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Evaluating the Presence of Pesticide Residues in Organic Rice Production in An Giang Province, Vietnam

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Abstract

The presence of pesticide residues was investigated in the organic rice production model in An Giang province, Vietnam. A total number of sixteen pesticide residues was been recorded during the investigation. Based on their contamination rate, they are classified as follows. The high-risk group includes tricyclazole (80%). The medium-risk group includes chlorpyrifos (47%), isoprothiolane (47%), difenoconazole (40%), propiconazole (40%), hexaconazole (40%), chlorfenapyr (33%), azoxystrobin (20%), and cypermethrin (20%). The low-risk group includes metalaxyl & metalaxyl-M, paclobutazol, niclosamide, chlorfenson, fipronil, fipronil-desulfinyl, and fenoxanil, which were detected with a contamination rate of 7%. There were seven insecticides, seven fungicides, one snail killer, and one growth regulator.

Keywords: pesticide residues, tricyclazole, prohibited pesticides, organic rice farming, organic production, An Giang province

1. Introduction

Pesticides in controlling rice diseases have increased in recent years due to the higher incidence of insects and pests. However, pesticide residues in food are a major public health concern and harm producers and consumers (Hou et al., 2013). Identifying the presence of such residues in all types of food (both fresh and industrialized) is important to guarantee food safety (Wang et al., 2012). At the same time, the use of pesticides on rice fields can affect the quality of environmental resources such as groundwater and surface water.

Under the European Union (EU) legislation (Article 32, Regulation (EC) No 396/2005), the European Food Safety Authority (EFSA) (2020) provides an annual report which analyses pesticide residue levels in foods in the EU market. According to the report, for 2018, 95.5% of the overall 91,015 analyzed samples fell below the maximum residue level (MRL), 4.5% exceeded MRL, of which 2.7% was non-compliant, i.e. samples exceeding the MRL after taking into account the measurement uncertainty. For the subset of 11,679 analyzed samples as part of the EU-coordinated control program, 1.4% exceeded the MRL, and 0.9% was non-compliant. Dietary exposure to pesticide residues was estimated and compared with health-based guidance values to assess acute and chronic risk to consumer health. The findings suggest that the assessed levels for the analyzed food commodities are unlikely to pose a concern for consumers' health. However, many recommendations are proposed to increase the efficiency of European control systems (e.g. optimizing traceability), thereby ensuring a high level of consumer protection (EFSA, 2020).

Since then, organic agriculture has helped farmers apply new farming techniques, replacing harmful chemical inputs with biological and organic derived substances. At the same time, it helps to improve the skills of producers to apply the smart methods "high-tech" like using good variety, smart agronomy techniques, machines, biology and organic products to replace traditional methods in non-organic agricultural production. The biggest challenge in organic agriculture is changing farmers' mindsets and farming practices that favor the application of toxic chemicals (Nguyen and Van, 2021).

According to the Farming Reader (2021) (www.farmingreader.com), organic farming is a farming method using crop, animal, aquatic wastes, and other biological materials. This method is completely safe for the soil and the environment as it does not use any toxic chemicals. The soil remains alive and in good condition. Martin (2009) reported that organic farming is a method, which crop and livestock production involves choosing not to use pesticides, fertilizers, genetically modified organisms, antibiotics, and growth hormones. Organic production is a holistic system designed to optimize the productivity and fitness of diverse communities within the agro-ecosystem, including soil organisms, plants, livestock, and people (Martin, 2009). Supporting the organic production program, the United States Department of Agriculture (USDA) has listed the substances, including pesticides that allow and prohibit organic production (USDA, 2021). EU provided the information on permitted substances in organic production (European Commission, 2021). Japan has released the guideline for organic production according to its standards called organic JAS (MAFF, 2021). According to these standards and regulations, synthetic pesticides are prohibited for crop production unless specifically allowed, and non-synthetic pesticides are allowed for crop production unless specifically prohibited.

Since 2015, researchers from the Asian Organic Agriculture Research and Development Institute (AOI) and the Institute of Agricultural Sciences for Southern Vietnam (IAS) have contributed to successfully building the internationally certified organic rice production models in rice-shrimp farming areas of the Mekong River delta. However, building a rice production model to convert from non-organic to fully organic in rice-intensive farming fields (2-3 crops per year) is challenging (Nguyen and Van, 2021). Organic production in intensive farming areas is not as favorable as in rice-shrimp farming areas (i.e. only one rice crop is rotated with one shrimp crop) because there is no reciprocity to help limit the pest and disease pressure (Nguyen et al., 2019). So it is tough to be certified according to EU, USDA, and JAS organic standards. Another challenge is changing farmers' farming practices that favor the application of pesticides. In some cases, the farmers did not follow organic production procedures to use the prohibited pesticides for their farms. This had led to failing the organic rice models, affecting the entire project and causing financial damage to the invested enterprises (Nguyen and Van, 2021). So, assessing the presence of prohibited residual pesticides in organic production is necessary to build the organic rice production model in rice-intensive farming areas.

The study evaluates the presence of residual pesticides, which are prohibited from being used in organic production, as part of the project "building an organic rice model according to organic standards (EU, USDA, JAS) in An Giang province, Vietnam". The study's aims were (i) to investigate the presence of nearly a thousand residual pesticides, which are prohibited from being used in organic production, in soil, water, and rice crop (ii) to document the common pesticides used in rice-intensive farming systems.

2. Material and Methods

2.1 Study Sites

An Giang is a border province in the Mekong River delta with a natural area of 3.536,83 km^2 (GSO, 2021) and a rice cultivation area of 637,200 ha (GSO, 2020). This is the province with the country's largest rice production and aquaculture. Since the 1990s, the province has actively built an irrigation system to exploit the potential of the available local land. Besides the achieved achievements, the province faces the risk of soil pollution, such as the heavy use of chemical fertilizers and pesticides in agricultural production, waste from probiotics and feed in aquaculture, and wastes from production facilities and residential areas (DoENR An Giang, 2020). Minimizing the risk of pesticide pollution is an urgent requirement to protect agriculture, which is the advantage of this province.

In this study, study sites were located in Tri Ton and Thoai Son districts of An Giang province (Fig. 1). These districts are the province's two largest cultivated paddy areas, with 115,065 ha and 114,629 ha (NIAPP, 2020). Similarly to other areas of the province, three crops are permanently cultivated per year. It could be said that rice production in this area represents the rice-intensive farming system in An Giang province.

