

Development and validation of a multiresidue method for high oil and intermediate water content commodities: olives



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1. Aim and scope

This document reports the validation data for 251 pesticides, most of them included in the European Union Multi Annual Control Program (EU-MACP) [1] and the Working Document SANCO/12745/2013 [2], using a multiresidue method by liquid chromatography coupled to triple quadrupole mass spectrometry (LC-MS/MS). An automatic extraction method based on automated pressurized liquid extraction employing the EDGE instrument has been developed for the extraction of these residues in olives.

2. Short description

A new automatic extraction method based on the use of EDGE has been validated for 251 pesticides in olives. With that purpose, homogeneous samples have been spiked at 0.010 mg/kg concentration level and extracted automatically. The EDGE combines the process of Pressurised Fluid Extraction (PFE) and Dispersive Solid Phase Extraction (dSPE) in one instrument. The obtained extracts have been analysed by LC-MS/MS.

The validation of the extraction method has been performed in terms of accuracy (recovery at 0.010 mg/kg), repeatability (5 replicates) and matrix effect. The recovery results have been compared to those obtained by extraction methods based on AcN (with a clean-up step or with freezing-out), showing a considerable improvement.

3. Apparatus and consumables

- Automatic pipettes, suitable for handling volumes from 1 µL to 5 mL
- Graduated 10 mL pipette
- 50 mL and 15 mL PTFE centrifuge tubes
- Vortex Shaker IKATM 4 Basic
- Axial shaker Agytax SR1 CP57
- Centrifuge Orto Alresa Consul 21, suitable for the centrifuge tubes employed in the procedure and capable of achieving 4000 rpm
- Concentration workstation
- Injection vials, 2 mL, suitable for LC auto-sampler
- Amber vials, 7 mL
- EDGE instrument supplied by CEM (Charlotte, North Carolina, United States of America)

- Q-Discs® (CEM) G1 and C9 varieties
- Q-Cups® (CEM)
- Laboratory grade fine sand (Panreac, Germany)

4. Chemicals

- Acetonitrile ultra-gradient grade (AcN)
- Bondesil - C18 (40 µm)
- Primary secondary amine (PSA)
- Anhydrous magnesium sulphate
- Ammonium formate
- Ultra-pure water
- Methanol HPLC grade (MeOH)
- Formic acid
- Pesticide analytical standards
- Dry ice

5. Procedure

5.1. Recovery experiments for method validation

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

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

5.2. Extraction methods

5.2.1. Automated sample extraction using EDGE

Four methods using EDGE were evaluated:

Method 01 (M01): Diaz-Galiano et al. [3] optimized this method previously, detailed hereafter. First, a layered Q-Disc® setup is placed in the Q-Cup in the following way: C9 (cellulose) Q-Disc®, then, G1 (glass fibre) Q-Disc® and, finally, a second C9 Q-Disc® on top of the G1 one. Following this step, 4 g of sample (after

cryogenic milling) are weighed into the Q-Cup and the sample is covered by approximately 5 g of fine laboratory grade sand. When weighing the sample into the Q-Cup, it is important to make the material sit as flat and as close to the bottom as possible, to ensure that the solvent will completely cover the matrix. The Q-Cup is placed into the EDGE instrument alongside a 50 mL PTFE falcon tube, which is used to collect the sample extract.

The solvent used is AcN in two consecutive cycles. During the first extraction cycle, the Q-Cup containing the sample is loaded within the instrument, sealed, and 10 mL AcN are added. The sample and the solvent are heated up to 40 °C, pressurized at 2 to 3 bar, these parameters are held for 150 sec. Afterwards, the 10 mL of AcN extract are transferred into the collection PTFE tube. In a second extraction cycle -the rinse cycle-, an additional volume of 5 mL can is added to the Q-Cup, let to mix with the sample for a short time, and then transferred into the collection PTFE tube. The total extraction volume is 15 mL. Finally, a wash step is performed by passing 10 mL of AcN at 40 °C to prepare the EDGE instrument for the next sample extraction.

Method 02 (M02): the method employs the same parameters as M01, except for an additional 60 s bubbling step with N₂ during the first extraction cycle.

Method 03 (M03): this method is comprised of three extraction cycles. During the first two, the Q-Cup containing the sample is loaded within the instrument, sealed, and 5 mL AcN are added. The sample and the solvent are heated up to 40 °C, pressurized at 2 to 3 bar, these parameters are held for 90 sec. Afterwards, the 10 mL of AcN extract are transferred into the collection PTFE tube. In a third extraction cycle -the rinse cycle-, an additional volume of 5 mL AcN is added to the Q-Cup, let to mix with the sample for a short time, and then transferred into the collection PTFE tube. The total extraction volume is 15 mL. Finally, a wash step is performed by passing 10 mL of AcN at 40 °C to prepare the EDGE instrument for the next sample extraction

Method 04 (M04): the method employs the same parameters as M03 together with bubbling a N₂ step in the first two cycles of 30 sec each one.

After the automated extraction procedure with every one of the aforementioned methods, a freezing-out step was carried out. The PTFE tubes were cooled with dry ice for 20 min, and then centrifuged at 3500 RPM for 1 min. The highest number of target pesticides within the 70-120 % recovery range was obtained by using M02 (Table 1).

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

Automatic extraction methods	M01	M02	M03	M04
% of compounds with recovery (70-120 %)	13	97	16	72

5.2.2 Manual extraction methods based on AcN

Two different extraction methods were evaluated based on a previous study of our research group, where 2 g of dry commodity samples were extracted with 10 mL of AcN [4].

Method with clean-up step:

1. Weigh 2 g of homogenate sample (after cryogenic milling) in a 50-mL PTFE centrifuge tube.
2. Add 10 mL of acetonitrile.
3. Shake the samples in an Agytax axial extractor for 4 min.
4. Centrifuge at 4000 rpm for 5 min.
5. Transfer a 4-mL aliquot of the supernatant to a 15 mL PTFE tube containing 100 mg anhydrous magnesium sulphate, 100 mg PSA and 100 mg C18.
6. Vortex the tubes for 30 sec.
7. Centrifuge at 4000 rpm for 5 min.
8. Acidify with 10 µL formic acid 5 % per mL of extract.

Method with freezing-out:

1. Weigh 2 g of homogenate sample (after cryogenic milling) in a 50-mL PTFE centrifuge tube.
2. Add 10 mL of acetonitrile.
3. Shake the samples in an Agytax axial extractor for 4 min.
4. Centrifuge at 4000 rpm for 5 min.
5. Transfer a 4-mL aliquot of the supernatant to a 15 mL PTFE tube and store with dry ice for 20 min
6. Centrifuge at 3500 RPM for 1 min.

5.3. Vial preparation

During the vial preparation, sample extracts were diluted 5-fold with ultrapure water of extracts and dimethoate-D₆ (LC) was added as an internal injection standard (0.050 mg/L).

5.4. Methodology

The LC-MS/MS system was operated in selected reaction monitoring (SRM). First, full scan (FS) analyses were carried out to select the most sensitive precursor ions. Then, product ion scans (PIS) were performed to select the most abundant product ions. Finally, two SRM transitions and the correct ratio between the abundances of the two optimised SRM transitions (SRM1/SRM2) were used, alongside retention time matching, to obtain the maximum sensitivity for the detection of the target molecules. The mass transitions used are presented in Appendix I (**Table A1**).

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

5.5.1. UHPLC (Thermo Scientific™ Transcend™ DUO LX-2 LC)

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

Mobile phase gradient for pesticides analysis:

Time [min]	Mobile phase A
0	100 %
1	100 %
2	70 %
3	50 %
11	0 %
14	0 %
14.1	100 %
17	100 %
Data window [min]	1.1-11.55

5.5.2. Triple quadrupole system (Transcend DUO LX-2. A TSQ Altis, Thermo Scientific)

Ion source: Opta Max NG

- Positive ion spray voltage: 3500 V
- Negative ion spray voltage: 2500 V
- Sheath gas: 50
- Aux gas: 10
- Sweep gas: 1
- Ion transfer tube temperature: 25 °C
- Vaporiser temperature: 350 °C

6. Results

6.1. Method validation

6.1.1. Recoveries and within-laboratory reproducibility

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

The relative standard deviation was found to be lower than 20 % for all compounds in the five replicates. Most recovery results are within the range 70-120 %. **Figure 1** summarises the recovery results obtained by the optimal automated sample extraction M02.

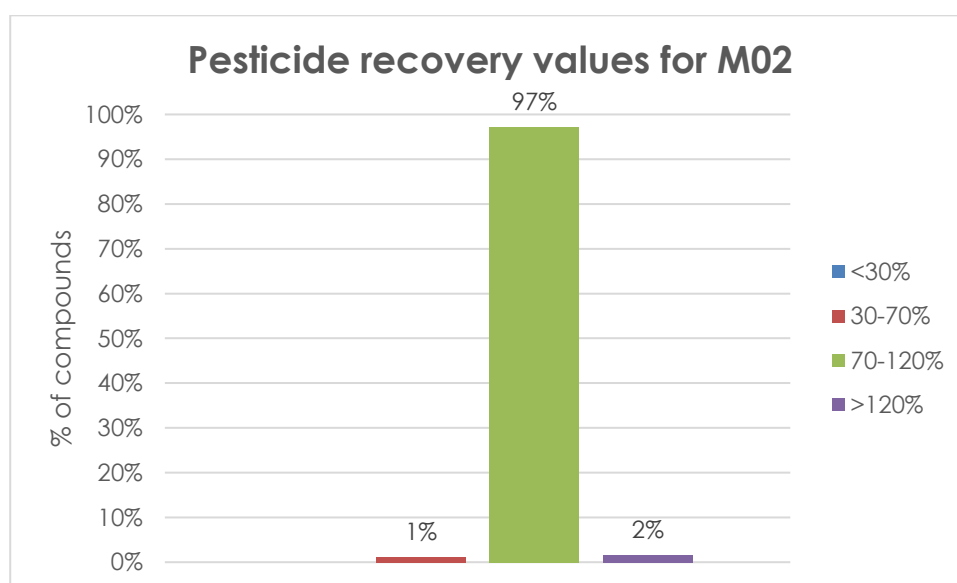


Figure 1. Percentage of compounds in each recovery range for M02.

6.1.2. Matrix effects

Matrix effects were assessed by comparison of the slopes of eight-point matrix-matched calibration curves with the slopes of the calibration curves in solvent. For values (in absolute terms) between 0 and 20 %, matrix effect is considered low; between 20 % and 50 % there is a moderate matrix effect, and for compounds with a value over 50 % matrix effect is classified as strong. These values of matrix effects are summarized in **Appendix I, Table A3**, and they are also represented in **Figure 2**.

