Quality Assessment of Soil and Rice from Good Agricultural Practice Rice Crops

Khin Moe Phyu, Pravate Tuitemwong, Kooranee Tuitemwong, Worapot Suntornsuk, and Panthira Ketkaew

Abstract— *Good Agricultural Practices (GAP) stands for food safety and quality, and the environmental sustainability of agriculture. Residues testing and certification are some of the main challenges related to GAP implementation. In this study a multiresidue method for the determination of 16 organochlorine pesticides in soil and rice using gas chromatography with electron capture detector (GC-ECD) was achieved and validated. Samples were prepared by QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. The linear range used in the calibration curves was from 0.01 to 1.0 mgkg^{-1} and the results showed that the calibration curve linearity was ≥0.99 for all target analytes. Analyses of fortified soil and rice samples were performed at different levels (0.05, 0.25 and 0.5 mgkg^{-1}).Mean recoveries from five replicates ranged from 70% to 120% for most target analytes except endosulfan-sulfate and endrin-ketone in both rice and soil. The RSD values were <20% for all compounds. Thirty samples each of soil and rice from GAP crops of northeast region of Thailand were analyzed by this method application.*

Keywords— **GAP, GC, Pesticides, QuEChERS.**

1. INTRODUCTION

Good Agricultural Practices (GAP) is a collection of principles to apply for on-farm production and post-production process [1]. The benefits of GAP for farmers and consumers are, to meet with specific objectives of food security, food quality, production efficiency, livelihood and environmental protection, economic and social sustainability [2].

The use of fertilizers and pesticides has steadily increased year to year for sustainable high crop yields. Pesticides may contaminate to soil through direct application, accidental emission, and run-off from plant surfaces or from absorption of pesticide contaminated plant materials [3]. Pesticides may cause acute and delayed health effects in those who are exposed. Exposure of pesticides can cause a variety of adverse health effects, ranging from simple irritation of the skin and eyes to more severe effects such as affecting the nervous system, mimicking hormones causing reproductive problems, and also causing cancer. There are strong evidence for other negative outcomes from pesticide exposure including neurological, birth defects, fetal death, and neuro-developmental disorder [4].

Persistent ogranic pollutants (POPs) are organic compounds. They can resist photolytic, biological and chemical degradation. POPs are often halogenated and characterised by low water solubility and high lipid solubility. They are able to enter human food chain, bio-accumulation and mamalian toxicity. POPs are semi-volatile compounds, so their mobility through the atmosphere that is sufficient to allow relatively great amounts to enter the atmosphere and be transported over long distances. OC insecticides such as, aldrin, DDT, dieldrin, endrin, heptachlor are POPs compounds [5].

The Thailand Department of Rice GAP program aims to guide to the proper rice plantation resulting in high quality rice that is safe from chemicals and contamination, with high yield and high head rice. The program includes 3 on-site inspections and the testing of physical (percent head rice, contamination of other rice species and red rice), and chemical (pesticide residues) qualities of soil, water and rice, in the certification process [6]. Therefore pesticides residues testing played an important role to this program.

Residues testing and certification are one of the main challenges related to GAP implementation. Insecticides, rodenticides, herbicides and fungicides are four important types of pesticides used in controlling pests in agricultural areas [7]. The majority pesticides in Thailand rice farming were organophosphates (OP), organochlorines (OC), and carbamates.

There are many methods for determination of pesticides multi-residues in agricultural products and animal derived foods. But the traditional methods are usually multi-stage procedures, requiring large samples and one or more extract cleanup steps. Consequently, they are time-consuming, labour-intensive, complicated,

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expensive and produce considerable amounts of wastes. Furthermore, the traditional methods often give poor quantitation and involve a single analyte or analytes from a single class of compounds. QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method for the multiclass multiresidue analysis of pesticides in fruit and vegetables was developed in 2003 by Anastassiades *et al*. QuEChERS method have become the main analytical tools in most pesticide monitoring laboratories to meet world standards as a result of the wide analytical scope and high degree of slectivity and sensivity provided by gas and liquid chromatography (GC and LC). Lehotay *et al.* modified QuEChERS method to used relatively strong acetate buffering conditions and Anastassiades *et al*. chose to use weaker citrate buffering conditions and both methods successfully met statistical criteria for accepatibility from independent scientific standards organizations. Acetate-buffering version became AOAC Official Method 2007,01 and the citrate-buffering version was named European Committee for Standardization (CEN) Standard Method EN 15662 [8], [9].