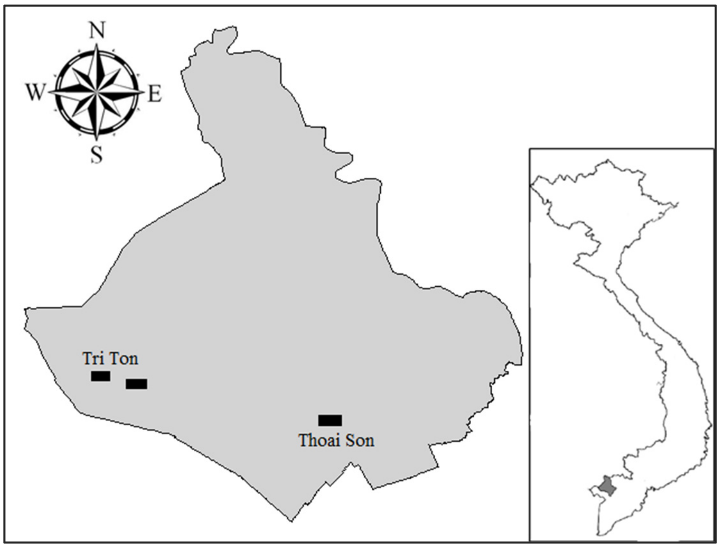

Figure 1. Location of the study sites

2.2 Studied Pesticides and Rice Cultivation

The study pesticides were selected according to the audit program of the organic certification body, ControlUnion (CU). The total number of investigated pesticides was 854 substrates (Appendix 1). Rice varieties grown in these studied areas were Hong Ngoc Oc Eo and ST 25, having a growing duration of 95 days and 114 days, respectively. Both varieties belong to the *Oryza sativa* species. In the organic production model, farmers were trained in organic rice cultivation that as described by Hay et al. (2018). Paddy was sowed and then constantly flooded with 5–7 cm depth from soil surface still 1 month before harvesting.

2.3 Sampling

Irrigation water, soil, and plants were sampled at the drought stage (around 20 days before harvest). Water samples were collected in 1 L plastic bottles, soil samples were collected at a depth of 0−10 cm, and plant samples were collected by chopping the above ground portion of the plants. The collection of soil, water, and rice samples should accurately represent the entire production batch and target to detect residual chemicals thoroughly at high-risk areas (different from random sampling). The number of samples depends on the farming conditions of each household group and risk capacity. If farming conditions of the household is not good with high-risk possibility, samples need to be taken more. If the number of households is too large, take at least 10% (Singh and Masuku, 2014). Each sample was collected from neighboring households and then mixed as 1 sample for analysis. A total number of 15 samples was sampled and analyzed during three years (2019 - 2021).

2.4 Pesticide Analysis

The samples were sent for analysis at the labs designated by CU. Without washing, the samples were then extracted and analyzed using GC-MS/MS, which was described by Braun et al. (2018) and LC-MS/MS described by Shah et al. (2015). The results from the analyses were reported in parts per million (ppm) or milligram per kilogram (mg kg^{-1}).

2.5 Evaluating Criteria and Statistical Analysis

In this study, we use the criteria to evaluate the presence of pesticides as follows. Limit of detection (LOD) is the lowest quantity of an active ingredient that can be distinguished from the absence of active ingredients. Pesticide concentration (PC) (mg kg^{-1}) is an amount of pesticide's active ingredient per total weight of the sample. Progressive presence (PP) is the cumulative number of impressions from previous times. Frequency of occurrence (FOO) is the number of times that pesticide detected with a concentration higher than LOD. Contamination rate (CR) (%) is determined by the percentage of frequency of occurrence per the total number of samples. Microsoft Excel was used for data analysis and graphing.

3. Results and Discussion

3.1 Pesticide Residues Were Present in Soil and Rice Samples in Organic Rice Fields of An Giang Province

A total number of 15 samples, including 13 rice samples, 1 soil sample, and 1 water sample, was analyzed during three years of project implementation. The results showed that 16 pesticide types were found in the samples, including tricyclazole, chlorpyrifos, isoprothiolane, difenoconazole, propiconazole, hexaconazole, chlorfenapyr, azoxystrobin, and cypermethrin, metalaxyl & metalaxyl-M, paclobutazol, niclosamide, chlorfenson, fipronil, fipronil-desulfinyl, and fenoxanil with concentrations from 0.005 mg kg^{-1} to 0.71 mg kg^{-1} (Table 1). Among the analyzed samples, sample No. 7 was the most contaminated, with 11 detected substances, while samples No. 2 and No. 15 were free of pesticide residues, and sample No. 9 only detected chlorpyrifos. This result indicates that different samples from different households found various contaminants in the same organic production model (i.e. leakage from neighboring fields were being controlled). We can infer that the contaminated pesticides came from two sources. The first, the pesticides came from a passive way, in which pesticides have been used in previous crops and are still remained up to the time of sampling. This correlates with several studies, which reported that pesticide persistence in the environment was determined by a measure known as the half-life or time for starting material to be reduced by 50%, where the half-life of pesticides can range from several hours up to 4-5 years (Hanson, 2015). Therefore, pesticides with high half-lives in soil (above 60 days), such as tricyclazole (305 days) (Thai et al., 2009), propiconazole (315 days) (Garrison et al., 2009, 2011), hexaconazole (69.3 and 86.6 days) (Maznah et al., 2015), and isoprothiolane (9.4 months) (Suzuki et al., 1998) can come in passive ways, such as pesticide residues detected in sample No. 1, No. 5, and No. 11. The second, pesticides with short soil half-lives such as fenoxanil (3.3–4.4 days) (Fu et al., 2016), azoxystrobin (7.5 days) (Gajbhiye et al., 2011), cypermethrin (0.5–8 weeks) (Paul, 2005) and chlorpyrifos (18.7 and 13.9) (Hwang et al., 2018) might actively be applied by farmers.

Table 1. Results of analysis of pesticide residues in rice fields of the organic rice production model in An Giang province during 2019 - 2021

Sample No.	Sampling sites	Sowing time	Sampling time	Detected pesticide substances	LOD	PC (mg kg^{-1})	PP
1	Tri Ton	20/01/2019	07/03/2019	Tricyclazole	0.005	0.71	1
				Propiconazole	0.007	0.6	1
2#	Tri Ton	20/01/2019	07/03/2019	ND	-	-	-
3*	Tri Ton	20/01/2019	07/03/2019	Isoprothiolane	0.003	0.02	1
				Tricyclazole	0.005	0.07	2
4	Tri Ton	22/06/2019	04/09/2019	Chlorpyrifos	0.002	0.01	1
				Hexaconazole	0.007	0.015	1
				Tricyclazole	0.005	0.01	3
				Chlorfenapyr	0.008	0.063	1
5	Tri Ton	25/10/2019	23/12/2019	Chlorpyrifos	0.002	0.022	2
				Isoprothiolane	0.003	0.021	2
				Tricyclazole	0.005	0.022	4
6	Tri Ton	25/10/2019	23/12/2019	Chlorpyrifos	0.002	0.011	3
				Isoprothiolane	0.003	0.03	3
				Tricyclazole	0.005	0.031	5
				Hexaconazole	0.007	0.011	2
7	Thoai Son	03/01/2020	31/03/2020	Chlorpyrifos	0.002	0.01	4
				Difenoconazole	0.003	0.038	1
				Hexaconazole	0.007	<0.02	3
				Isoprothiolane	0.003	0.01	4
				Metalaxyl & metalaxyl-M	0.003	2.1	1
				Propiconazole	0.007	0.036	2