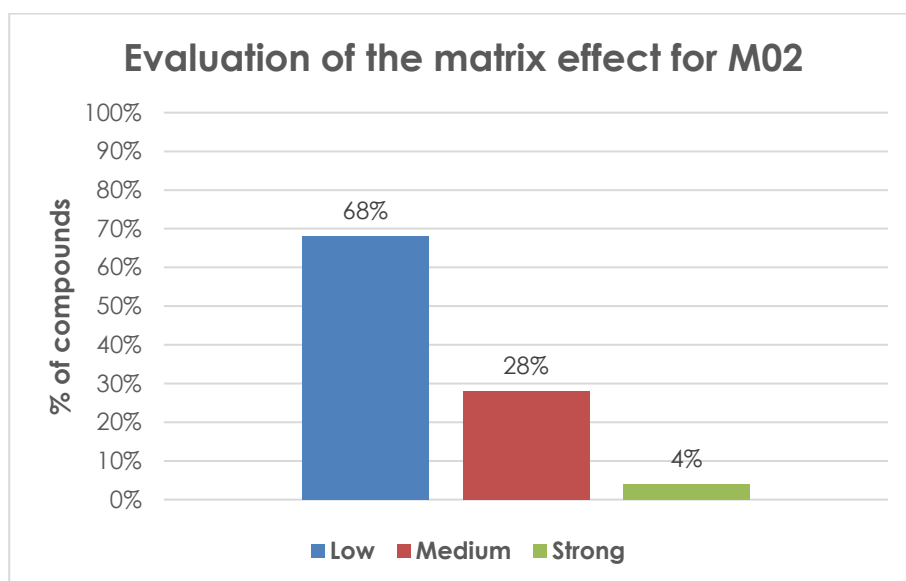


Figure 2. Matrix effects for the target analytes using M02.

6.2. Comparison with manual extraction method based on AcN

The automated extraction method M02 has been compared to manual extraction methodologies in terms of method accuracy (as recovery (%)) of the compounds included in the scope) and matrix effects. The results of all pesticide residues included in the scope for LC-MS/MS are summarized in **Figure 3** and **Figure 4** below in terms of recovery. It can be observed that the percentage of compounds with recovery values between 70 and 120 % is higher in the automated, EDGE-based extractions. In the case of the manual extraction method with a clean-up step or with a freezing-out step, the percentage of compounds with recovery values between 30 and 70 % is higher than in the case of M02 (4 % vs. 1 %). Regarding the manual extraction method with a freezing-out step, the percentage of compounds with recovery values above 120 % was found to be the highest, with 9 % of the target analytes above 120 % recovery values.

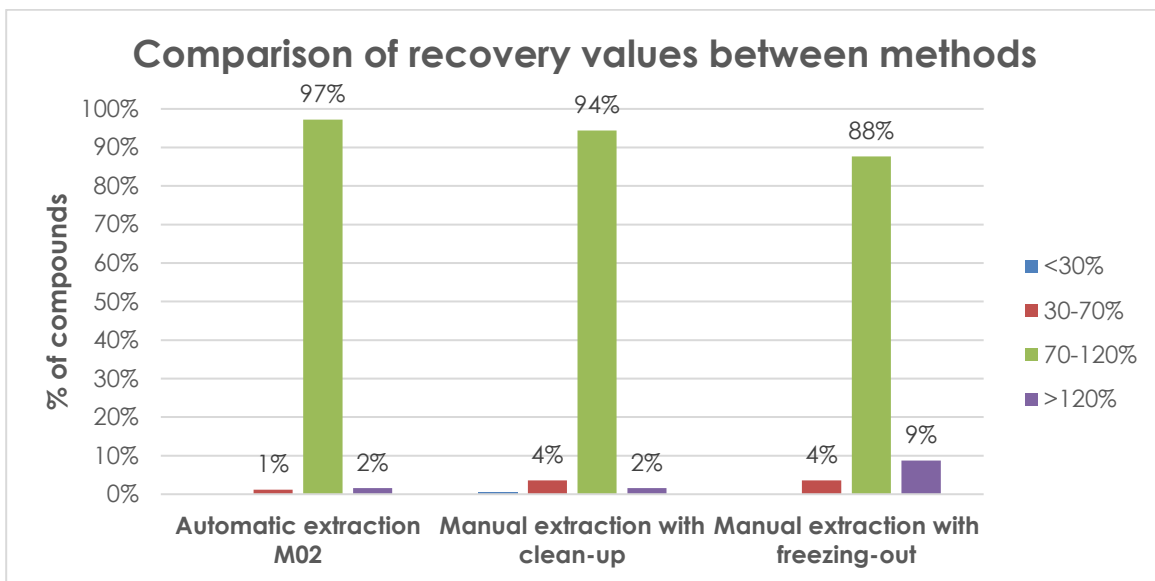


Figure 3. Recovery values for M02 and two manual methods.

Matrix effects were lower in average for the M02 automated extraction methods, with only 4 % of the target analytes presenting a strong matrix effect, and 68 % of the target analytes with low or negligible matrix effect. In the case of the manual extraction methods, both using a clean-up step or a freezing-out step resulted in 8 % of the target analytes presenting a strong matrix effect, with up to 34 % of them presenting a medium or moderate matrix effect in the case of the manual extraction with a freezing-out step (**Figure 4**). Furthermore, the appearance of extract can be seen in **Figure 5** and detailed information can be found in **Appendix I (Table A3)**.

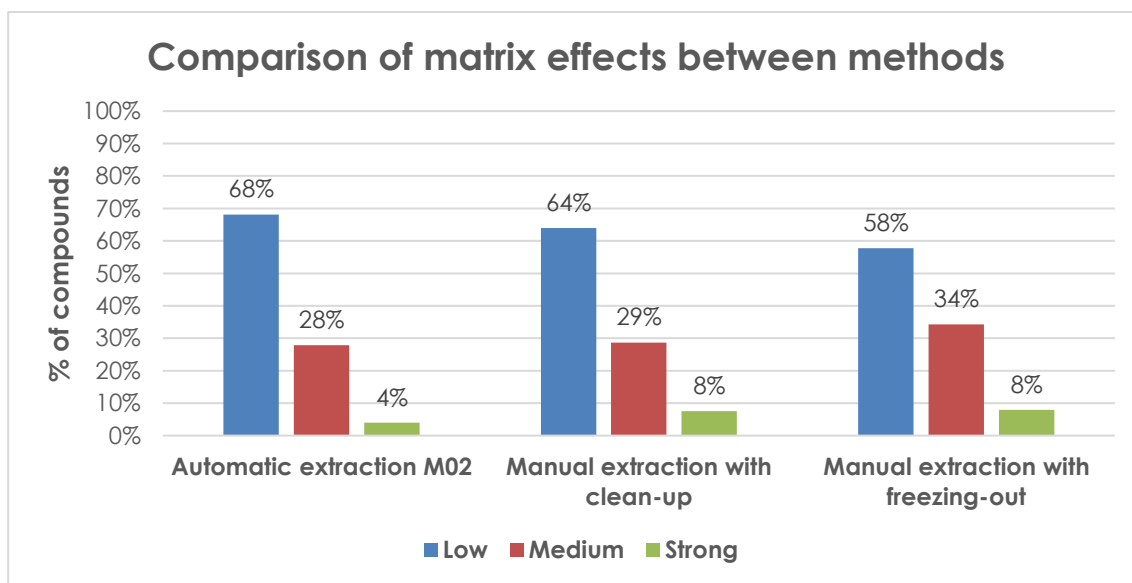


Figure 4. Matrix effects for M02 and two manual methods.

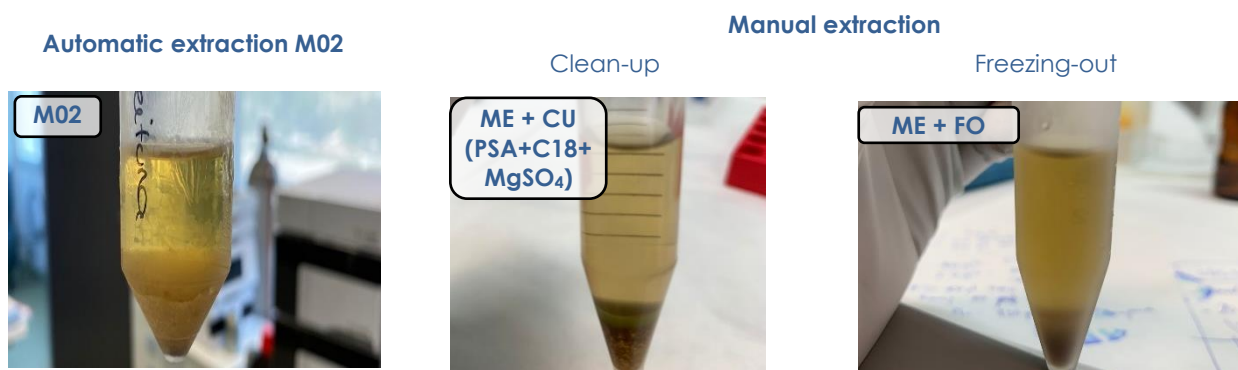


Figure 5. Extracts of olives samples with automatic extraction (M02) -which includes a freezing-out step (FO) and manual extraction (ME) (with clean-up (CU) step or with FO).

6.3. Conclusion

A new automated extraction method based on pressurised liquid extraction and LC-MS/MS has been developed for the analysis of olive samples. Four different automated extraction methods were evaluated, and the best performing was selected (M02). This method was compared to a manual extraction methodology using either a clean-up step based on PSA, C18 and magnesium sulphate or a freezing-out step. The automated method M02 performed better than either manual extraction method, with 97 % of the target analytes within the 70-120 % recovery range and up to 68 % of them presenting low or negligible matrix effect.

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] Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin. Document N° SANCO/12745/2013.
- [3] Díaz-Galiano, F. J.; Murcia-Morales, M.; Gómez-Ramos, M. M.; Ferrer, C.; Fernández-Alba, A. R. Presence of anthraquinone in coffee and tea samples. An