This paper describes a method for the determination of organochlorine pesticide residues in soil and rice from GAP crops from northeast region of Thailand by QuEChERS (EN-15662) method using gas chromatography with electron capture detector (GC-ECD).

2. MATERIALS AND METHODS

2.1. Materials and reagents

Certified pesticide reference standards of high purity were purchased from Dr. Ehrenstorfer (Germany). Acetonitrile (Pesticide residues grade) was purchased from (RCI Labscan Limited). Disodium hydrogencitrate sesquihydrate was purchased from (MERCK 1.122 64.0500). Trisodium citrate dihydrate was purchased from (Sigma, Aldrich 32320). Bondesil-PSA 40 µm (Varian article no.12213024) was purchased from Crawford Scientific Ltd. Magnesium sulphate anhydrous coarsely grained were purchase from (Merck 1.06067). Magnesium sulfate fine powder was purchased from Sigma-Aldrich. Formic acid (85% extrapure) was purchased from (QReC BRIGHTCHEM SDN BHD). Pesticides analyses were done in RADAL (Risk and Decision Analysis Laboratory) Laboratory, Microbiology Department of King Mongkut's University of Technology Thonburi.

2.2. Samples

Soil and rice samples were obtained from GAP rice crops of northeast region of Thailand.

2.3. Apparatus and conditions

Gas chromatographic analysis was performed using PerkinElmer Auto-System XL GC-ECD with a fused silica capillary column (SPB-1701, 30 m x 0.32 mm x 0.25µm film thickness). Helium gas (99.9999% purity) was used as carrier gas at a constant flow-rate of 1 ml min^{-1}. The oven temperature program was 150 $^{\circ}$C at 4°C min^{-1} and raise to 275 $^{\circ}$C at 4 $^{\circ}$C min^{-1}. The injector temperature was set at 210°C and split ratio (2:1) with injection volume 1µL. The detector temperature was kept at 300°C.

Analytical balance (A&D Company and DENVER Instrument), refrigerated centrifuge (ScanSpeed 1580 MGR), shaker (New Brunswick Scientific), vortex (Scien-tific Industries) and dispersing homogenizer (ULTRA TURRAX IKA T-18 basic) were employed.