				Tricyclazole	0.005	0.021	6
8	Tri Ton	05/01/2020	20/03/2020	Azoxystrobin	0.005	0.22	1
				Chlorfenapyr	0.008	0.053	2
				Chlorpyrifos	0.002	0.016	5
				Difenoconazole	0.003	0.56	2
				Fenoxanil	0.003	0.019	1
				Fipronil	0.002	0.0065	1
				Fipronil-desulfinyl	0.002	0.005	1
				Propiconazole	0.007	<0.02	3
				Isoprothiolane	0.003	0.014	5
				Hexaconazole	0.007	0.028	4
				Tricyclazole	0.005	0.16	7
9	Tri Ton	15/10/2020	27/11/2020	Chlorpyrifos	0.002	0.01	6
10	Tri Ton	15/10/2020	27/11/2020	Azoxystrobin	0.005	0.036	2
				Chlorfenapyr	0.008	<0.025	3
				Chlorfenson	0.005	0.11	1
				Cypermethrin	0.003	<0.01	1
				Difenoconazole	0.003	1.4	3
				Niclosamide	0.003	0.2	1
				Hexaconazole	0.007	1.2	5
				Tricyclazole	0.005	0.16	8
				Propiconazole	0.007	1.3	4
11	Thoai Son	22/01/2021	14/04/2021	Hexaconazole	0.007	0.016	6
				Isoprothiolane	0.003	0.036	6
				Tricyclazole	0.005	0.01	9
12	Tri Ton	20/06/2021	17/08/2021	Chlorfenapyr	0.008	0.015	4
				Cypermethrin	0.003	0.042	2
				Difenoconazole	0.003	0.033	4
				Paclobutrazol	0.003	0.054	1
				Propiconazole	0.007	0.036	5
				Tricyclazole	0.005	0.023	10
13	Tri Ton	25/06/2021	24/08/2021	Azoxystrobin	0.005	0.015	3
				Chlorpyrifos	0.002	0.017	7
				Difenoconazole	0.003	0.01	5
				Isoprothiolane	0.003	0.01	7
				Tricyclazole	0.005	0.01	11
14	Tri Ton	23/06/2021	24/08/2021	Chlorfenapyr	0.008	0.01	5
				Cypermethrin	0.003	0.011	3
				Difenoconazole	0.003	<0.01	6
				Paclobutrazol	0.003	<0.01	2
				Propiconazole	0.007	<0.01	6
				Tricyclazole	0.005	0.01	12
15	Tri Ton	20/06/2021	14/09/2021	ND	-	-	-

Note. #: water sample; *: soil sample, ND: Not detected

3.2 Summarizing the Presence of Pesticide Residues in Organic Rice Fields in An Giang Province

The contaminated pesticides in this study can be classified according to their contamination rate into 3 groups. The high-risk group has pesticides with a contamination rate greater than 50%, including only tricyclazole (80%). The medium-risk group is those with contamination rate ranged from 10% to 50%, including chlorpyrifos (47%), isoprothiolane (47%), difenoconazole (40%), propiconazole (40%), hexaconazole (40%), chlorfenapyr (33%), azoxystrobin (20%), and cypermethrin (20%). The low-risk group is those with a contamination rate of less than 10%, including metalaxyl & metalaxyl-M, paclobutazol, niclosamide, chlorfenson, fipronil, fipronil-desulfinyl, and fenoxanil, detected with a contamination rate of 7% (Fig. 2). Among them, tricyclazole was the most prominent with a contamination rate of 80%. This is reasonable because tricyclazole (5-methyl-1,2,4-triazolo[3,4-b]benzothiazole) is a unique fungicide to control rice blast disease caused by the fungus *Pyricularia oryzae* (Peterson, 1990). It is worth noting that among these 16 active substances, there are substances such as chlorpyrifos and fipronil that have been prohibited from use by the Ministry of Agriculture and Rural Development (MARD, 2019). Abroad, the use of some fipronil-based products in domestic animals has not been recommended for a long time (Colin et al., 2003).

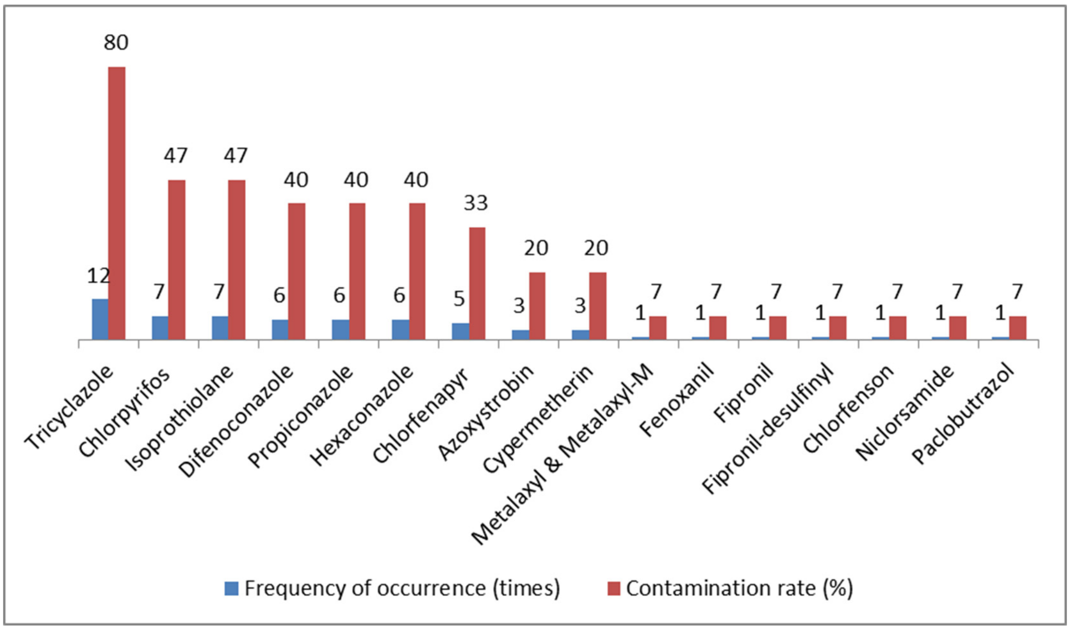

Figure 2. Frequency of occurrence and contamination rate (%) of pesticide residues in rice fields of the organic rice production model in An Giang province during 2019 – 2021