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APPENDIX I: MASS TRANSITIONS AND VALIDATION RESULTS

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

Compound	Relative Retention Time (min) 100 mm column	RT Window (min)	Polarity	Precursor (m/z)	Product (m/z)	Collision Energy (V)	RF Lens (V)
2,4-D	4.47	0.5	Negative	218.962	125	27.4	36
2,4-D	4.47	0.5	Negative	218.962	160.958	12.24	36
Acephate	1.42	0.5	Positive	184.019	48.845	20.58	30
Acephate	1.42	0.5	Positive	184.019	142.863	10.23	30
Acetamiprid	2.74	0.5	Positive	223.074	55.786	16.52	45
Acetamiprid	2.74	0.5	Positive	223.074	125.702	21.18	45
Alachlor	6.30	2	Positive	270.125	162	20.35	33
Alachlor	6.30	2	Positive	270.125	238	10.23	33
Albendazole	4.79	0.5	Positive	266.095	190.845	32.86	50
Albendazole	4.79	0.5	Positive	266.095	233.929	19.67	50
Aldicarb	3.21	0.5	Positive	116.053	61.082	13.72	30
Aldicarb	3.21	0.5	Positive	116.053	88.97	9.51	30
Aldicarb-sulfone	1.95	0.5	Positive	240.101	86	10.23	30
Aldicarb-sulfone	1.95	0.5	Positive	240.101	147.97	14.09	30
Aldicarb-sulfoxide	1.82	0.5	Positive	207.079	88.988	13.18	27
Aldicarb-sulfoxide	1.82	0.5	Positive	207.079	131.97	10.23	27
Ametoctradin	7.43	0.5	Positive	276.218	176	37.71	70
Ametoctradin	7.43	0.5	Positive	276.218	176.97	28.99	70
Anilofos	6.91	0.5	Positive	368.03	124.845	31.27	45
Anilofos	6.91	0.5	Positive	368.03	198.762	14.21	45
Atrazine	4.38	0.5	Positive	216.101	103.917	28.35	50
Atrazine	4.38	0.5	Positive	216.101	173.97	17.35	50
Azinphos-ethyl	6.07	0.5	Positive	346.044	131.917	16.07	33
Azinphos-ethyl	6.07	0.5	Positive	346.044	260.887	10.23	33
Azinphos-methyl	5.02	0.5	Positive	318.013	131.804	14.3	36
Azinphos-methyl	5.02	0.5	Positive	318.013	167.016	11.3	36
Azoxystrobin	5.19	0.5	Positive	404.124	343.929	25.35	45
Azoxystrobin	5.19	0.5	Positive	404.124	371.929	14.74	45
BAC 10	5.79	0.5	Positive	276.268	91.054	27.96	50
BAC 10	5.79	0.5	Positive	276.268	184.292	19.45	50
BAC 8	4.29	0.5	Positive	248.237	91.054	26.82	50
BAC 8	4.29	0.5	Positive	248.237	156.208	18.4	50
Benalaxyl	6.88	0.5	Positive	326.175	148	21.75	40
Benalaxyl	6.88	0.5	Positive	326.175	293.929	10.23	40
Bendiocarb	3.70	0.5	Positive	224.091	108.774	17.47	33
Bendiocarb	3.70	0.5	Positive	224.091	166.857	10.23	33
Bifenazate	5.98	0.5	Positive	301.154	170	19.36	33

Bifenazate	5.98	0.5	Positive	301.154	197.857	10.23	33
Bifenthrin	9.96	0.5	Positive	440.088	166.125	40.18	45
Bifenthrin	9.96	0.5	Positive	440.088	181.125	12.69	45
Bitertanol	7.27	0.5	Positive	338.186	69.929	10.23	36
Bitertanol	7.27	0.5	Positive	338.186	269.012	10.23	36
Boscalid	5.52	0.5	Positive	343.039	270.94	31.76	90
Boscalid	5.52	0.5	Positive	343.039	306.845	19.86	90
Bromacil	3.69	0.5	Positive	261.023	187.762	28.54	40
Bromacil	3.69	0.5	Positive	261.023	204.762	14.47	40
Bromuconazole	6.30	1.5	Positive	375.961	69.917	20.8	60
Bromuconazole	6.30	1.5	Positive	375.961	158.899	28.16	60
BTS_44595	7.13	0.5	Positive	325.027	129	17.89	50
BTS_44595	7.13	0.5	Positive	325.027	281.97	15.4	50
BTS_44596	6.98	0.5	Positive	353.022	265.958	17.64	56
BTS_44596	6.98	0.5	Positive	353.022	308.042	13.8	56
BTS-40348	3.74	0.5	Positive	282.021	44.054	21.93	60
BTS-40348	3.74	0.5	Positive	282.021	86.125	17.59	60
Bupirimate	5.98	0.5	Positive	317.164	165.929	24.67	60
Bupirimate	5.98	0.5	Positive	317.164	237.012	19.89	60
Buprofezin	8.01	0.5	Positive	306.163	115.845	16.56	40
Buprofezin	8.01	0.5	Positive	306.163	200.857	12.54	40
Butoxycarboxim	1.90	0.5	Positive	223.074	62.845	10.99	30
Butoxycarboxim	1.90	0.5	Positive	223.074	105.845	11.48	30
Carbaryl	3.96	0.5	Positive	202.086	126.929	28.96	30
Carbaryl	3.96	0.5	Positive	202.086	144.929	10.23	30
Carbendazim	2.18	0.5	Positive	192.076	131.929	30.7	40
Carbendazim	2.18	0.5	Positive	192.076	159.929	18.15	40
Carbofuran	3.69	0.5	Positive	222.112	122.845	22.02	36
Carbofuran	3.69	0.5	Positive	222.112	164.929	12.43	36
Chlorantraniliprole	4.93	0.5	Positive	481.978	283.875	12.69	50
Chlorantraniliprole	4.93	0.5	Positive	481.978	450.815	16.82	50
Chlorbromuron	5.52	0.5	Positive	292.968	181.845	16.79	50
Chlorbromuron	5.52	0.5	Positive	292.968	203.69	19.71	50
Chlorfenvinphos	7.01	0.5	Positive	358.976	98.774	28.92	45
Chlorfenvinphos	7.01	0.5	Positive	358.976	154.899	12.88	45
Chlorfluazuron	9.11	0.5	Positive	539.97	157.97	19.06	60
Chlorfluazuron	9.11	0.5	Positive	539.97	382.833	20.08	60
Chloridazon	2.79	0.5	Positive	222.042	76.929	33.73	60
Chloridazon	2.79	0.5	Positive	222.042	103.845	23.16	60
Chlorotoluron	4.32	0.5	Positive	213.078	71.857	18.95	45
Chlorotoluron	4.32	0.5	Positive	213.078	139.845	25.09	45
Chloroxuron	6.01	0.5	Positive	291.089	71.917	20.77	50
Chloroxuron	6.01	0.5	Positive	291.089	163.929	17.24	50
Chlorpyrifos	8.51	0.5	Positive	349.933	197.762	19.4	50

Chlorpyrifos	8.51	0.5	Positive	349.933	321.679	12.05	50
Chromafenozide	6.04	0.5	Positive	395.232	174.982	16.96	36
Chromafenozide	6.04	0.5	Positive	395.232	339.149	7.4	36
Clofentezine	7.27	0.5	Positive	303.019	101.845	34.41	36
Clofentezine	7.27	0.5	Positive	303.019	137.845	14.97	36
Clomazone	4.97	0.5	Positive	240.078	88.899	46.28	40
Clomazone	4.97	0.5	Positive	240.078	124.845	21.11	40
Clothianidin	2.58	0.5	Positive	250.015	131.762	16.82	36
Clothianidin	2.58	0.5	Positive	250.015	168.899	12.99	36
Coumaphos	6.91	0.5	Positive	363.021	226.774	26.07	60
Coumaphos	6.91	0.5	Positive	363.021	306.762	17.85	60
Cyazofamid	6.33	0.5	Positive	325.052	107.774	14.28	40
Cyazofamid	6.33	0.5	Positive	325.052	260.929	10.23	40
Cyflufenamid	7.24	0.5	Positive	413.128	240.917	23.19	50
Cyflufenamid	7.24	0.5	Positive	413.128	294.899	15.5	50
Cyprodinil	6.24	0.5	Positive	226.133	92.857	34.98	60
Cyprodinil	6.24	0.5	Positive	226.133	107.857	26.68	60
Cyromazine	0.60	2	Positive	167.103	67.988	34.57	40
Cyromazine	0.60	2	Positive	167.103	125	19.14	40
DEET	4.49	0.5	Positive	192.138	90.929	29.64	45
DEET	4.49	0.5	Positive	192.138	118.845	17.89	45
Demeton-S-methyl	3.77	0.5	Positive	231.027	60.917	30.36	27
Demeton-S-methyl	3.77	0.5	Positive	231.027	88.899	10.23	27
Demeton-S-methylsulfone	2.19	0.5	Positive	263.017	124.845	23.5	50
Demeton-S-methylsulfone	2.19	0.5	Positive	263.017	168.845	16.37	50
Demeton-S-methylsulfoxide	2.10	0.5	Positive	247	109.042	27.07	40
Demeton-S-methylsulfoxide	2.10	0.5	Positive	247	169.054	14.27	40
Diazinon	6.93	0.5	Positive	305.11	153.208	21.26	50
Diazinon	6.93	0.5	Positive	305.11	169.137	21.09	50
Dichlorvos	3.65	0.5	Positive	220.938	108.774	17.85	50
Dichlorvos	3.65	0.5	Positive	220.938	144.845	14.02	50
Dicrotophos	2.41	0.5	Positive	238.08	112.125	12.71	36
Dicrotophos	2.41	0.5	Positive	238.08	126.97	18.35	36
Diethofencarb	5.18	0.5	Positive	268.154	179.929	18.26	33
Diethofencarb	5.18	0.5	Positive	268.154	225.929	10.23	33
Difenoconazole	7.51	0.5	Positive	406.071	250.845	25.7	60
Difenoconazole	7.51	0.5	Positive	406.071	336.845	17.62	60
Difenoxyuron	4.66	0.5	Positive	287	71.929	20.35	50
Difenoxyuron	4.66	0.5	Positive	287	122.899	19.89	50
Diflubenzuron	6.60	0.5	Positive	311.039	140.917	32.1	36
Diflubenzuron	6.60	0.5	Positive	311.039	157.97	14.06	36
Dimethoate	2.77	0.5	Positive	230.006	124.845	21.75	33
Dimethoate	2.77	0.5	Positive	230.006	198.762	10.23	33
Dimethomorph	5.50	1.5	Positive	388.131	164.929	31.61	60

Dimethomorph	5.50	1.5	Positive	388.131	300.845	20.73	60
Dimethylvinphos	5.93	0.5	Positive	331	127.042	13.51	50
Dimethylvinphos	5.93	0.5	Positive	331	170.042	36.34	50
Diniconazole	7.46	0.5	Positive	326.082	69.929	25.47	70
Diniconazole	7.46	0.5	Positive	326.082	158.887	30.47	70
Diuron	4.70	0.5	Positive	233.024	71.917	18.87	50
Diuron	4.70	0.5	Positive	233.024	159.815	26.95	50
DMF	3.41	0.5	Positive	150.091	106.125	33.14	55
DMF	3.41	0.5	Positive	150.091	107.125	21.43	55
Dodine	7.19	0.5	Positive	228.264	56.929	23.99	60
Dodine	7.19	0.5	Positive	228.264	59.929	23.8	60
Edifenphos	6.76	0.5	Positive	311.032	108.845	32.18	45
Edifenphos	6.76	0.5	Positive	311.032	282.845	13.79	45
Emamectin B1a	8.26	0.5	Positive	886.538	125.929	38.24	90
Emamectin B1a	8.26	0.5	Positive	886.538	158	35.32	90
Epoxiconazole	6.25	0.5	Positive	330.08	100.917	43.32	50
Epoxiconazole	6.25	0.5	Positive	330.08	120.899	21.3	50
Ethiofencarb	4.14	0.5	Positive	226.089	106.845	15.72	30
Ethiofencarb	4.14	0.5	Positive	226.089	164	10.23	30
Ethion	8.34	0.5	Positive	384.994	142.762	25.13	40
Ethion	8.34	0.5	Positive	384.994	198.774	10.23	40
Ethiprole	5.51	0.5	Negative	394.975	261.845	28.8	50
Ethiprole	5.51	0.5	Negative	394.975	330.917	10.23	50
Ethirimol	3.15	0.5	Positive	210.16	97.97	27.44	50
Ethirimol	3.15	0.5	Positive	210.16	140	22.55	50
Ethoprofos	6.15	0.5	Positive	243.063	130.905	20.35	36
Ethoprofos	6.15	0.5	Positive	243.063	172.958	14.63	36
Etofenprox	9.87	0.5	Positive	394.24	135.156	26	33
Etofenprox	9.87	0.5	Positive	394.24	359.174	12	33
Etoxazol	8.69	0.5	Positive	360.176	140.845	30.93	60
Etoxazol	8.69	0.5	Positive	360.176	303.929	18.23	60
Famoxadone	7.11	0.5	Positive	392.16	237.911	17.28	36
Famoxadone	7.11	0.5	Positive	392.16	330.911	10.23	36
Fenamidone	5.42	0.5	Positive	312.116	91.899	24.71	50
Fenamidone	5.42	0.5	Positive	312.116	235.929	15.08	50
Fenamiphos	6.51	0.5	Positive	304.113	216.845	23.27	50
Fenamiphos	6.51	0.5	Positive	304.113	233.917	17.13	50
Fenamiphos-sulfone	3.87	0.5	Positive	336.102	265.917	20.12	60
Fenamiphos-sulfone	3.87	0.5	Positive	336.102	307.917	15.72	60
Fenamiphos-sulfoxide	3.71	0.5	Positive	320.107	232.845	25.05	50
Fenamiphos-sulfoxide	3.71	0.5	Positive	320.107	291.899	16.26	50
Fenarimol	6.14	0.5	Positive	331.039	188.845	47.19	70
Fenarimol	6.14	0.5	Positive	331.039	267.917	22.93	70
Fenazaquin	9.27	0.5	Positive	307.18	146.917	19.74	50