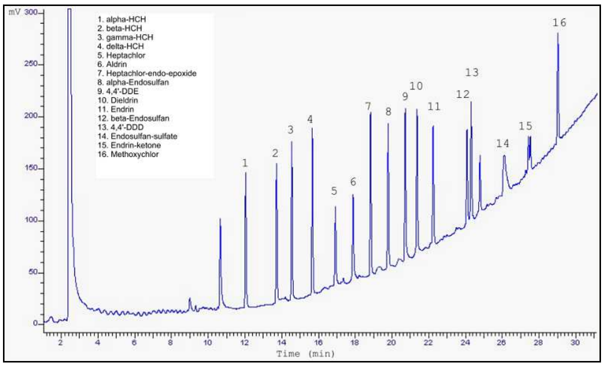


Fig.1. Chromatogram of organochlorine mixed standards at 0.1µg ml^{-1}

2.4. Sample homogenisation, extraction and clean-up

The QuEChERS extraction method was used for samples preparation. Dry samples with water content of lower than 25% like cereals, dried fruits and spices, were used. The sample amount may have to be reduced and water has to be added to make sample pores more accessible to the extraction solvent [8]. For the rice samples, 5 g of previously ground samples were weighed in a 50 ml centrifuge tube and added with 10 ml of cold deionized water (<4°C). Homogenized for 2 minutes and added with 10 ml of acetonitrile and shaken by hand for 1 minute. Four (4) g of $MgSO_4$, 1 g of NaCl, 1 g of trisodium citrate dihydrate and 0.5 g of sodium hydrogen (Na_2H) citrate sesqui-hydrate were added and vigorously vortex for 1 minute and centrifuged at 3000 rpm at 4°C for 5 minutes. After being centrifuged, 6 ml of supernatant (acetonitrile layer) were transferred into 15 ml centrifuge tube containing 200 mg of primary secondary amine (PSA) and 900 mg of $MgSO_4$. Vigorously shaken by vortex for 30 seconds and centrifuged at 3000 rpm for 5 minutes at 4 $^{\circ}$C then 4 ml of the cleaned extract were transferred into a screw cap vial and adjusted to pH between 5 to 5.5 with 5 % formic acid in acetonitrile. The extract was dried under mild stream of nitrogen. The dried extract was reconstituted with 500 µL acetonitrile and analyzed by GC/ECD [8] – [11].

For the soil samples, 20 g of soil samples were weighed in a 50 ml centrifuge tube. They were added with 12 ml of DI water and shaken for 4 hours. Then, 20 ml of acetonitrile were added and shaken by hand for 1 minute. The supernatant was transferred into a second centrifuge tube containing 6 g of $MgSO_4$, 1.5 g of NaCl, 1.5 g of Na_3-Citrate dihydrate and 0.75 g of Na_2H-Citrate-sesqui-hydrate. They were shaken for 1 minute and centrifuged at 3000 rpm for 5 minutes at 4°C [12].

Clean-up step was followed as of the rice samples preparation procedures and analyzed by GC/ECD.

2.5. Method performance and validation

Sixteen components of mixed organochlorine pesticides standard, containing Aldrin, 4,4'-DDD, 4,4'-DDE, Dieldrin, alpha-Endosulfan, beta-Endosulfan, Endosulfan-sulfate, Endrin, Endrin-ketone, alpha- HCH, beta-HCH, gamma-HCH, delta-HCH, Heptachlor, Heptachlor endo-epoxide (trans-, isomer A), Methoxychlor were used for the validation of the QuEChERS method.

For linearity study, the calibration curve was evaluated in the concentrations 0.01, 0.02, 0.03, 0.04, 0.05, 0.1, 0.25, 0.5 and 1.0 µg ml^{-1}.

For recovery study, (accuracy and precision) known pesticide concentration initially added to the soil and rice samples at three levels (0.05,0.25 and 0.5 µg ml^{-1}) and 5 replicates each.

3. RESULTS AND DISCUSSION

3.1. Chromatographic determination by GC-ECD

A good resolution of all pesticides studied was achieved as shown in the chromatogram (Fig.1).

Table.1. Determination of coefficient (r^2)

Sr.	Name of Organochlorines	Coefficient (r^2)
1.	alpha-HCH	0.9999
2.	beta-HCH	0.9998
3.	gamma-HCH	0.9998
4.	delta-HCH	0.9998
5.	Heptachlor	1
6.	Aldrin	0.9999
7.	Heptachlor-endo-epoxide	0.9997
8.	alpha-Endosulfan	0.9990
9.	4,4'-DDE	0.9984
10.	Dieldrin	0.9990
11.	Endrin	0.9999
12.	beta-endosulfan	0.9998
13.	4,4'-DDD	0.9999
14.	Endosulfan-sulfate	0.9988
15.	Endrin-ketone	0.9988
16.	Methoxychlor	0.9999

3.2. Method performance and recoveries

Calibration curves were linear over the range of 0.01 to 1.0 µg ml^{-1} with correlation coefficients of (r^2) ≥0.99 for all analytes (Table 1).

The recoveries of the studied pesticides at three levels (0.05, 0.25 and 0.5 µg ml^{-1}) were checked by spiking to the soil and rice samples. Mean results showed recoveries between 70% and 120% in most target analytes except endosulfan-sulfate and endrin-ketone in both rice and soil. The RSD values were <20% for all compounds**.** (Table 2).