Compared to other research on pesticides assessment in Mekong Delta, Berg (2001) reported that 64 different pesticides were used in rice and rice-fish farms of the Mekong River delta. Therein, five pesticides, including propiconazole, hexaconazole, isoprothiolane, cypermethrin, and fipronil, are matched with the result of this study. A decade after that, another study by Berg and Tam (2012) reported that twenty pesticides used most in rice-fish farms by farmers in Tien Giang and Can Tho provinces in 2007, in which seven of them are matched with this discussing result, including propiconazole, hexaconazole, isoprothiolane, tricyclazole, cypermethrin, fipronil, and chlorpyrifos. Along with that, the study on pesticides and antibiotics in permanent rice, alternating rice-shrimp and permanent shrimp systems of the coastal Mekong Delta, Vietnam by Braun et al, (2019) reported that analyzed chemicals comprised 12 pesticides most commonly used in rice paddies, among them seven pesticides are found in this study, including chlorpyrifos, fipronil, difenoconazole, propiconazole, hexaconazole, isoprothiolane, and azoxystrobin. Summarizing the previous researches and this study we can infer that propiconazole, hexaconazole, isoprothiolane, and fipronil were the main contaminated pesticides in rice production in An Giang province in detail and Mekong River delta in general for more than two decades.

In this study, among 16 detected substances, there were 7 insecticides, 7 fungicides, 1 snail killer, and 1 growth regulator (Table 2). We can realize that various types of pesticides were detected in organic rice fields. This is more evidence to prove that farmers had broken the rules in organic production to apply pesticides in their fields, which agrees with Nguyen and Van's report (2021).

Table 2. Summarizing the presence of pesticide residues in rice fields of the organic rice production model in An Giang province during 2019 - 2021

No.	Pesticide substances	Main usages	CR (%)
1	Tricyclazole	Fungicide	80
2	Chlorpyrifos	Fungicide	47
3	Isoprothiolane	Fungicide	40
4	Difenoconazole	Fungicide	40
5	Propiconazole	Insecticide	40
6	Hexaconazole	Fungicide	40
7	Chlorfenapyr	Fungicide	33
8	Azoxystrobin	Insecticide	20
9	Cypermethrin	Insecticide	20
10	Metalaxyl & metalaxyl-M	Fungicide	13
11	Fenoxanil	Insecticide	7
12	Fipronil	Insecticide	7
13	Fipronil-desulfinyl	Insecticide	7
14	Chlorfenson	Insecticide	7
15	Niclorsamide	Snail killer	7
16	Paclobutrazol	Growth regulator	7

4. Conclusion and Recommendation

The study of evaluating the presence of pesticide residues, which are prohibited from being used in organic production in An Giang province, Vietnam, has been summarized. Thereby, 16 pesticide residues have been recorded with the contamination rate compared to the total number of samples tested from 2019 to 2021 as follows: the high-risk group includes tricyclazole (80%); The medium-risk group includes chlorpyrifos (47%), isoprothiolane (47%), difenoconazole (40%), propiconazole (40%), hexaconazole (40%), chlorfenapyr (33%), azoxystrobin (20%), and cypermethrin (20%); The low-risk group includes metalaxyl & metalaxyl-M, paclobutazol, niclosamide, chlorfenson, fipronil, fipronil-desulfinyl, and fenoxanil, all were detected with a contamination rate of 7%. There were 7 insecticides, 7 fungicides, 1 snail killer, and 1 growth regulator.

Research results serve as the basis for pre-inspecting raw material areas for organic rice production to reduce risks, analysis, and evaluation costs. We recommend that the provincial, national, and international organizations increase funding support for AOI researchers to organize and build the linkage models in rice value chains according to organic standards in the rice-intensive farming areas to minimize the application of harmful pesticides in the environment.

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Conflicts of interest

The authors declare that they have no conflict of interest.

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Appendix 1

List of pesticides under audit program of Control Union tested in SGS Laboratory (SGS Vietnam Co. Ltd.)

LC-MS/MS (LOD* mg/kg)

1-Naphthylacetamide	0.003	Daimuron	0.003	Imazalil	0.005	Phosfolan-methyl	0.003	Thifluzamide	0.01
4-(Trifluoromethyl)nicotinamide	0.002	DEET (Diethyl-m-toluamide, N,N-)	0.005	Imibenconazole	0.007	Phosmet	0.01	Thiobencarb	0.003
Abamectin (sum)	.(a)	Demeton (sum of Demeton-O and Demeton-S)	.(a)	Imidacloprid	0.005	Phosmet (phosmet and phosmet oxon expressed as phosmet)	0.01	Thiodicarb	0.01
Abamectin B1a (Avermectin B1a)	0.02	Demeton-S-methyl sulfone	0.003	Imidaclothiz	0.003	Phosmet-oxon	0.01	Thiophanate-methyl	0.003
Abamectin B1b (Avermectin B1b)	0.02	Demeton-S-methyl sulfoxide (Oxydemeton-methyl)	0.003	Indanofan	0.005	Phosphamidon	0.003	Thiram	0.01
Acephate	0.002	Desmedipham	0.007	Indaziflam	0.002	Phoxim	0.003	Tiadinil	0.01
Acequinocyl	0.01	Diafenthiuron	0.007	Indoxacarb (sum)	0.005	Pinoxaden	0.01	Tolfenpyrad	0.003
Acetamiprid	0.003	Dichlorvos	0.007	Iodosulfuron-methyl (sum)	0.008	Piperonyl butoxide	0.003	Tolylfluanid	0.01
Acibenzolar-S-methyl	0.007	Diclomezine	0.01	Isofetamid	0.002	Pirimicarb	0.003	Tolylfluanid (sum)	.(a)
Aclonifen	0.007	Dicrotophos	0.002	Isonoruron	0.005	Pirimicarb-Desmethyl	0.002	Topramezone	0.003
Albendazole	0.003	Diflubenzuron	0.01	Isoprocarb	0.005	Prochloraz	0.005	Tralkoxydim (sum of isomers)	0.008