Fenazaquin	9.27	0.5	Positive	307.18	161.054	16.98	50
Fenbuconazole	6.46	0.5	Positive	337.121	88.899	55	60
Fenbuconazole	6.46	0.5	Positive	337.121	124.845	30.24	60
Fenhexamide	6.07	0.5	Positive	302.07	55.054	34.23	60
Fenhexamide	6.07	0.5	Positive	302.07	97.208	23.28	60
Fenobucarb	5.21	0.5	Positive	208.133	94.917	15.08	30
Fenobucarb	5.21	0.5	Positive	208.133	151.97	10.23	30
Fenoxycarb	6.60	0.5	Positive	302.138	87.97	18.98	40
Fenoxycarb	6.60	0.5	Positive	302.138	115.857	10.68	40
Fenpropidin	4.76	0.5	Positive	274.252	116.857	52.12	70
Fenpropidin	4.76	0.5	Positive	274.252	146.929	29.07	70
Fenpropimorph	4.95	0.5	Positive	304.263	130.054	25.43	70
Fenpropimorph	4.95	0.5	Positive	304.263	147.054	28.88	70
Fenpyrazamine	5.85	0.5	Positive	332.142	230.083	19.25	45
Fenpyrazamine	5.85	0.5	Positive	332.142	231.054	18.11	45
Fenpyroximate	8.87	0.5	Positive	422.207	137.929	31.53	60
Fenpyroximate	8.87	0.5	Positive	422.207	366.012	16.03	60
Fenthion	6.89	0.5	Positive	279.027	168.899	18.3	60
Fenthion	6.89	0.5	Positive	279.027	246.845	13.37	60
Fenthion-oxon-sulfone	3.86	0.5	Positive	295.039	104.137	25.94	50
Fenthion-oxon-sulfone	3.86	0.5	Positive	295.039	263.095	20.59	50
Fenthion-sulfone	4.07	0.5	Positive	311.017	124.917	20.05	70
Fenthion-sulfone	4.07	0.5	Positive	311.017	278.917	17.92	70
Fenthion-sulfoxide	3.86	0.5	Positive	295.022	263.845	16.75	60
Fenthion-sulfoxide	3.86	0.5	Positive	295.022	279.845	18.98	60
Fipronil	6.67	0.5	Negative	434.945	329.845	15.46	90
Fipronil	6.67	0.5	Negative	434.945	398.815	10.23	90
Fipronil-sulfone	7.09	0.5	Negative	450.926	282	26.19	80
Fipronil-sulfone	7.09	0.5	Negative	450.926	414.917	15.38	80
Flazasulfuron	5.02	0.5	Positive	408.058	139	39.12	50
Flazasulfuron	5.02	0.5	Positive	408.058	181.97	18.42	50
Flonicamid	2.17	0.5	Positive	230.053	173.917	18.34	50
Flonicamid	2.17	0.5	Positive	230.053	202.845	17.35	50
Fluacypirim	7.49	0.5	Positive	427.062	144.982	24.92	36
Fluacypirim	7.49	0.5	Positive	427.062	205.196	9.34	36
Fluazifop	5.44	0.5	Positive	328.079	253.929	26.83	50
Fluazifop	5.44	0.5	Positive	328.079	281.929	19.1	50
Flubendiamide	6.79	0.5	Positive	683.03	273.833	31.38	50
Flubendiamide	6.79	0.5	Positive	683.03	407.887	10.23	50
Fludioxonil	5.57	0.5	Negative	247.032	168.857	32.78	100
Fludioxonil	5.57	0.5	Negative	247.032	179.857	28.69	100
Flufenacet	6.19	0.5	Positive	364.073	151.97	18.87	40
Flufenacet	6.19	0.5	Positive	364.073	193.929	10.23	40
Flufenoxuron	8.84	0.5	Positive	489.043	140.917	42.83	60

Flufenoxuron	8.84	0.5	Positive	489.043	157.97	18.95	60
Fluometuron	4.24	0.5	Positive	233.089	71.917	19.1	45
Fluometuron	4.24	0.5	Positive	233.089	159.899	27.44	45
Fluopicolide	5.63	0.5	Positive	382.97	109.042	55	60
Fluopicolide	5.63	0.5	Positive	382.97	173.042	23.87	60
Fluopyram	6.03	0.5	Positive	397.053	172.845	28.69	60
Fluopyram	6.03	0.5	Positive	397.053	207.845	21.9	60
Fluquinconazole	6.03	0.5	Positive	376.016	307.042	26.23	60
Fluquinconazole	6.03	0.5	Positive	376.016	349.042	19.7	60
Flusilazole	6.56	0.5	Positive	316.107	164.929	27.25	70
Flusilazole	6.56	0.5	Positive	316.107	246.929	18.34	70
Flutriafol	4.49	0.5	Positive	302.109	94.917	47.91	50
Flutriafol	4.49	0.5	Positive	302.109	122.845	28.01	50
Fluxapyroxad	5.74	0.5	Positive	382.097	341.929	21.18	50
Fluxapyroxad	5.74	0.5	Positive	382.097	362	14.4	50
Formetanate-hydrochloride	1.62	0.5	Positive	222.123	92.845	35.44	50
Formetanate-hydrochloride	1.62	0.5	Positive	222.123	165	15.57	50
Fosthiazate	4.17	0.5	Positive	284.053	103.845	21.71	33
Fosthiazate	4.17	0.5	Positive	284.053	227.845	10.23	33
Haloxypop	6.74	0.5	Positive	362.04	287.845	27.25	60
Haloxypop	6.74	0.5	Positive	362.04	315.649	18.53	60
Hexaconazole	7.16	0.5	Positive	314.082	69.899	20.77	50
Hexaconazole	7.16	0.5	Positive	314.082	158.845	31.15	50
Hexaflumuron	7.75	0.5	Positive	460.988	140.988	38.4	45
Hexaflumuron	7.75	0.5	Positive	460.988	158.042	17.58	45
Hexythiazox	8.48	0.5	Positive	353.108	168.042	24.18	45
Hexythiazox	8.48	0.5	Positive	353.108	227.988	14.66	45
Imazalil	4.17	0.5	Positive	297.055	158.958	23.04	60
Imazalil	4.17	0.5	Positive	297.055	200.863	17.77	60
Imidacloprid	2.53	0.5	Positive	256.059	175.071	18.15	45
Imidacloprid	2.53	0.5	Positive	256.059	209.006	15.72	45
Indoxacarb	7.62	0.5	Positive	528.077	149.97	24.14	60
Indoxacarb	7.62	0.5	Positive	528.077	248.97	16.71	60
Ioxynil	4.84	0.5	Negative	369.823	126.958	33	100
Ioxynil	4.84	0.5	Negative	369.823	214.958	32	100
Iprovalicarb	5.99	0.5	Positive	321.217	119.018	19.48	36
Iprovalicarb	5.99	0.5	Positive	321.217	203.071	10.23	36
Isocarbofos	4.67	0.5	Positive	307	230.917	15.35	30
Isocarbofos	4.67	0.5	Positive	307	272.988	10.23	30
Isofenphos-methyl	6.73	0.5	Positive	332.107	120.899	33.96	45
Isofenphos-methyl	6.73	0.5	Positive	332.107	230.917	14.44	45
Isoprocab	4.45	0.5	Positive	194.117	94.97	14.63	30
Isoprocab	4.45	0.5	Positive	194.117	137.071	10.23	30
Isoprothiolane	5.61	0.5	Positive	291.071	188.887	20.99	33

Isoprothiolane	5.61	0.5	Positive	291.071	230.97	10.23	33
Isoproturon	4.57	0.5	Positive	207.149	71.988	18.42	40
Isoproturon	4.57	0.5	Positive	207.149	165.071	14.09	40
Isoxaflutole	4.61	0.5	Positive	360.051	219.845	38.62	50
Isoxaflutole	4.61	0.5	Positive	360.051	250.917	15.91	50
Kresoxim-methyl	6.68	0.5	Positive	314.138	222.071	10.23	30
Kresoxim-methyl	6.68	0.5	Positive	314.138	267.071	10.23	30
Lenacil	4.51	0.5	Positive	235.144	135.988	31.72	36
Lenacil	4.51	0.5	Positive	235.144	153	15.31	36
Linuron	5.31	0.5	Positive	249.019	159.976	17.58	45
Linuron	5.31	0.5	Positive	249.019	181.988	15.46	45
Lufenuron	8.38	0.5	Negative	508.971	326.042	18.65	80
Lufenuron	8.38	0.5	Negative	508.971	339.113	11.32	80
Malaoxon	3.72	0.5	Positive	315.066	126.988	11.48	36
Malaoxon	3.72	0.5	Positive	315.066	142.917	10.23	36
Malathion	7.38	0.5	Positive	331.043	124.845	28.54	30
Malathion	7.38	0.5	Positive	331.043	126.899	11.71	30
Mandipropamid	5.56	0.5	Positive	412.131	328.018	14.28	45
Mandipropamid	5.56	0.5	Positive	412.131	356.018	10.23	45
Mepanipyrim	5.95	0.5	Positive	224.118	77	37.79	50
Mepanipyrim	5.95	0.5	Positive	224.118	105.929	26	50
Meptyldinocap	9.25	0.5	Negative	295.15	134.042	55	96
Meptyldinocap	9.25	0.5	Negative	295.15	194.083	25.39	96
Metaflumizone	8.28	0.5	Positive	507.125	177.97	25.24	60
Metaflumizone	8.28	0.5	Positive	507.125	287	25.09	60
Metalaxyl	4.54	0.5	Positive	280.154	220.054	14.4	45
Metalaxyl	4.54	0.5	Positive	280.154	248.054	10.23	45
Metamitron	2.72	0.5	Positive	203.092	103.917	23.34	50
Metamitron	2.72	0.5	Positive	203.092	175	17.09	50
Metconazole	7.20	0.5	Positive	320.152	69.97	24.03	50
Metconazole	7.20	0.5	Positive	320.152	124.97	38.85	50
Methamidophos	0.54	1.5	Positive	142.008	93.917	14.51	36
Methamidophos	0.54	1.5	Positive	142.008	124.917	14.4	36
Methiocarb	5.41	0.5	Positive	226.089	106.917	37.64	30
Methiocarb	5.41	0.5	Positive	226.089	121	10.23	30
Methiocarb-sulfone	2.83	0.5	Positive	258.079	122.071	18.98	45
Methiocarb-sulfone	2.83	0.5	Positive	258.079	200.97	10.23	45
Methiocarb-sulfoxide	2.60	0.5	Positive	242.084	122	29.03	45
Methiocarb-sulfoxide	2.60	0.5	Positive	242.084	184.97	13.68	45
Methomyl	2.12	0.5	Positive	163.053	87.97	10.23	27
Methomyl	2.12	0.5	Positive	163.053	105.917	10.23	27
Methoxyfenozide	5.76	0.5	Positive	369.217	149	17.28	30
Methoxyfenozide	5.76	0.5	Positive	369.217	312.982	10.23	30
Metobromuron	4.38	0.5	Positive	259.007	148	15.5	50