Table 2. (a) Average recovery % and RSD% from rice samples spiked at three levels (n=5)

No.	Pesticide's name	Average recovery % ± RSD% for rice		
		0.05 mg kg^{-1}	0.25mg kg^{-1}	0.5 mg kg^{-1}
1	alpha-HCH	79 ± 5.5	82 ± 0.6	80 ± 6.9
2	beta-HCH	80 ± 15.5	85 ± 1.8	87 ± 5.2
3	gamma-HCH	69 ± 6.6	99 ± 2.7	104 ± 5.2
4	delta-HCH	107 ± 11.2	114 ± 3.6	108 ± 5.2
5	Heptachlor	72 ± 1.7	80 ± 7.8	89 ± 9.8
6	Aldrin	70 ± 3.8	88 ± 2.1	72 ± 5.2
7	Heptachlor-endo-epoxide	61 ± 8.1	78 ± 3.3	84 ± 4.4
8	alpha-Endosulfan	90 ± 11.3	95 ± 3.3	87 ± 6.9
9	4,4'-DDE	82 ± 3.5	85 ± 4.6	79 ± 6.3
10	Dieldrin	114 ± 4.8	92 ± 3.1	83 ± 13.3
11	Endrin	72 ± 18.7	94 ± 1.9	98 ± 5.0
12	beta-Endosulfan	85 ± 14.1	99 ± 3.6	97 ± 6.3
13	4,4'-DDD	79 ± 1.0	98 ± 2.7	101 ± 5.1
14	Endosulfan-sulfate	67 ± 1.8	66 ± 3.6	68 ± 9.2
15	Endrin-ketone	64 ± 5.9	66 ± 1.9	67 ± 1.0
16	Methoxychlor	74 ± 7.4	86 ± 2.0	77 ± 5.9

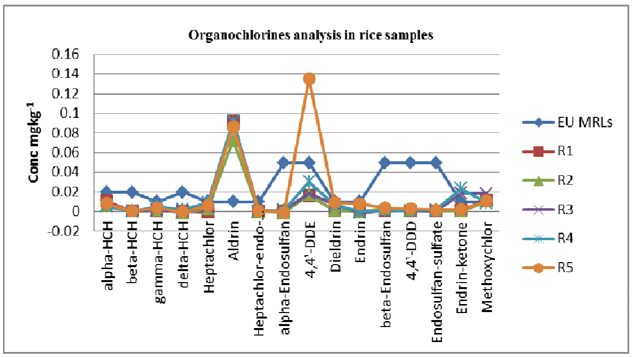
Table.2. (b) Average recovery % and RSD% from soil samples spiked at three levels (n=5)

No	Pesticide's name	Average recovery % ± RSD% for soil		
		0.05 mg kg^{-1}	0.25 mg kg^{-1}	0.5 mg kg^{-1}
1	alpha-HCH	111 ± 11.1	80 ± 18.2	98 ± 9.6
2	beta-HCH	66 ± 4.6	92 ± 10.6	106 ± 11.0
3	gamma-HCH	55 ± 13.7	77 ± 5.2	96 ± 8.1
4	delta-HCH	105 ± 18.0	79 ± 16.0	97 ± 8.9
5	Heptachlor	75 ± 9.6	94 ± 4.1	83 ± 1.7
6	Aldrin	91 ± 10.6	81 ± 2.7	91 ± 3.6
7	Heptachlor-endo-epoxide	118 ± 10.4	111 ± 4.9	114 ± 3.2
8	alpha-Endosulfan	61 ± 15.5	72 ± 4.1	101 ± 4.8
9	4,4'-DDE	119 ± 5.7	118 ± 5.5	84 ± 3.9
10	Dieldrin	110 ± 17.5	79 ± 4.0	110 ± 4.1
11	Endrin	98 ± 8.5	95 ± 3.0	104 ± 3.5
12	beta-Endosulfan	82 ± 18.9	104 ± 1.6	105 ± 1.9
13	4,4'-DDD	82 ± 17.4	99 ± 1.7	102 ± 3.8
14	Endosulfan-sulfate	54 ± 18.0	57 ± 3.9	58 ± 2.3
15	Endrin-ketone	56 ± 4.0	60 ± 5.0	63 ± 2.9
16	Methoxychlor	78 ± 16.7	98 ± 2.2	107 ± 7.4

3.3. Method application

The QuEChERS method was also applied to the determination of pesticides in 30 samples of soil and 30 samples of rice from GAP crops of northeast region of Thailand. The results showed, for the rice samples most of the analytes were ≤ MRLs (maximum residues limits of EU) except aldrin for all samples and 4,4'DDE for some samples (Fig.2). For the soil samples, all of the analytes were not detected for all samples except heptachlor but the concentrations were less than those of the MRLs for all soil samples (Table 3).