Aldicarb	0.007	Diflufenican	0.007	Isoprothiolane	0.003	Profoxydim (sum of R- and S-isomers)	0.005	Triadimefon	0.003
Aldicarb (sum)	-(a)	Dimethametryn	0.005	Isoproturon	0.003	Promecarb	0.003	Triasulfuron	0.003
Aldicarb sulfone	0.002	Dimethoate	0.002	Isopyrazam	0.003	Propamocarb (sum)	0.002	Tribenuron methyl	0.01
Aldicarb sulfoxide	0.003	Dimethomorph (sum of isomers)	0.003	Isotianil	0.005	Propanil	0.003	Trichlorfon	0.007
Allethrin	0.007	Dimethylaniline, 2,4-	0.007	Isouron	0.005	Propaphos	0.008	Tricyclazole	0.005
Allidochlor	0.003	Dinotefuran	0.003	Isoxaben	0.003	Propaquizafop	0.003	Tridemorph	0.007
Alloxydim	0.007	Dioxathion (sum of isomers)	0.005	Isoxadifen-ethyl	0.008	Propargite	0.007	Trifloxystrobin	0.003
Ametoctradin	0.008	Disulfoton Sulfone	0.005	Isoxaflutole	0.01	Propoxur	0.003	Trifloxysulfuron	0.003
Ametryn	0.003	Disulfoton Sulfoxide	0.007	Isoxathion	0.007	Propoxycarbazone	0.01	Triflumezopyrim	0.005
Amidosulfuron	0.008	Diuron	0.005	Ivermectin (22,23-dihydroavermectin B1a)	0.01	Propoxycarbazone (propoxycarbazone)	0.01	Triflumuron	0.003
Amisulbrom	0.008	DMSA (Dimethylaminosulfanilide)	0.01	Jasmolin I	0.03	Propoxycarbazone-2-OH	0.007	Triflusulfuron-methyl	0.007
Anilazine	0.01	DMST (Dimethylaminosulfotoluidide)	0.01	Jasmolin II	0.03	Propyrisulfuron	0.007	Triforine	0.01
Aramite	0.003	Dodemorph	0.008	Lenacil	0.003	Proquinazid	0.003	Tritosulfuron	0.01
Asulam	0.007	Dodine	0.007	Linuron	0.007	Prosulfocarb	0.002	Valifenalate	0.003
Atrazine-desisopropyl	0.002	Edifenphos	0.005	Lufenuron (any ratio of constituent isomers)	0.01	Prosulfuron	0.005	Vamidothion	0.005
Azadirachtin	0.02	Emamectin benzoate B1a, expressed as emamectin	0.007	Malaoxon	0.005	Prothioconazole-desthio	0.005	Vernolate	0.003
Azamethiphos	0.005	EPTC	0.008	Malathion	0.005	Pymetrozine	0.005	Warfarin	0.003
Azimsulfuron	0.005	Ethaboxam	0.01	Malathion (sum)	-(a)	Pyracarbolid	0.005	XMC	0.003
Azinphos-methyl	0.007	Ethametsulfuron-methyl	0.003	Mandipropamid	0.005	Pyraclofos	0.005	Ziram	0.005
Azoxystrobin	0.005	Ethiofencarb	0.01	Mebendazole	0.002	Pyraclonil	0.007	Zoxamide	0.003
Barban	0.007	Ethiofencarb (sum)	-(a)	Mepanipyrim	0.005	Pyraclostrobin	0.005	Acequinocyl-hydroxy	0.01
Bendiocarb	0.003	Ethiofencarb-sulfone	0.003	Mephosfolan	0.005	Pyrasulfotole	0.003	Atrazine, 2-hydroxy-	0.01
Benodanil	0.003	Ethiofencarb-sulfoxide	0.003	Mepronil	0.003	Pyrazolynate	0.01	Benzobicyclon	0.003
Bensulfuron-methyl	0.003	Ethion	0.008	Merphos	0.003	Pyrazosulfuron-ethyl	0.003	Bicyclopyrone	0.003
Bensulide	0.008	Ethiprole	0.005	Mesosulfuron-methyl	0.01	Pyrazoxyfen	0.003	Bromadiolone	0.01
Bentazone	0.005	Ethirimol	0.003	Mesotrione	0.007	Pyrethrin I	0.02	Bufencarb	0.003

Benthiavalicarb	0.003	Ethoprophos	0.007	Metaflumizone (sum of E- and Z-isomers)	0.005	Pyrethrin II	0.02	Cafenstrole	0.003
Benthiazole (TCMTB)	0.01	Ethoxysulfuron	0.007	Metalaxyl & Metalaxyl-M and Metalaxyl & Metalaxyl-M-M (sum of isomers)	0.003	Pyrethrins (sum)	.(a)	Coumatetralyl	0.01
Benzovindiflupyr	0.01	Etrimfos	0.007	Metamifop	0.007	Pyribenzoxim	0.01	Cyenopyrafen	0.01
Benzoximate	0.007	Famoxadone	0.01	Metamitron	0.003	Pyridaphenthion	0.003	Deguelin	0.003
Benzyladenine, 6-	0.007	Fenamiphos	0.01	Methabenzthiazuron	0.003	Pyridate	0.003	Denatonium Benzoate (sum)	0.003
Bifenazate (sum)	0.007	Fenamiphos (sum)	.(a)	Methamidophos	0.003	Pyrifenox	0.01	Diclosulam	0.003
Bistrifluron	0.01	Fenamiphos sulfone	0.005	Methiocarb	0.005	Pyrifluquinazon	0.003	Diphacinone	0.01
Boscalid	0.005	Fenamiphos sulfoxide	0.005	Methiocarb (sum)	.(a)	Pyrimethanil	0.007	Doramectin	0.01
Brodifacoum	0.002	Fenbuconazole	0.003	Methiocarb sulfone	0.01	Pyrimidifen	0.002	Eprinomectin B1a	0.01
Bromacil	0.007	Fenfuram	0.003	Methiocarb sulfoxide	0.01	Pyroquilon	0.005	Fenpicoxamid	0.003
Bromfenvinfos-methyl	0.003	Fenhexamid	0.005	Methomyl	0.01	Pyroxsulam	0.005	Fensulfothion-oxon	0.003
Buprofezin	0.003	Fenobucarb	0.008	Methomyl and Thiodicarb (sum)	.(a)	Quinalphos	0.003	Flocoumafen	0.01
Butocarboxim	0.003	Fenothiocarb	0.003	Methoprotryne	0.005	Quinoclamine	0.007	Flucetosulfuron	0.003
Carbaryl	0.003	Fenoxanil	0.003	Methoxyfenozide	0.01	Quinoxyfen	0.005	halauxifen-methyl	0.003
Carbendazim and benomyl (sum)	0.003	Fenoxycarb	0.003	Metobromuron	0.003	Rimsulfuron	0.007	Imicyafos	0.003
Carbetamide (sum)	0.003	Fenpiclonil	0.007	Metolcarb	0.003	Rotenone	0.005	Metazachlor Metabolite 479M6	0.002
Carbofuran	0.002	Fenpyrazamine	0.007	Metominostrobin	0.007	Saflufenacil	0.01	Metazosulfuron	0.003
Carbofuran (sum)	.(a)	Fenpyroximate	0.003	Metosulam	0.002	Sedaxane	0.003	Neburon	0.003
Carbofuran-3-OH	0.002	Fensulfothion	0.003	Metoxuron	0.003	Sethoxydim	0.007	Niclosamide	0.003
Carbophenothion	0.007	Fensulfothion-oxon-sulfone	0.005	Metsulfuron methyl	0.003	Siduron	0.002	Norea (noruron)	0.003
Carbosulfan	0.01	Fensulfothion-sulfone	0.008	Mevinphos (sum of E- and Z-isomers)	0.002	Simazine	0.007	Oxolinic acid	0.003
Carboxin	0.003	Fenthion	0.005	Milbemectin (sum)	0.01	Simetryn	0.003	Prothoate	0.003
Carpropamid	0.003	Fenthion sulfone	0.007	Molinate	0.007	Spinetoram (175-J + 175-L)	.(a)	Pyribencarb	0.003
Chlorantraniliprole	0.005	Fenthion sulfoxide	0.003	Monocrotophos	0.003	Spinetoram 175-J	0.008	Pyriftalid	0.003
Chlorbenzuron	0.02	Fentrazamide	0.007	Monolinuron	0.005	Spinetoram 175-L	0.008	Ronidazole	0.003
Chlorbromuron	0.005	Fenuron	0.003	Monuron	0.003	Spinosad (sum of spinosyn A + D)	.(a)	Temephos	0.003
Chlorfluazuron	0.008	Ferimzone	0.005	MPMC (Xylylcarb)	0.003	Spinosyn A	0.01	Tiafenacil	0.003
Chloridazon	0.003	Fipronil	0.002	Napropamide	0.005	Spinosyn D	0.01	Tritosulfuron	0.003