Metobromuron	4.38	0.5	Positive	259.007	169.887	18.91	50
Metolachlor	6.26	0.5	Positive	284.141	176.054	25.85	40
Metolachlor	6.26	0.5	Positive	284.141	252.054	15.19	40
Metolcarb	3.41	0.5	Positive	166.086	93.97	30.78	30
Metolcarb	3.41	0.5	Positive	166.086	108.929	10.23	30
Metrafenone	7.31	0.5	Positive	409.064	208.929	13.98	50
Metrafenone	7.31	0.5	Positive	409.064	226.845	20.46	50
Monocrotophos	2.31	0.5	Positive	224.068	126.899	15.8	36
Monocrotophos	2.31	0.5	Positive	224.068	192.845	10.23	36
Monolinuron	4.11	0.5	Positive	215.058	125.917	17.89	40
Monolinuron	4.11	0.5	Positive	215.058	148	14.78	40
Monuron	3.58	0.5	Positive	199.063	71.97	16.71	45
Monuron	3.58	0.5	Positive	199.063	125.97	26.11	45
Myclobutanil	5.88	0.5	Positive	289.121	89	54.73	50
Myclobutanil	5.88	0.5	Positive	289.121	124.899	32.59	50
Neburon	6.68	0.5	Positive	275.071	57	21.22	45
Neburon	6.68	0.5	Positive	275.071	88.054	16.56	45
Nitenpyram	2.00	0.5	Positive	271.095	189	14.06	45
Nitenpyram	2.00	0.5	Positive	271.095	225	11.97	45
Novaluron	7.86	0.5	Negative	491.005	304.982	14.97	80
Novaluron	7.86	0.5	Negative	491.005	470.97	12.5	80
Omethoate	1.68	0.5	Positive	214.029	124.917	22.36	36
Omethoate	1.68	0.5	Positive	214.029	182.887	11.36	36
Oxadiargyl	7.15	0.5	Positive	341.045	222.917	16.07	60
Oxadiargyl	7.15	0.5	Positive	341.045	229.958	14.81	60
Oxadixyl	3.31	0.5	Positive	279.133	132	31.15	36
Oxadixyl	3.31	0.5	Positive	279.133	219.054	10.23	36
Oxamyl	1.98	0.5	Positive	237.025	71.97	10.23	27
Oxamyl	1.98	0.5	Positive	237.025	89.97	10.23	27
Oxasulfuron	3.43	0.5	Positive	407	107.196	43.25	50
Oxasulfuron	3.43	0.5	Positive	407	150.196	18.44	50
Oxfendazole	3.35	0.5	Positive	316.075	159.071	33.81	77
Oxfendazole	3.35	0.5	Positive	316.075	191.196	20.84	77
Paclobutrazol	5.65	0.5	Positive	294.136	69.97	20.92	50
Paclobutrazol	5.65	0.5	Positive	294.136	124.899	37.37	50
Paraoxon Methyl	3.31	0.5	Positive	248.03	109.071	19.49	100
Paraoxon Methyl	3.31	0.5	Positive	248.03	202.125	19.07	100
Penconazole	6.81	0.5	Positive	284.071	122.917	49.28	50
Penconazole	6.81	0.5	Positive	284.071	158.917	29.6	50
Pencycuron	7.38	0.5	Positive	329.141	124.917	25.81	50
Pencycuron	7.38	0.5	Positive	329.141	218	16.1	50
Penflufen	6.68	0.5	Positive	318.2	141.083	29.35	50
Penflufen	6.68	0.5	Positive	318.2	234.125	15.19	50
Penthiopyrad	6.89	0.5	Positive	360.14	177.125	34.82	45

Penthiopyrad	6.89	0.5	Positive	360.14	276.125	14.6	45
Permethrin	9.70	0.75	Positive	408.112	183.125	19.79	43
Permethrin	9.70	0.75	Positive	408.112	355.125	8.71	43
Phenthoate	6.67	0.5	Positive	321.037	79	39.61	36
Phenthoate	6.67	0.5	Positive	321.037	246.917	10.23	36
Phosalone	7.25	0.5	Positive	367.994	110.917	37.52	50
Phosalone	7.25	0.5	Positive	367.994	181.887	15	50
Phosmet	5.02	0.5	Positive	318.001	77	51.32	36
Phosmet	5.02	0.5	Positive	318.001	159.97	10.23	36
Phoxim	7.13	0.5	Positive	299.061	77.125	29.33	36
Phoxim	7.13	0.5	Positive	299.061	129.113	10.23	36
Pirimicarb	3.26	0.5	Positive	239.15	71.97	21.45	40
Pirimicarb	3.26	0.5	Positive	239.15	181.982	16.1	40
Pirimicarb-desmethyl	2.46	0.5	Positive	225.134	71.929	20.99	40
Pirimicarb-desmethyl	2.46	0.5	Positive	225.134	168.054	14.74	40
Pirimiphos-methyl	7.08	0.5	Positive	306.103	107.929	30.55	50
Pirimiphos-methyl	7.08	0.5	Positive	306.103	164.054	22.47	50
Prochloraz	7.01	0.5	Positive	376.038	69.929	25.77	40
Prochloraz	7.01	0.5	Positive	376.038	307.815	11.71	40
Profenophos	7.89	0.5	Positive	372.942	302.72	18.68	50
Profenophos	7.89	0.5	Positive	372.942	344.833	13.34	50
Promecarb	5.57	0.5	Positive	208.133	108.929	16.48	30
Promecarb	5.57	0.5	Positive	208.133	151.054	10.23	30
Prometryn	5.30	0.5	Positive	242.143	157.97	23.65	60
Prometryn	5.30	0.5	Positive	242.143	199.929	18.76	60
Propamocarb	1.70	0.5	Positive	189.159	101.917	17.58	40
Propamocarb	1.70	0.5	Positive	189.159	144	13.41	40
Propaquizafop	8.10	0.5	Positive	444.132	99.929	18.76	60
Propaquizafop	8.10	0.5	Positive	444.132	371	16.29	60
Propargite	8.70	0.5	Positive	368	175.054	16.03	36
Propargite	8.70	0.5	Positive	368	231.065	10.23	36
Propazine	5.19	0.5	Positive	230.116	145.917	23.31	45
Propazine	5.19	0.5	Positive	230.116	187.899	17.85	45
Propiconazole	7.02	0.5	Positive	342.077	69	20.12	60
Propiconazole	7.02	0.5	Positive	342.077	158.917	29.14	60
Propoxur	3.64	0.5	Positive	210.112	110.917	14.28	30
Propoxur	3.64	0.5	Positive	210.112	168.054	10.23	30
Propyzamide	5.69	0.5	Positive	256.03	172.958	22.86	33
Propyzamide	5.69	0.5	Positive	256.03	190.042	14.14	33
Proquinazid	9.05	0.5	Positive	373.04	288.851	23.46	50
Proquinazid	9.05	0.5	Positive	373.04	330.774	14.47	50
Prosulfocarb	7.74	0.5	Positive	252.141	90.97	22.55	40
Prosulfocarb	7.74	0.5	Positive	252.141	128.054	12.65	40
Pymetrozine	1.62	0.5	Positive	218.103	77.929	38.4	50

Pymetrozine	1.62	0.5	Positive	218.103	104.917	20.24	50
Pyraclostrobin	7.13	0.5	Positive	388.105	162.982	23.72	40
Pyraclostrobin	7.13	0.5	Positive	388.105	193.899	12.5	40
Pyretrins I	8.87	0.5	Positive	329	143	16.14	36
Pyretrins I	8.87	0.5	Positive	329	161.042	10.23	36
Pyretrins II	7.60	0.5	Positive	373	143	17.43	45
Pyretrins II	7.60	0.5	Positive	373	161.054	10.23	45
Pyridaben	9.20	0.5	Positive	365.144	147.054	24.41	40
Pyridaben	9.20	0.5	Positive	365.144	308.988	12.09	40
Pyridalyl	10.26	0.5	Positive	489.975	108.887	27.21	70
Pyridalyl	10.26	0.5	Positive	489.975	182.917	17.35	70
Pyridaphenthion	5.87	0.5	Positive	341.071	188.97	21.41	60
Pyridaphenthion	5.87	0.5	Positive	341.071	204.899	21.03	60
Pyridate	9.60	0.5	Positive	379.124	206.845	17.17	45
Pyridate	9.60	0.5	Positive	379.124	350.982	10.23	45
Pyriofenone	7.33	0.5	Positive	366.11	184.042	23.96	60
Pyriofenone	7.33	0.5	Positive	366.11	209.125	24.46	60
Pyrimethanil	4.84	0.5	Positive	200.118	106.917	24.18	60
Pyrimethanil	4.84	0.5	Positive	200.118	183.042	24.03	60
Pyriproxyfen	8.34	0.5	Positive	322.143	95.988	15.61	45
Pyriproxyfen	8.34	0.5	Positive	322.143	227.071	14.51	45
Quinalphos	6.61	0.5	Positive	299.061	147	21.71	60
Quinalphos	6.61	0.5	Positive	299.061	162.97	21.11	60
Quinoclamine	3.47	0.5	Positive	208.015	76.97	36.35	45
Quinoclamine	3.47	0.5	Positive	208.015	104.917	24.97	45
Quinoxiphen	8.45	0.5	Positive	308.003	161.988	45.29	60
Quinoxiphen	8.45	0.5	Positive	308.003	196.833	31.87	60
Quizalofop	6.47	0.5	Positive	345.063	244.042	29.81	79
Quizalofop	6.47	0.5	Positive	345.063	299.125	18.86	79
Quizalofop-P-ethyl	7.91	0.5	Positive	373.094	271	25.54	60
Quizalofop-P-ethyl	7.91	0.5	Positive	373.094	298.899	18.98	60
Rotenone	6.38	0.5	Positive	395.148	191.917	23.27	60
Rotenone	6.38	0.5	Positive	395.148	212.929	22.28	60
Simazine	3.63	0.5	Positive	202.09	124.125	18.56	45
Simazine	3.63	0.5	Positive	202.09	132.054	19.53	45
Spinetoram J	7.50	0.5	Positive	748.499	97.946	42.38	83
Spinetoram J	7.50	0.5	Positive	748.499	142	28.8	83
Spinetoram L	7.93	0.5	Positive	760.499	97.946	42.38	83
Spinetoram L	7.93	0.5	Positive	760.499	142	28.8	83
Spinosyn A	7.06	0.5	Positive	732.46	97.94	42.19	94
Spinosyn A	7.06	0.5	Positive	732.46	141.982	29.49	94
Spinosyn D	7.46	0.5	Positive	746.483	97.94	42.19	94
Spinosyn D	7.46	0.5	Positive	746.483	141.982	29.49	94
Spirodiclofen	8.94	0.5	Positive	411.112	71	16.22	36