(a)



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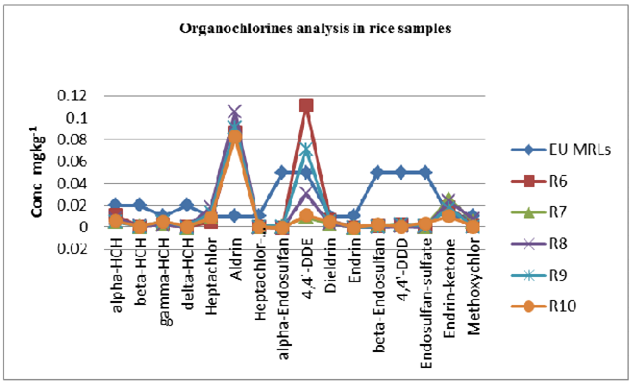

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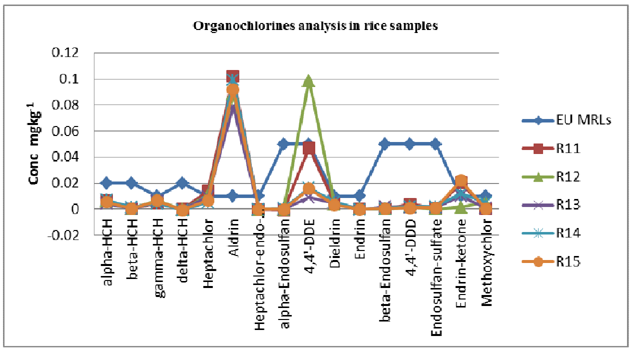

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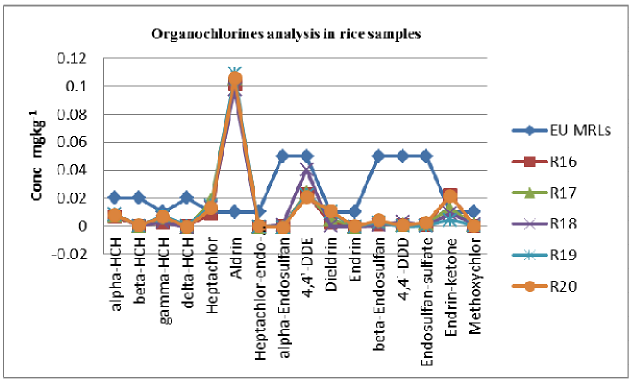

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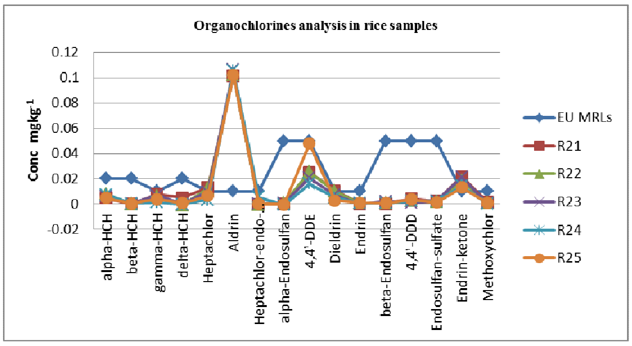

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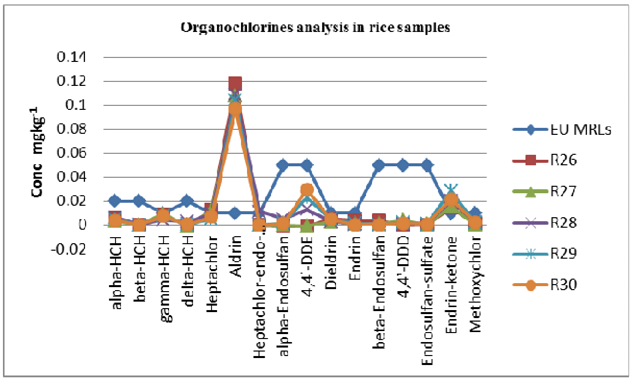

Fig.2. Determination of organochlorine residues in rice samples (a) rice samples R1 to R5 (b) rice samples R6 to R10 (c) rice samples R11 to R15 (d) rice samples R15 to R20 (e) rice samples R21 to R25 (f) rice samples R25 to R30.