								Metabolite AMTT	
Chlorimuron-ethyl	0.007	Fipronil (sum)	-(a)	Nicosulfuron	0.005	Spirodiclofen	0.008	Abamectin B1a, 8,9-Z (Avermectin B1a, 8,9-Z-)	0.01
Chloroaniline, 3-	0.005	Fipronil desulfinyl	0.002	Nicotine	0.003	Spiromesifen	0.01	Alanycarb	0.01
Chlorotoluron	0.003	Fipronil sulfide	0.002	Nitenpyram	0.005	Spirotetramat	0.01	Amitraz	0.005
Chloroxuron	0.005	Fipronil Sulfone	0.002	Nitralin	0.01	Spirotetramat (sum)	-(a)	Amitraz (sum)	-(a)
Chlorpyrifos-methyl	0.003	Florasulam	0.005	Norflurazon	0.005	Spirotetramat-cis-enol	0.01	Chloridazon (sum)	-(a)
Chlorsulfuron	0.005	Fluazifop-P-butyl	0.007	Novaluron	0.007	Spirotetramat-cis-keto-hydroxy	0.01	Chloridazon-desphenyl	0.003
Chlorthiamid	0.003	Fluazinam	0.007	Ofurace	0.003	Spirotetramat-enol-glucoside	0.003	Dikegulac	0.01
Chromafenozide	0.007	Flubendazole	0.002	Omethoate	0.002	Spirotetramat-mono-hydroxy	0.003	DNOC	0.003
Cinerin I	0.05	Flubendiamide	0.007	Orthosulfamuron	0.01	Spiroxamine (sum of isomers)	0.01	Fenthion	-(a)
Cinerin II	0.05	Flucycloxuron	0.007	Oryzalin	0.01	Sulcotrione	0.007	Fenthion-oxon	0.003
Cinosulfuron	0.005	Flufenoxuron	0.01	Oxadiargyl	0.007	Sulfaquinoxaline	0.003	Fenthion-oxon-sulfone	0.003
Clethodim	0.003	Flumethrin	0.01	Oxamyl	0.003	Sulfentrazone	0.007	Fenthion-oxon-sulfoxide	0.003
Clethodim (sum)	-(a)	Fluometuron	0.008	Oxasulfuron	0.005	Sulfosulfuron	0.007	Flazasulfuron	0.007
Clofentezine	0.007	Flupyradifurone	0.002	Oxathiapiprolin	0.002	Sulfoxaflor (sum of isomers)	0.005	Imazosulfuron	0.01
Clomeprop	0.003	Flupyrsulfuron-methyl	0.003	Oxaziclomefone	0.007	Sulprofos	0.008	Isoxaflutole (sum)	-(a)
Clothianidin	0.003	Fluridone	0.007	Oxibendazole	0.002	Tebufenozide	0.01	Isoxaflutole diketonitrile RPA 202248	0.01
Coumaphos	0.005	Flusulfamide	0.003	Oxycarboxin	0.003	Tebutam	0.002	Naled	0.01
CPMC (Etrofol)	0.007	Fluthiacet-methyl	0.007	Oxydemeton-methyl (sum)	-(a)	Tebuthiuron	0.005	Phorate-oxon	0.01
Cyanazine	0.002	Fluxapyroxad	0.003	Oxymatrine	0.003	Teflubenzuron	0.007	Phorate-oxon-sulfone	0.002
Cyantraniliprole	0.007	Foramsulfuron	0.01	Paclobutrazol	0.003	Tembotrione	0.01	Phorate-oxon-sulfoxide	0.002
Cyazofamid	0.003	Forchlorfenuron	0.005	Paraoxon (-ethyl)	0.005	Tepraloxydim	0.008	Pirimicarb (sum)	-(a)
Cyclaniliprole	0.003	Formetanate (Sum)	0.005	Paraoxon-methyl	0.007	Terbufos-sulfone	0.01	Pirimicarb-desmethyl-formamido	0.003
Cycloprothrin	0.02	Fuberidazole	0.002	Pencycuron	0.003	Terbufos-sulfoxide	0.007	Prothioconazole and prothioconazole-desthio (sum)	-(a)

Cyclosulfamuron	0.01	Halosulfuron-methyl	0.007	Penflufen	0.002	Tetraconazole	0.005	Pyraclostrobin (sum)	-(a)
Cycloxydim	0.007	Hexaflumuron	0.01	Penoxsulam	0.007	Thiabendazole	0.003	pyraclostrobin metabolite	0.01
Cyflumetofen	0.02	Hexazinone	0.005	Penthiopyrad	0.005	Thiacloprid	0.003	Saflufenacil (sum of saflufenacil,	-(a)
Cymoxanil	0.007	Hexythiazox	0.003	Phenmedipham	0.003	Thiamethoxam	0.003	Saflufenacil Metabolite M800H11	0.003
Cyprazine	0.007	Hydroxyquinoline, 8-(sum)	0.01	Phorate sulfoxide	0.002	Thidiazuron	0.003	Saflufenacil Metabolite M800H35	0.01
Cyromazine	0.007	Hymexazol	0.008	Phosfolan (-ethyl)	0.003	Thifensulfuron -methyl	0.005		
Dichlofluanid (sum)	-(a)	Disulfoton (sum)	-(a)	Phorate (sum)	-(a)	Prochloraz (sum)	-(a)		