Spirodiclofen	8.94	0.5	Positive	411.112	312.917	10.68	36
Spiromesifen	8.64	0.5	Positive	371.221	255.054	23.69	33
Spiromesifen	8.64	0.5	Positive	371.221	273.054	10.23	33
Spirotetramat	6.04	0.5	Positive	374.196	302.054	16.94	50
Spirotetramat	6.04	0.5	Positive	374.196	330.065	15.5	50
Spirotetramat-enol	2.49	0.5	Positive	302.175	216	28.16	70
Spirotetramat-enol	2.49	0.5	Positive	302.175	270.012	21.11	70
Spirotetramat-enolglukoside	2.49	0.5	Positive	464.227	216.054	44.93	40
Spirotetramat-enolglukoside	2.49	0.5	Positive	464.227	302.28	14.35	40
Spirotetramat-ketohydroxy	4.78	0.5	Positive	318.169	214.125	27.11	36
Spirotetramat-ketohydroxy	4.78	0.5	Positive	318.169	300.208	12.75	36
Spirotetramat-monohydroxy	3.56	0.5	Positive	304.19	211.208	18.9	45
Spirotetramat-monohydroxy	3.56	0.5	Positive	304.19	254.137	18.27	45
Spiroxamine	5.31	0.5	Positive	298.274	99.929	30.81	45
Spiroxamine	5.31	0.5	Positive	298.274	144.054	20.58	45
Sulfoxaflor	2.86	0.5	Positive	278.056	153.97	28.39	33
Sulfoxaflor	2.86	0.5	Positive	278.056	173.97	10.23	33
Tebuconazole	6.88	0.5	Positive	308.152	69.97	22.93	50
Tebuconazole	6.88	0.5	Positive	308.152	124.899	37.14	50
Tebufenozide	6.58	0.5	Positive	353.222	132.97	19.4	30
Tebufenozide	6.58	0.5	Positive	353.222	296.982	10.23	30
Tebufenpyrad	8.04	0.5	Positive	334.168	116.917	35.78	70
Tebufenpyrad	8.04	0.5	Positive	334.168	144.97	27.14	70
Teflubenzuron_neg	8.40	0.5	Negative	379	196.042	21.26	45
Teflubenzuron_neg	8.40	0.5	Negative	379	339.042	9.72	45
Terbutylazine	5.37	0.5	Positive	230.116	103.845	32.18	45
Terbutylazine	5.37	0.5	Positive	230.116	173.97	17.17	45
Terbutylazine-desethyl	3.88	0.5	Positive	202.08	104.042	28.13	40
Terbutylazine-desethyl	3.88	0.5	Positive	202.08	146.113	16.75	40
Terbutryn	5.41	0.5	Positive	242.143	90.988	40.52	45
Terbutryn	5.41	0.5	Positive	242.143	185.988	18.34	45
Tetraconazole	6.29	0.5	Positive	372.028	69.97	22.28	60
Tetraconazole	6.29	0.5	Positive	372.028	158.887	29.79	60
Thiabendazole	2.43	0.5	Positive	202.043	130.97	32.82	70
Thiabendazole	2.43	0.5	Positive	202.043	174.97	25.54	70
Thiacloprid	2.98	0.5	Positive	253.03	89.97	35.63	50
Thiacloprid	2.98	0.5	Positive	253.03	125.917	20.92	50
Thiobencarb	7.29	0.5	Positive	258.071	89	47.23	36
Thiobencarb	7.29	0.5	Positive	258.071	124.97	19.14	36
Thiophanate-methyl	3.63	0.5	Positive	343.052	150.827	19.71	50
Thiophanate-methyl	3.63	0.5	Positive	343.052	310.631	10.23	50
Tolfenpyrad	8.20	0.5	Positive	384.147	170.982	24.63	70
Tolfenpyrad	8.20	0.5	Positive	384.147	197.208	25.35	70
Triadimefon	5.77	0.5	Positive	294.1	196.899	15.35	50

Triadimefon	5.77	0.5	Positive	294.1	224.929	13.07	50
Triallate	8.56	0.5	Positive	304.009	86.113	16.29	60
Triallate	8.56	0.5	Positive	304.009	142.833	27.02	60
Triazophos	5.95	0.5	Positive	314.072	118.929	34	50
Triazophos	5.95	0.5	Positive	314.072	162	18.45	50
Trichlorfon	2.75	0.5	Positive	256.892	109.125	19.15	54
Trichlorfon	2.75	0.5	Positive	256.892	220.905	9.76	54
Triclocarban	7.81	0.5	Positive	314.985	126.857	32.75	50
Triclocarban	7.81	0.5	Positive	314.985	161.887	18.83	50
Tricyclazole	3.11	0.5	Positive	190.043	135.917	28.5	50
Tricyclazole	3.11	0.5	Positive	190.043	162.97	22.59	50
Trifloxystrobin	7.60	0.5	Positive	409.136	144.917	43.06	50
Trifloxystrobin	7.60	0.5	Positive	409.136	185.917	17.39	50
Triflumizole	7.70	0.5	Positive	346.092	73	16.37	33
Triflumizole	7.70	0.5	Positive	346.092	277.97	10.23	33
Triflumuron	7.28	0.5	Positive	359.04	138.917	30.47	45
Triflumuron	7.28	0.5	Positive	359.04	155.988	15.46	45
Triticonazole	6.17	0.5	Positive	318.136	69.917	18.3	45
Triticonazole	6.17	0.5	Positive	318.136	124.97	32.9	45
Tritosulfuron	5.09	0.5	Positive	446.035	194.917	18.64	50
Tritosulfuron	5.09	0.5	Positive	446.035	220.929	18.72	50
XMC	4.15	1.5	Positive	180.101	94.929	20.01	30
XMC	4.15	1.5	Positive	180.101	122.929	12.16	30
Zoxamide	7.02	0.5	Positive	336.031	158.917	39	50
Zoxamide	7.02	0.5	Positive	336.031	186.815	22.17	50

Table A2. Accuracy data (as recovery values (%)) and precision data (as repeatability RSD_r, n = 5) at 0.010 mg/kg for olive samples with different extraction methods.

No.	Compound	Manual method with clean-up		Manual method with freezing-out		Automatic extraction Method 02	
		Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
1	2,4-D	87	17	99	12	104	12
2	Acephate	80	5	63	15	89	14
3	Acetamiprid	86	4	88	7	107	9
4	Alachlor	80	3	96	5	108	2
5	Albendazole	84	3	93	4	102	2
6	Aldicarb	87	5	90	8	100	17
7	Aldicarb Sulfone	86	2	90	8	110	4
8	Aldicarb Sulfoxide	84	8	77	7	87	2
9	Ametoctradin	86	4	93	4	106	2
10	Anilofos	87	4	100	4	105	4
11	Atrazine	84	4	95	2	103	4
12	Azinphos-Ethyl	84	14	96	3	103	2
13	Azinphos-Methyl	91	7	93	5	110	4
14	Azoxystrobin	90	5	94	5	112	4
15	BAC 10	74	7	121	7	120	2
16	BAC 8	78	4	95	7	108	2
17	Benalaxyl	88	4	94	5	110	2
18	Bendiocarb	90	11	94	9	106	16
19	Bifenazate	71	5	66	10	75	12
20	Bifenthrin	102	7	126	9	100	15
21	Bitertanol	75	14	98	19	111	15
22	Boscalid	94	4	92	4	113	3
23	Bromacil	87	5	94	4	96	2
24	Bromuconazole	86	2	95	4	102	3
25	BTS-44595	88	3	96	7	104	10
26	BTS-44596	92	4	96	3	114	4
27	BTS-40348	26	6	82	9	74	13
28	Bupirimate	87	5	96	5	111	2
29	Buprofezin	81	3	97	6	92	3
30	Butoxycarboxim	88	8	80	12	113	14
31	Carbaryl	88	5	96	5	102	3
32	Carbendazim	82	2	85	8	95	17
33	Carbofuran	87	6	95	4	104	3
34	Chlorantraniliprole	86	2	96	7	114	8
35	Chlorbromuron	90	3	96	10	104	12
36	Chlorfenvinphos	85	3	96	3	110	2
37	Chlorfluazuron	147	11	184	13	139	2
38	Chloridazon	84	5	87	7	95	2
39	Chlorotoluron	89	5	94	5	102	2

40	Chloroxuron	89	2	95	6	110	3
41	Chlorpyrifos	100	11	121	5	116	4
42	Chromafenozide	89	3	97	6	108	4
43	Clofentezine	90	8	98	5	101	3
44	Clomazone	88	3	93	4	107	4
45	Clothianidin	87	4	91	6	106	4
46	Coumaphos	91	6	101	5	104	4
47	Cyazofamid	90	3	92	7	111	4
48	Cyflufenamid	97	7	100	6	110	4
49	Cyprodinil	86	2	95	4	89	2
50	Cyromazine	100	6	56	15	81	12
51	DEET	91	3	94	3	107	2
52	Demeton-S-Methyl	80	5	94	5	101	4
53	Demeton-S-Methylsulfone	86	4	93	8	112	13
54	Demeton-S-Methylsulfoxide	87	2	83	7	97	6
55	Diazinon	85	4	91	6	100	3
56	Dichlorvos	94	9	97	10	106	4
57	Dicrotophos	93	4	85	3	108	4
58	Diethofencarb	90	6	94	4	110	3
59	Difenoconazole	91	9	101	5	112	3
60	Difenoxyuron	87	3	93	5	113	3
61	Diflubenzuron	94	7	98	5	114	3
62	Dimethoate	86	4	88	8	107	9
63	Dimethomorph	90	4	92	5	117	2
64	Dimethylvinphos	92	10	96	11	112	5
65	Diniconazole	83	6	91	5	111	3
66	Diuron	91	3	95	4	106	2
67	DMF	89	5	96	3	108	2
68	Dodine	77	9	123	8	100	16
69	Edifenphos	91	5	94	6	110	2
70	Eamectin B1a	81	7	102	4	114	2
71	Epoxiconazole	85	4	92	4	109	4
72	Ethiofencarb	83	2	91	4	105	3
73	Ethion	103	9	129	7	114	4
74	Ethiprole	104	12	91	7	104	4
75	Ethirimol	82	4	85	5	85	2
76	Ethoprofos	89	4	96	4	107	4
77	Etofenprox	116	5	158	6	104	4
78	Etoxazol	97	8	130	4	109	3
79	Famoxadone	93	5	103	9	114	12
80	Fenamidone	90	6	99	3	109	3
81	Fenamiphos	88	5	95	6	115	3
82	Fenamiphos-Sulfone	91	3	90	4	106	4
83	Fenamiphos-Sulfoxide	88	3	92	3	102	2