4. CONCLUSION

As shown above, the establishment of GAP standard is important to significantly promote and encourage the quality and safety development of the rice production in order to be accepted for both domestic and international trade. GAP concepts in Thailand rice about the efficient production of rice with high yield and safe from toxic chemical residues. Improper use of pesticides can contaminate the environment or leave potentially harmful residues on the crop. High levels of pesticide residues on crops can control by GAP implementation. Appropriately use of pesticide, such as follow the pre-harvest interval (PHI) and the pre-grazing interval (PGI) requirements on the label, keep record of pesticide application are some important points of GAP implementation. High levels of pesticide residues on crops may be a hazard to humans who eat the produce. Thus, pesticide determination is playing an important role in food safety and international trade.

The QuEChERS extraction method used in our study minimized the time, labour consumption and cost of the sample preparation. In the rice samples we detected aldrin residues and also in the soil samples heptachlor residues were be detected. Both of aldrin and heptachlor are POPs compounds. POPs are characterized by their lipophilicity, persistence and semi-volatility. The semi-volatility of these substances facilitates their long range transport to and accumulation, far removed from any source of use. Aldrin residues bind strongly to soil particles and is very resistant to leaching into groundwater. Even though, they can loss from the soil by volatilization. Conversely, the heptachlor has a half-life of approximately 1.3 to 4.2 days in air, 0.03 to 0.11 years in water and 0.11 to 0.34 years in soil. Some study described that heptachlor residues could be found in soil 14 years after its initial application [13]. In our study we detected aldrin and heptachlor hence they are POPs compounds.

This paper showed QuEChERS method was suitable for organo-chlorines pesticide residues analysis for rice and soil in routine work.

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APPENDIX

Table.3. Determination of organochlorine residues in soil samples (a) soil samples S1 to S10 (b) soil samples S11 to S20 (c) soil samples S21 to S30

(a)

No.	OC	MRLs	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
1	alpha-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	beta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3	gamma-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4	delta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5	Heptachlor	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
6	Aldrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7	Heptachlor-endo-epoxide	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8	alpha-Endosulfan	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
9	4,4'-DDE		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	Dieldrin	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
11	Endrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12	beta-Endosulfan	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
13	4,4'-DDD		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
14	Endosulfan-sulfate	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
15	Endrin-ketone		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
16	Methoxychlor	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

(b)

No.	OC	MRLs	S11	S12	S13	S14	S15	S16	S17	S18	S19	S20
1	alpha-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	beta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3	gamma-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4	delta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5	Heptachlor	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
6	Aldrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7	Heptachlor-endo-epoxide	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8	alpha-Endosulfan	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
9	4,4'-DDE		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	Dieldrin	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
11	Endrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12	beta-Endosulfan	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
13	4,4'-DDD		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
14	Endosulfan-sulfate	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
15	Endrin-ketone		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
16	Methoxychlor	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

(c)

No.	OC	MRLs	S21	S22	S23	S24	S25	S26	S27	S28	S29	S30
1	alpha-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2	beta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3	gamma-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4	delta-HCH		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5	Heptachlor	0.03	0.01	0.01	0.00	0.01	0.01	0.01	0.01	0.00	0.01	0.01
6	Aldrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
7	Heptachlor-endo-epoxide	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8	alpha-Endosulfan	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
9	4,4'-DDE		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
10	Dieldrin	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
11	Endrin	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12	beta-Endosulfan	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
13	4,4'-DDD		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
14	Endosulfan-sulfate	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
15	Endrin-ketone		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
16	Methoxychlor	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00