Note: (a) Refer to LOD/LOQ of the individual pesticides that were used to calculate the sum item

GC-MS/MS (LOD* mg/kg)

2,6-Diisopropylnaphthalene	0.003	Cyanofenphos	0.008	Fenamidone	0.005	Leptophos	0.003	Pyriminobac-methyl (sum)	0.003
2-Naphthol	0.02	Cyanophos	0.005	Fenarimol	0.003	Mecarbam	0.007	Pyriproxyfen	0.003
Acetochlor	0.005	Cycloate	0.003	Fenazaquin	0.003	Mefenacet	0.003	Quintozene	0.005
Acrinathrin	0.003	Cyflufenamide (sum)	0.007	Fenchlorphos	0.003	Mefenpyr-diethyl	0.007	Quintozene and Pentachloroaniline (sum)	-(a)
Alachlor	0.003	Cyfluthrine (sum of isomers)	0.003	Fenchlorphos (sum)	-(a)	Metazachlor	0.005	Quizalofop-ethyl	0.005
Aldrin	0.003	Cyhalofop-butyl	0.008	Fenchlorphos oxon	0.005	Metconazole (sum of isomers)	0.005	Resmethrin (sum)	0.008
Aldrin and Dieldrin (sum, expressed as dieldrin)	-(a)	Cyhalothrin-lambda + Cyhalothrin-gamma (sum)	0.003	Fenclorim	0.005	Methacrifos	0.007	S421	0.02
Anilofos	0.007	Cymiazole	0.007	Fenitrothion	0.003	Methidathion	0.003	Secbumeton	0.003
Anthraquinone	0.003	Cypermethrin (sum of isomers)	0.003	Fenoxaprop-ethyl (sum)	0.008	Methoprene	0.003	Silafluofen	0.003
Aspon	0.002	Cyphenothrin (sum)	0.005	Fenpropathrin	0.005	Methoxychlor	0.003	Silthiofam	0.007
Atraton	0.003	Cyproconazole	0.005	Fenpropidin (sum)		Metolachlor and S-metolachlor (sum of isomers)	0.003	Simeconazole	0.008
Atrazine	0.008	Cyprodinil	0.005	Fenpropimorph (sum of isomers)	0.003	Metrafenone	0.007	Sulfotep	0.007

Atrazine-desethyl	0.003	Dazomet	0.01	Fenson	0.007	Metribuzin	0.003	TDE, p,p'-	0.005
Azaconazole	0.007	DBCP (Dibromo-3-chloropropane, 1,2-)	0.005	Fenvalerate	0.005	MGK 326	0.003	Tebuconazole	0.005
Azinphos-ethyl	0.008	DDD-o,p'-	0.005	Flonicamid	0.007	MGK-264	0.007	Tebufenpyrad	0.003
Beflubutamid	0.007	DDE-o,p'-	0.003	Fluacrypyrim	0.003	Mirex	0.003	Tebupirimfos	0.005
Benalaxyl+Benalaxyl-M (sum)	0.005	DDE-p,p'-	0.005	Fluchloralin	0.005	Myclobutanil	0.005	Tecnazene	0.005
Benfluralin	0.003	DDT (sum of p,p´-DDT, o,p´-DDT,		Flucythrinate (sum of isomers)	0.003	Nitrapyrin	0.007	Tefluthrin	0.003
Benoxacor	0.008	p,p´-DDE and p,p´-TDE expressed as DDT)	- (a)	Fludioxonil	0.007	Nitrofen	0.003	TEPP	0.01
Benzoylprop-ethyl	0.005	DDT-o,p'-	0.005	Fluensulfone	0.007	Nitrothal-isopropyl	0.005	Terbacil	0.007
Bifenox	0.005	DDT-p,p'- DDT (sum of p,p´-DDT, o,p´-DDT,	0.005	Flufenacet	0.003	Nonachlor, cis-	0.003	Terbufos	0.003
Bifenthrin (sum of isomers)	0.007	Deltamethrin and Tralomethrin (sum)	0.003	Flufenpyr-ethyl	0.008	Nonachlor, trans-	0.003	Terbumeton	0.003
Binapacryl	0.01	Demeton-S-methyl	0.01	Flufiprole	0.005	Nuarimol	0.003	Terbuthylazine	0.005
Bioresmethrin (cis-trans)	0.008	Desmetryn	0.007	Flumetralin	0.005	Octachlorostyrene	0.002	Terbutryn	0.007
Biphenyl	0.01	Di-allate (sum of isomers)	0.003	Flumiclorac-pentyl	0.005	Oxadiazon	0.003	Tetrachloroaniline, 2,3,5,6-	0.005
Bitertanol (sum of isomers)	0.005	Diazinon	0.003	Flumioxazin	0.005	Oxadixyl	0.003	Tetrachlorvinphos	0.007
Bixafen	0.007	Dichlobenil	0.01	Fluopicolide	0.003	Oxyfluorfen	0.005	Tetradifon	0.007
Bromobutide	0.003	Dichlofenthion	0.002	Fluopyram	0.007	Parathion (-ethyl)	0.005	Tetramethrin (sum of isomers)	0.003
Bromocyclen	0.003	Dichlofluanid	0.01	Fluorodifen	0.002	Parathion-methyl	0.005	Tetrasul	0.007
Bromophos (-methyl)	0.003	Dichlormid	0.005	Fluoroglycofen-ethyl	0.003	Parathion-methyl (sum)	-(a)	Thenylchlor	0.005
Bromophos-ethyl	0.007	Dichloroaniline, 3,5-	0.003	Fluoxastrobin (sum)	0.01	Pebulate	0.003	Thiazopyr	0.005
Bromopropylate	0.003	Dichlorophenol, 2,4-	0.007	Fluquinconazole	0.005	Penconazole	0.005	Thiocyclam	0.01
Bromoxynil-octanoate	0.002	Diclobutrazol	0.007	Flurochloridone	0.005	Pendimethalin	0.003	Thiofanox	0.007

