%	GC
16	Cyfluthrin	0.01	0.01	0.001-0.1	0.001-0.1	0.9997	0.9970	-23%	-5%	GC
17	Cypermethrin	0.01	NA	0.005-0.1	NA	0.9997	NA	-25%	NA	GC
18	Cyproconazole	0.01	0.01	0.001-0.1	0.001-0.1	0.9995	0.9985	3%	-53%	GC
19	Cyprodinil	0.01	0.01	0.005-0.1	0.001-0.1	0.9993	0.9980	4%	33%	GC
20	Deltamethrin	0.01	0.01	0.005-0.1	0.005-0.1	0.9994	0.9998	-37%	-25%	GC
21	Diazinon	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9982	6%	26%	GC
22	Dicloran	0.01	0.01	0.005-0.1	0.005-0.1	0.9988	0.9992	-10%	28%	GC
23	Diclorvos	N.F.R.	N.F.R.	0.001-0.1	0.001-0.1	1.0000	0.9979	0%	24%	GC
24	Dieldrin	0.05	0.01	0.005-0.1	0.005-0.1	0.9945	0.9958	-49%	-16%	GC

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25	Endosulfan sulfate	0.01	0.01	0.001-0.1	0.001-0.1	0.9991	0.9919	-38%	-38%	GC
26	Endosulfan-alpha	0.01	0.01	0.005-0.1	0.001-0.1	0.9997	0.9975	-26%	-12%	GC
27	Endosulfan-beta	0.05	0.05	0.005-0.1	0.005-0.1	0.9998	0.9986	-47%	-29%	GC
28	EPN	0.01	0.01	0.001-0.1	0.001-0.1	0.9928	0.9978	-25%	10%	GC
29	Epoxiconazole	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9964	-40%	-25%	GC
30	Ethion	0.01	0.01	0.001-0.1	0.001-0.1	0.9997	0.9983	26%	58%	GC
31	Etofenprox	0.01	0.01	0.001-0.1	0.001-0.1	0.9993	0.9978	-32%	-9%	GC
32	Fenamidon	0.01	0.01	0.005-0.1	0.001-0.1	0.9995	0.9982	-6%	19%	GC
33	Fenarimol	0.01	0.01	0.001-0.1	0.001-0.1	0.9995	0.9989	-26%	1%	GC
34	Fenazaquin	0.01	0.01	0.005-0.1	0.001-0.1	0.9999	0.9987	-14%	16%	GC
35	Fenbuconazole	0.01	0.01	0.001-0.1	0.001-0.1	0.9993	0.9973	-35%	-11%	GC
36	Fenhexamid	0.05	0.01	0.005-0.1	0.005-0.1	0.9999	0.9996	29%	176%	GC
37	Fenitrothion	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9987	1%	41%	GC
38	Fenpropathrin	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9980	-1%	25%	GC
39	Fenpropidin	0.01	0.01	0.005-0.1	0.005-0.1	0.9983	0.9917	-64%	1%	GC
40	Fenpropimorph	0.01	0.01	0.001-0.1	0.001-0.1	0.9940	0.9956	-14%	20%	GC
41	Fenvalerate	0.01	NA	0.001-0.1	NA	0.9997	NA	-24%	NA	GC
42	Fludioxonil	0.01	0.01	0.005-0.1	0.001-0.1	0.9986	0.9973	10%	75%	GC
43	Fluopicolide	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9987	10%	41%	GC
44	Fluopyram	0.01	0.01	0.001-0.1	0.001-0.1	0.9987	0.9978	30%	45%	GC
45	Fluquinconazole	0.01	0.01	0.001-0.1	0.001-0.1	0.9994	0.9984	-22%	3%	GC
46	Flusilazole	0.01	0.01	0.001-0.1	0.001-0.1	1.0000	0.9984	-17%	27%	GC
47	Flutriafol	0.01	0.01	0.005-0.1	0.001-0.1	0.9994	0.9990	13%	48%	GC
48	Fluvalinate-tau	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9981	-20%	12%	GC
49	Phthalimide (Folpet)	0.01	0.01	0.001-0.1	0.001-0.1	0.9997	0.9936	-107%	130%	GC
50	Fosthiazate	0.05	0.01	0.005-0.1	0.005-0.1	0.9994	0.9948	-58%	-48%	GC
51	Hexaconazole	0.01	0.01	0.005-0.1	0.005-0.1	0.9982	0.9977	9%	51%	GC
52	Indoxacarb	0.01	0.01	0.001-0.1	0.001-0.1	0.9971	0.9997	-56%	-33%	GC
53	Iprodione	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	N.F.R.	GC