84	Fenarimol	91	8	104	6	100	3
85	Fenazaquin	85	7	122	5	98	3
86	Fenbuconazole	84	3	101	7	114	4
87	Fenhexamide	86	3	95	5	92	2
88	Fenobucarb	86	4	93	3	112	3
89	Fenoxycarb	87	5	97	5	107	4
90	Fenpropidin	54	13	97	6	102	4
91	Fenpropimorph	54	4	87	4	84	3
92	Fenpyrazamine	90	7	93	4	108	3
93	Fenpyroximate	101	7	133	4	110	2
94	Fenthion	82	21	104	15	92	14
95	Fenthion-Oxon-Sulfone	80	3	92	5	97	4
96	Fenthion-Sulfone	91	5	95	5	120	4
97	Fenthion-Sulfoxide	87	2	93	5	107	2
98	Fipronil	92	2	98	4	115	2
99	Fipronil-Sulfone	93	6	100	4	117	4
100	Flazasulfuron	83	4	95	5	113	2
101	Flonicamid	83	8	97	8	111	7
102	Fluacypirim	89	4	95	5	115	2
103	Fluazifop	79	10	95	4	120	3
104	Flubendiamide	99	10	106	14	117	13
105	Fludioxonil	84	3	87	2	115	4
106	Flufenacet	87	4	94	4	107	2
107	Flufenoxuron	125	5	153	4	129	3
108	Fluometuron	87	3	94	5	105	4
109	Fluopicolide	89	4	94	5	108	4
110	Fluopyram	93	4	95	5	111	3
111	Fluquinconazole	91	2	91	7	117	3
112	Flusilazole	92	5	93	4	109	2
113	Flutriafol	90	5	92	8	118	7
114	Fluxapyroxad	90	2	95	5	113	4
115	Formetanate-Hydrochloride	81	3	72	4	88	2
116	Fosthiazate	87	4	95	5	112	4
117	Haloxypop	89	14	93	11	102	4
118	Hexaconazole	86	6	102	4	108	3
119	Hexaflumuron	110	21	114	12	86	8
120	Hexythiazox	93	6	114	7	103	4
121	Imazalil	46	3	71	8	72	5
122	Imidacloprid	88	2	92	7	112	4
123	Indoxacarb	94	6	106	4	116	4
124	Ioxynil	88	3	93	3	116	3
125	Iprovalicarb	89	4	93	5	114	4
126	Isocarbofos	88	10	99	15	119	3
127	Isfenphos-Methyl	85	6	96	4	103	3

128	Isoprocab	84	4	94	7	107	4
129	Isoprothiolane	89	3	94	4	107	3
130	Isoproturon	88	3	95	5	105	3
131	Isoxaflutole	95	6	94	5	118	2
132	Kresoxim-Methyl	89	3	94	4	117	4
133	Lenacil	86	4	93	5	100	3
134	Linuron	91	4	93	5	107	3
135	Lufenuron	117	17	179	16	112	13
136	Malaoxon	74	4	78	5	106	3
137	Malathion	83	5	91	5	101	4
138	Mandipropamid	92	3	96	3	120	4
139	Mepanipyrim	86	2	101	6	92	2
140	Meptyldinocap	135	26	125	9	118	5
141	Metaflumizone	134	7	157	7	126	2
142	Metalaxyl	86	5	93	6	108	2
143	Metamitron	94	10	92	13	97	10
144	Metconazole	86	6	92	4	108	3
145	Methidathion	88	3	98	6	102	3
146	Methiocarb	94	7	97	10	99	7
147	Methiocarb-Sulfone	82	7	87	12	112	3
148	Methiocarb-Sulfoxide	87	5	86	9	104	14
149	Methomyl	87	5	90	5	114	4
150	Methoxyfenozide	89	5	96	5	115	3
151	Metobromuron	84	7	92	7	104	4
152	Metolachlor	88	2	93	4	111	3
153	Metolcarb	84	5	92	7	104	4
154	Metrafenone	89	7	96	4	113	2
155	Monocrotophos	88	2	83	5	101	2
156	Monolinuron	87	4	95	6	99	2
157	Monuron	85	6	93	5	98	2
158	Myclobutanil	95	6	96	5	111	3
159	Neburon	90	10	104	3	102	4
160	Nitenpyram	84	7	70	8	98	15
161	Novaluron	97	10	118	8	117	12
162	Omethoate	87	3	71	6	90	3
163	Oxadiargyl	89	11	90	12	116	10
164	Oxadixyl	87	5	94	8	111	17
165	Oxamyl	89	6	92	5	110	2
166	Oxasulfuron	81	5	96	5	110	4
167	Oxfendazole	85	3	91	7	103	8
168	Paclobutrazol	85	5	95	3	119	3
169	Paraoxon Methyl	84	14	86	6	109	2
170	Penconazole	93	6	96	3	115	4
171	Pencycuron	91	6	100	5	108	3

172	Penflufen	89	4	96	6	112	2
173	Penthiopyrad	90	5	96	6	114	4
174	Permethrin	116	9	141	4	100	2
175	Phenthoate	90	5	99	6	108	3
176	Phosalone	90	8	102	4	112	2
177	Phosmet	89	5	98	6	103	4
178	Phoxim	90	7	103	4	108	4
179	Pirimicarb	86	4	91	4	106	2
180	Pirimicarb-Desmethyl	79	5	83	6	104	4
181	Pirimiphos-Methyl	85	4	97	5	103	2
182	Prochloraz	82	4	88	5	102	2
183	Profenophos	87	5	99	6	99	3
184	Promecarb	87	4	94	4	109	2
185	Prometryn	86	3	94	4	100	4
186	Propamocarb	71	4	63	8	74	2
187	Propaquizafop	94	7	104	4	111	2
188	Propargite	104	9	133	6	118	2
189	Propazine	92	9	98	8	87	16
190	Propiconazole	87	6	91	4	52	2
191	Propoxur	85	7	95	5	102	2
192	Propyzamide	88	5	95	1	106	3
193	Proquinazid	81	6	105	5	86	4
194	Prosulfocarb	86	7	94	5	96	2
195	Pymetrozine	72	16	47	5	60	4
196	Pyraclostrobin	93	3	99	6	106	2
197	Pyretrins I	102	6	131	3	115	2
198	Pyretrins II	72	16	81	14	115	5
199	Pyridaben	104	6	146	6	111	4
200	Pyridalyl	84	5	105	6	76	2
201	Pyridaphenthion	92	3	95	3	113	2
202	Pyridate	102	5	153	4	109	2
203	Pyriproxyfen	88	5	93	4	102	4
204	Pyrimethanil	88	5	97	5	94	3
205	Pyriproxyfen	94	7	122	5	107	4
206	Quinalphos	92	5	93	4	102	2
207	Quinoclamine	79	7	94	10	111	10
208	Quinoxiphen	85	5	109	6	95	4
209	Quizalofop	84	10	82	6	120	3
210	Quizalofop-P-Ethyl	93	7	100	5	108	3
211	Rotenone	87	5	93	5	110	2
212	Simazine	89	7	90	9	93	11
213	Spinetoram J	60	11	100	4	108	2
214	Spinetoram L	57	14	102	6	117	4
215	Spinosyn A	66	8	95	3	113	2

216	Spinosyn D	61	12	105	7	106	3
217	Spirodiclofen	108	8	146	5	112	3
218	Spiromesifen	99	12	117	6	101	3
219	Spirotetramat	84	5	95	5	107	4
220	Spirotetramat-Enol	82	5	66	10	81	3
221	Spirotetramat-Enolglukoside	84	3	65	15	76	14
222	Spirotetramat-Ketohydroxy	92	4	90	8	122	8
223	Spirotetramat-Monohydroxy	86	2	87	5	110	4
224	Spiroxamine	42	19	93	5	96	3
225	Sulfoxaflor	89	7	88	11	99	6
226	Tebuconazole	85	4	97	9	114	3
227	Tebufenozide	86	4	94	7	106	4
228	Tebufenpyrad	87	7	96	4	102	4
229	Teflubenzuron	92	6	104	8	84	4
230	Terbuthylazine	86	2	94	4	101	2
231	Terbuthylazine Desethyl	86	1	96	7	99	3
232	Terbutryn	86	3	92	4	102	4
233	Tetraconazole	88	5	96	4	115	3
234	Thiabendazole	82	6	88	8	103	2
235	Thiacloprid	85	5	92	7	103	2
236	Thiobencarb	84	7	98	6	97	3
237	Thiophanate-Methyl	57	4	67	6	69	3
238	Tolfenpyrad	93	6	114	3	114	4
239	Triadimefon	87	4	88	9	114	4
240	Triallate	76	18	104	13	84	5
241	Triazophos	90	3	96	4	113	2
242	Trichlorfon	84	6	73	5	103	2
243	Triclocarban	110	11	128	4	106	4
244	Tricyclazole	86	4	87	5	95	2
245	Trifloxystrobin	93	7	99	4	114	4
246	Triflumizole	83	6	101	5	105	4
247	Triflumuron	86	5	103	5	110	4
248	Triticonazole	86	5	89	7	103	2
249	Tritosulfuron	88	9	85	6	118	3
250	XMC	90	6	92	4	107	4
251	Zoxamide	92	3	97	4	106	3

Table A3. Matrix effects for olive samples with different extraction methods.