Bromuconazole (sum of diasteroisomers)	0.003	Diclofop-methyl	0.003	Fluroxypyr-meptyl	0.003	Pentachloroaniline	0.003	Thiometon	0.008
Bupirimate	0.005	Dicloran	0.008	Flurprimidol	0.003	Pentachloroanisole	0.003	Thionazin	0.003
Butachlor	0.003	Dicofol (-o,p')	0.007	Flurtamone	0.003	Pentachlorobenzene	0.003	Tiocarbazil	0.005
Butafenacil	0.003	Dicofol (-p,p')	0.003	Flusilazole	0.007	Pentachlorophenol	0.007	Tolclofos-methyl	0.007
Butamifos	0.003	Dicofol (sum)	- (a)	Flutolanil	0.005	Pentachlorothioanisole	0.003	Transfluthrin	0.005
Butralin	0.005	Dieldrin	0.005	Flutriafol	0.003	Pentoxazone	0.005	Triadimenol (sum of isomers)	0.007
Butylate	0.003	Diethatyl-ethyl	0.003	Fluvalinate (sum)	0.003	Permethrin (sum)	0.007	Triadimenol (sum)	0.007
Cadusafos	0.005	Diethofencarb	0.007	Fonofos	0.003	Perthane	0.007	Triallate	0.005
Carbofuran-3-keto	0.005	Difenoconazole	0.003	Formothion	0.007	Pethoxamide	0.005	Triazophos	0.003
Carbophenothion-Methyl	0.007	Diflovidazin	0.01	Fosthiazate	0.01	Phenothrin (sum of isomers)	0.008	Tribufos (DEF)	0.003
Carfentrazone-ethyl	0.003	Dimepiperate	0.007	Furalaxyl	0.007	Phenthoate	0.003	Trichlorobenzene,1,2,3-	0.003
Carvacrol	0.01	Dimethachlor	0.003	Furametpyr	0.007	Phenylphenol, 2-	0.005	Trichlorobenzene,1,2,4-	0.003
Chinomethionat	0.007	Dimethenamid (sum)	0.003	Halfenprox	0.003	Phorate	0.003	Trichlorobenzene,1,3,5-	0.003
Chlorbenside	0.003	Dimethipin	0.01	Haloxyfop-methyl	0.007	Phorate sulfone	0.007	Trichloronate	0.003
Chlorbufam	0.007	Dimethylvinphos	0.002	HCH-alpha	0.003	Phosalone	0.003	Trichlorophenol, 2,4,6-	0.01
Chlordane (sum of cis- and trans- chlordane)	-(a)	Dimoxystrobin	0.008	HCH-beta	0.005	Phthalide	0.007	Triclosan	0.01
Chlordane, cis-	0.003	Diniconazole (sum of isomers)	0.007	HCH-delta	0.005	Picolinafen	0.005	Tridiphane	0.01
Chlordane, oxy-	0.003	Dinitramine	0.005	HCH-epsilon	0.005	Picoxystrobin	0.005	Triflumizole	0.003
Chlordane, trans-	0.003	Diofenolan	0.005	HCH-gamma (Lindane)	0.003	Piperophos	0.003	Trifluralin	0.003
Chlordecone	0.005	Dioxabenzofos	0.003	Heptachlor	0.003	Pirimiphos-ethyl	0.003	Trimethacarb, 2,3,5-	0.007
Chlordimeform	0.003	Diphenamid	0.007	Heptachlor (sum)	-(a)	Pirimiphos-methyl	0.005	Trimethacarb, 3,4,5-	0.007
Chlorethoxyfos	0.008	Diphenylamine	0.005	Heptachlor endo-epoxide (isomer A)	0.005	Plifenate	0.01	Triticonazole	0.007
Chlorfenapyr	0.008	Ditalimfos	0.01	Heptachlor exo-epoxide (isomer B)	0.003	Prallethrin (sum of (R)- and (S)- stereoisomers)	0.01	Uniconazole	0.003
Chlorfenson	0.005	Dithiopyr	0.003	Heptenophos	0.005	Pretilachlor	0.007	Vinclozolin	0.003
Chlorfenvinphos	0.003	Endosulfan (sum)	- (a)	Hexachlorobenzene (HCB)	0.005	Procymidone	0.005	2,6-Dichlorobenzamide	0.003
Chlorflurenol-Methyl	0.007	Endosulfan I	0.003	Hexachlorocyclohexane (HCH), (sum)	-(a)	Profenofos	0.005	3-decen-2-one	0.003
Chlormephos	0.007	Endosulfan II	0.003	Hexaconazole	0.007	Profluralin	0.007	Benazolin-ethyl	0.003
Chlorobenzilate	0.002	Endosulfan sulfate	0.003	Hydroprene (sum)	0.003	Prometon	0.003	chlornitrofen	0.003

Chloroneb	0.003	Endrin	0.003	Imazamethabenz-methyl	0.01	Prometryn	0.003	Crufomate	0.003
Chloropropylate	0.007	Endrin aldehyde	0.003	Ipconazole	0.003	Propachlor	0.003	Demeton-O	0.01
Chlorpropham	0.007	Endrin ketone	0.003	Iprobenfos	0.008	Propazine	0.005	Demeton-S	0.01
Chlorpyrifos (-ethyl)	0.002	EPN	0.003	Iprodione	0.01	Propetamphos	0.007	Dialifos	0.003
Chlorthal-dimethyl	0.007	Epoxiconazole	0.002	Iprovalicarb	0.003	Propham	0.007	Dimefox	0.01
Chlorthion	0.007	Esprocarb	0.003	Isazofos	0.002	Propiconazole (sum of isomers)	0.007	Dimethylnaphthalene, 1,4-	0.01
Chlorthiophos	0.007	Etaconazole (sum)	0.002	Isobenzan	0.007	Propisochlor	0.007	Ethychlozate	0.003
Chlozolinate	0.003	Ethalfluralin	0.005	Isocarbophos	0.003	Propyzamide	0.003	Flutianil	0.01
Cinidon-ethyl (sum)	0.007	Ethofumesate	0.003	Isodrin	0.007	Prothioconazole	0.005	Mandestrobin	0.003
Cinmethylin (sum of isomers)	0.007	Ethofumesate (sum)	- (a)	Isofenphos	0.005	Prothiofos	0.005	Pentachlorobenzonitrile 2,3,4,5,6-	0.003
Clodinafop-propargyl	0.003	Ethofumesate, 2-keto-	0.002	Isofenphos-methyl	0.005	Pyraflufen-ethyl	0.007	Pyriofenone	0.003
Clomazone	0.003	Etofenprox	0.003	Isopropalin	0.005	Pyrazophos	0.007	Tetrahydrophthalimide 1,2,3,6-(THPI)	0.003
Cloquintocet-mexyl	0.003	Etoxazole	0.007	Jodfenphos	0.005	Pyributicarb	0.005	Triclopyr-2-Butoxyethyl	0.003
Crimidine	0.005	Etridiazole	0.007	Kresoxim-methyl	0.003	Pyridaben	0.007	Triflumizole (sum)	-(a)
Crotoxyphos	0.002	Famphur	0.003	Lactofen	0.003	Pyridalyl	0.003	Triflumizole metabolite FM-6-1	0.003

Note: (a) Refer to LOD of the individual pesticides that were used to calculate the sum item

LC-MS/MS + GC-MS/MS (LOD* mg/kg)

Dichlofluanid (sum)	- (a)	Disulfoton (sum)	- (a)	Phorate (sum)	- (a)	Prochloraz (sum)	- (a)		

Note: (a) Refer to LOD/LOQ of the individual pesticides that were used to calculate the sum item