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54	Iprovalicarb	0.05	0.01	0.01-0.1	0.005-0.1	0.9974	0.9987	-56%	68%	GC
55	Isoprothiolane	0.01	0.01	0.005-0.1	0.001-0.1	0.9997	0.9992	-6%	37%	GC
56	Kresoxim-methyl	0.01	0.01	0.005-0.1	0.001-0.1	0.9996	0.9982	-27%	18%	GC
57	Lambda-Cyhalothrin	0.01	NA	0.001-0.1	NA	0.9999	NA	4%	NA	GC
58	Malathion	0.01	0.01	0.001-0.1	0.001-0.1	1.0000	0.9981	12%	47%	GC
59	Mepanipyrim	0.05	0.01	0.005-0.1	0.005-0.1	0.9911	0.9980	39%	63%	GC
60	Methidathion	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9979	-4%	11%	GC
61	Myclobutanil	0.01	0.01	0.005-0.1	0.001-0.1	0.9995	0.9990	-25%	20%	GC
62	Oxadixyl	0.01	0.01	0.005-0.1	0.001-0.1	0.9997	0.9988	-31%	-3%	GC
63	Paclobutrazol	0.01	0.01	0.005-0.1	0.001-0.1	0.9998	0.9983	-100%	78%	GC
64	Parathion	0.01	0.01	0.001-0.1	0.001-0.1	0.9994	0.9986	1%	63%	GC
65	Parathion-methyl	0.01	0.01	0.001-0.1	0.001-0.1	0.9997	0.9989	-11%	31%	GC
66	Penconazole	0.01	0.01	0.001-0.1	0.001-0.1	1.0000	0.9986	4%	24%	GC
67	Pendimethalin	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9979	11%	52%	GC
68	Permethrin	0.01	0.01	0.005-0.1	0.005-0.1	0.9998	0.9974	-15%	5%	GC
69	Phosmet	0.01	0.01	0.001-0.1	0.001-0.1	0.9962	0.9983	-9%	4%	GC
70	Procymidone	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9993	-3%	12%	GC
71	Profenofos	0.01	0.01	0.001-0.1	0.001-0.1	0.9995	0.9984	-17%	25%	GC
72	Propiconazole	0.01	0.01	0.005-0.1	0.005-0.1	0.9990	0.9989	13%	14%	GC
73	Propyzamide	0.01	0.01	0.001-0.1	0.001-0.1	1.0000	0.9984	-24%	25%	GC
74	Prosulfocarb	0.05	0.01	0.01-0.1	0.005-0.1	0.9975	0.9983	2%	25%	GC
75	Pyridaben	0.01	NA	0.001-0.1	NA	0.9997	NA	-4%	NA	GC
76	Pyrimethanil	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9995	4%	37%	GC
77	Pyriproxyfen	0.01	0.01	0.005-0.1	0.001-0.1	0.9997	0.9986	-14%	15%	GC
78	Quinoxifen	0.01	0.01	0.001-0.1	0.001-0.1	0.9996	0.9993	-13%	5%	GC
79	Spirodiclofen	0.01	NA	0.001-0.1	NA	0.9997	NA	-10%	NA	GC
80	Spiromesifen	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9989	-14%	7%	GC
81	Tebuconazole	0.01	NA	0.001-0.1	NA	0.9999	NA	2%	NA	GC
82	Tebufenpyrad	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9989	17%	42%	GC

83	Tefluthrin	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9980	8%	32%	GC
84	Tetraconazole	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9986	7%	44%	GC
85	Tetradifon	0.01	0.01	0.001-0.1	0.001-0.1	0.9998	0.9989	-25%	-9%	GC
86	Tolclofos-methyl	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9982	-7%	10%	GC
88	Triadimefon	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9980	1%	32%	GC
89	Triazophos	0.01	0.01	0.005-0.1	0.001-0.1	0.9999	0.9972	-52%	-20%	GC
90	Trifloxystrobin	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9985	-3%	16%	GC
91	Vinclozolin	0.01	0.01	0.001-0.1	0.001-0.1	0.9999	0.9984	4%	23%	GC

**LOQ:** limit of quantification

**R:** correlation coefficient

**NA:** Not analysed (present in the sample)

**N.F.R.:** Not fulfilling requirements for quantitative method (Recovery < 30 % and/or R.S.D. > 20 %)

**GC:** Gas chromatography coupled to tandem mass spectrometry