No.	Compound	Manual methods		Automatic extraction M02
		Clean-up	Freezing-out	
1	2,4-D	82	107	80
2	Acephate	-56	-16	-32
3	Acetamiprid	-27	-29	-27
4	Alachlor	-7	-8	-5
5	Albendazole	-23	-29	-22
6	Aldicarb	-44	-49	-39
7	Aldicarb Sulfone	-30	-36	-43
8	Aldicarb Sulfoxide	-18	-19	-23
9	Ametoctradin	-9	-14	-7
10	Anilofos	-8	-11	-7
11	Atrazine	-26	-30	-24
12	Azinphos-Ethyl	-6	-8	0
13	Azinphos-Methyl	-7	-5	2
14	Azoxystrobin	-8	-7	-6
15	BAC 10	-18	-33	-16
16	BAC 8	-11	-14	-11
17	Benalaxyl	-6	-7	-4
18	Bendiocarb	-29	-34	-28
19	Bifenazate	-13	-35	-20
20	Bifenthrin	-69	-24	1
21	Bitertanol	-2	-7	-4
22	Boscalid	-4	0	-1
23	Bromacil	-43	-49	-41
24	Bromuconazole	-7	-8	-6
25	BTS-44595	-5	-6	-4
26	BTS-44596	-5	-4	-3
27	BTS-40348	-23	-29	-23
28	Bupirimate	-8	-10	-9
29	Buprofezin	-7	-8	-8
30	Butoxycarboxim	-49	-56	-62
31	Carbaryl	-28	-31	-25
32	Carbendazim	-50	-56	-58
33	Carbofuran	-29	-34	-27
34	Chlorantraniliprole	1	3	5
35	Chlorbromuron	-13	-11	-9
36	Chlorfenvinphos	-7	-7	-6
37	Chlorfluazuron	-59	-33	-18
38	Chloridazon	-38	-41	-40
39	Chlorotoluron	-20	-23	-19
40	Chloroxuron	-9	-8	-7
41	Chlorpyrifos	-29	-43	-22
42	Chromafenozide	-5	-6	-4
43	Clofentezine	-5	-12	0
44	Clomazone	-14	-14	-9
45	Clothianidin	-20	-22	-23
46	Coumaphos	-6	-13	-6

47	Cyazofamid	-7	-9	-5
48	Cyflufenamid	-8	-16	-8
49	Cyprodinil	-11	-19	-12
50	Cyromazine	-11	-14	-15
51	DEET	-13	-16	-12
52	Demeton-S-Methyl	-25	-30	-23
53	Demeton-S-Methylsulfone	-30	-34	-37
54	Demeton-S-Methylsulfoxide	-27	-32	-35
55	Diazinon	-7	-8	-5
56	Dichlorvos	-18	-20	-16
57	Diclotophos	-23	-21	-24
58	Diethofencarb	-3	-3	0
59	Difenoconazole	-10	-19	-9
60	Difloxuron	-12	-11	-10
61	Diflubenzuron	-6	-8	-3
62	Dimethoate	-29	-31	-31
63	Dimethomorph	9	13	8
64	Dimethylvinphos	-6	-6	3
65	Diniconazole	-6	-10	-6
66	Diuron	-17	-19	-13
67	DMF	-47	-54	-48
68	Dodine	-55	-60	-34
69	Edifenphos	-5	-8	-5
70	Enamectin B1 a	-11	-14	-10
71	Epoxiconazole	-5	-5	-4
72	Ethiofencarb	-22	-27	-20
73	Ethion	-30	-40	-22
74	Ethiprole	43	46	37
75	Ethirimol	-58	-60	-55
76	Ethoprofos	-8	-7	-5
77	Etofenprox	-62	-70	-24
78	Etoxadol	-34	-50	-23
79	Famoxadone	-12	-25	-9
80	Fenamidone	6	6	5
81	Fenamiphos	-5	-3	-3
82	Fenamiphos-Sulfone	-1	-5	2
83	Fenamiphos-Sulfoxide	-9	-14	-9
84	Fenarimol	-1	0	1
85	Fenazaquin	-36	-46	-24
86	Fenbuconazole	-6	-7	-4
87	Fenhexamide	-3	-4	-2
88	Fenobucarb	-12	-13	-10
89	Fenoxycarb	-7	-9	-5
90	Fenpropidin	-9	-10	-9
91	Fenpropimorph	-10	-13	-8
92	Fenpyrazamine	-7	-7	-5
93	Fenpyroximate	-35	-43	-22
94	Fenthion	-10	-15	-7
95	Fenthion-Oxon-Sulfone	-8	-9	-8

96	Fenthion-Sulfone	3	0	9
97	Fenthion-Sulfoxide	-11	-13	-9
98	Fipronil	-2	-1	-2
99	Fipronil-Sulfone	-9	-18	-8
100	Flazasulfuron	25	27	27
101	Flonicamid	-41	-47	-50
102	Fluacypirim	-8	-11	-6
103	Fluazifop	10	16	10
104	Flubendiamide	-3	-7	-3
105	Fludioxonil	0	-1	2
106	Flufenacet	-7	-7	-4
107	Flufenoxuron	-57	-39	-25
108	Fluometuron	-23	-28	-21
109	Fluopicolide	-7	-6	-4
110	Fluopyram	-7	-6	-7
111	Fluquinconazole	-10	-9	-10
112	Flusilazole	-9	-13	-8
113	Flutriafol	9	10	10
114	Fluxapyroxad	-4	-3	-3
115	Formetanate-Hydrochloride	-20	-8	-15
116	Fosthiazate	-17	-19	-15
117	Haloxifop	0	3	-4
118	Hexaconazole	-5	-10	-6
119	Hexaflumuron	-20	-35	-15
120	Hexythiazox	-27	-39	-20
121	Imazalil	-17	-20	-16
122	Imidacloprid	7	4	3
123	Indoxacarb	-11	-22	-11
124	Ioxynil	-2	-2	-1
125	Iprovalicarb	-6	-5	-2
126	Isocarbofos	-14	-18	-9
127	Isofenphos-Methyl	-8	-11	-5
128	Isoprocarb	-15	-18	-13
129	Isoprothiolane	-9	-8	-7
130	Isoproturon	-15	-17	-13
131	Isoxaflutole	48	58	52
132	Kresoxim-Methyl	-6	-8	-5
133	Lenacil	-22	-24	-21
134	Linuron	-12	-13	-9
135	Lufenuron	-47	-34	-19
136	Malaoxon	-18	-22	-17
137	Malathion	-9	-3	-6
138	Mandipropamid	-4	-3	-2
139	Mepanipyrim	-12	-15	-9
140	Meptyldinocap	-51	-64	-27
141	Metaflumizone	-60	-57	-17
142	Metaxyl	-12	-13	-11
143	Metamitron	-30	-26	-39
144	Metconazole	-4	-9	-5

145	Methidathion	-14	-15	-12
146	Methiocarb	-10	-8	-8
147	Methiocarb-Sulfone	-26	-28	-27
148	Methiocarb-Sulfoxide	-32	-33	-35
149	Methomyl	-30	-36	-38
150	Methoxyfenozide	-5	-5	-3
151	Metobromuron	-18	-21	-15
152	Metolachlor	-6	-5	-4
153	Metolcarb	-29	-33	-29
154	Metrafenone	-8	-13	-7
155	Monocrotophos	-30	-28	-31
156	Monolinuron	-21	-26	-20
157	Monuron	-35	-42	-34
158	Myclobutanil	-1	-1	0
159	Neburon	-8	-10	-6
160	Nitenpyram	-14	-21	-25
161	Novaluron	-35	-38	-27
162	Omethoate	-34	-18	-27
163	Oxadiargyl	-5	-9	-4
164	Oxadixyl	-27	-32	-28
165	Oxamyl	-15	-17	-21
166	Oxasulfuron	-4	-10	-5
167	Oxfendazole	12	8	14
168	Paclobutrazol	-5	-4	-6
169	Paraoxon Methyl	-42	-50	-43
170	Penconazole	-6	-9	-5
171	Pencycuron	-12	-22	-10
172	Penflufen	-6	-5	-4
173	Penthiopyrad	-7	-8	-5
174	Permethrin	-66	-26	-21
175	Phenthoate	-8	-12	-8
176	Phosalone	-10	-20	-9
177	Phosmet	-5	-5	-2
178	Phoxim	-11	-19	-9
179	Pirimicarb	-26	-30	-27
180	Pirimicarb-Desmethyl	-29	-29	-32
181	Pirimiphos-Methyl	-6	-11	-5
182	Prochloraz	-9	-18	-10
183	Profenophos	-11	-18	-9
184	Promecarb	-11	-11	-8
185	Prometryn	-11	-10	-9
186	Propamocarb	-26	-13	-20
187	Propaquizafop	-11	-20	-9
188	Propargite	-34	-43	-24
189	Propazine	-16	-21	-25
190	Propiconazole	-6	-8	-5
191	Propoxur	-29	-36	-27
192	Propyzamide	-9	-8	-7
193	Proquinazid	-23	-36	-17

194	Prosulfocarb	-11	-20	-10
195	Pymetrozine	-26	3	-13
196	Pyraclostrobin	-9	-15	-8
197	Pyretrins I	-31	-42	-21
198	Pyretrins II	-26	-31	-29
199	Pyridaben	-50	-55	-27
200	Pyridalyl	-66	-53	-4
201	Pyridaphenthion	-2	-1	-1
202	Pyridate	-62	-58	-19
203	Pyriproxyfen	-5	-8	-6
204	Pyrimethanil	-13	-14	-10
205	Pyriproxyfen	-28	-41	-22
206	Quinalphos	-6	-8	-4
207	Quinoclamine	-53	-59	-56
208	Quinoxiphen	-26	-44	-22
209	Quizalofop	-1	2	0
210	Quizalofop-P-Ethyl	-11	-21	-10
211	Rotenone	-3	-4	-1
212	Simazine	-39	-45	-37
213	Spinetoram J	-7	-11	-10
214	Spinetoram L	-12	-21	-14
215	Spinosyn A	-5	-4	-6
216	Spinosyn D	-9	-11	-10
217	Spirodiclofen	-39	-47	-23
218	Spiromesifen	-34	-38	-21
219	Spirotetramat	8	10	12
220	Spirotetramat-Enol	-26	-25	-29
221	Spirotetramat-Enolglukoside	-25	-25	-26
222	Spirotetramat-Ketohydroxy	74	91	60
223	Spirotetramat-Monohydroxy	50	47	55
224	Spiroxamine	-7	-11	-7
225	Sulfoxaflor	-48	-52	-48
226	Tebuconazole	-5	-7	-2
227	Tebufenozide	-5	-7	-4
228	Tebufenpyrad	-13	-22	-10
229	Teflubenzuron	-3	-22	5
230	Terbuthylazine	-19	-20	-16
231	Terbuthylazine Desethyl	-32	-36	-30
232	Terbutryn	-9	-9	-7
233	Tetraconazole	-7	-7	-6
234	Thiabendazole	-45	-48	-51
235	Thiacloprid	-51	-56	-49
236	Thiobencarb	-9	-14	-6
237	Thiophanate-Methyl	-16	-25	-12
238	Tolfenpyrad	-21	-37	-16
239	Triadimefon	2	3	3
240	Triallate	-21	-37	-17
241	Triazophos	-7	-6	-6

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242	Trichlorfon	-11	-13	-12
243	Triclocarban	-41	-57	-26
244	Tricyclazole	-55	-62	-51
245	Trifloxystrobin	-11	-18	-9
246	Triflumizole	-13	-29	-13
247	Triflumuron	-13	-21	-10
248	Triticonazole	-1	-1	2
249	Tritosulfuron	24	31	24
250	XMC	-22	-26	-20
251	Zoxamide	-7	-10	-6