CHAPTER 5
CONCLUSIONS

5.1 Qualitative and quantitative analysis of synthetic pyrethroids

Retention times of individual synthetic pyrethroids in standard mixture are showing multiple peaks. The calibration curves were linear with correlation coefficients (R^2) between area ratio of sample and internal standard for all pesticides were 0.9835 to 0.9972. The linearity ranged differs by their compounds from 5 – 500 ng/ml.

5.2 Quality control of synthetic pyrethroids analysis

The limits of detection and the limits of quantification of individual synthetic pyrethroid are varied depending on its component. Values of LOD differ from 0.001 to 0.005 mg/kg. In addition, LOQ ranged from 0.005 to 0.01 mg/kg These results show the method has suitable range with lower value when compare to minimum residue limits (MRLs) recommended by Thai agriculture commodity and food standard (2006) and Codex alimentarius pesticide residues in food.

Percent recoveries of the six pyrethroids from fortified crop at three concentrations are in ranged 96.8% to 109.3%, from 86.5% to 96.9 % and 83.8% to 98.4% for spiked at low, medium and high level, respectively. The result of this study showed that the values of recoveries percentage in all synthetic pyrethroids were in range of acceptable criteria recommended by CODEX (70-110%).

Variations of intra-batch and inter-batch of pooled vegetable and fruit samples are in ranged from 87.9 to 102.1 and CV percentage ranged from 1.9 to 7.4. The recoveries of inter-batch ranged from 81.0 to 113.9 and CV percentage ranged from 5.8 to 15.7. Both of them were acceptable for criteria determine at less than 21%.

5.3 Qualitative and quantitative analysis of 3-PBA

3 phenoxybenzoic acid, a common metabolite of pyrethroid insecticides. The calibration curves were linear with correlation coefficients (R^2) between area ratio of sample and internal standard for 3 PBA at 0.9939.

Recoveries of 3-PBA at 91.4 and CV percentage is 0.13. For limit of detection at 0.8 µg/l, and limit of quantification of 3-PBA is 1 µg/l. These data were acceptable when compare to the reference value of Germany which determines urinary concentration of metabolites of pyrethroid insecticides at not exceed 2 ng/ml (Human Biomonitoring Commission of the German Federal Environment Agency 2005) .

Variations of intra-batch of pooled urine samples is 0.18 for 3-PBA. For inter-batch, CV percentage is 0.12 for 3-PBA. Both of them were in the acceptable criteria, which were less than 21%

5.4 Development method for detecting synthetic pyrethroid residues in vegetables and fruits using GC-ECD

In this study, fruits and vegetables extraction efficiency for synthetic pyrethroid residues of four solvent extractions consist of ethyl acetate, dichloromethane, acetonitrile, and acetone. Dichloromethane obtained highest

recoveries percentage in most of synthetic pyrethroid residues except in deltamethrin. Recoveries percentage of dichloromethane ranged from 28% in deltamethrin to 90% in cypermethrin and fenvalerate.

The effectiveness of various solid-phase extraction cartridges to clean up sample which comprise SAX/PSA, activated carbon, and octadecyl (C_{18}) were determined. From initial study, due to inability to remove pigment from sample of octadecyl (C_{18}), the researcher decided to stop the study on this solid-phase extraction. Further study, activated carbon has better ability to remove pigment from sample than SAX/PSA. Similar to recoveries percentage, activated carbon have higher value than SAX/PSA in all synthetic pyrethroid pesticides. Recoveries percentage of activated carbon range from 63.3% to 101.0 and standard deviation range from 1.14 to 20.41.

Six synthetic pyrethroid insecticides are various matrices vegetable and fruit samples. Four vegetable and fruit samples consist of cabbage, kale, longan, and tangerine were determined. Recoveries percentages of various matrices are vegetable and fruit samples range from 69 to 131 and standard deviation range from 1.66 to 33.1.

In rugged test, fruits and vegetables extraction efficiency for synthetic pyrethroid residues from three scientists obtained the recoveries of synthetic pyrethroid in vegetable and fruit samples are higher than 80%.

5.5 Development method for detecting a synthetic pyrethroid metabolite in urine using GC-ECD

In this study, Ethyl acetate obtained highest recoveries percentage in most of synthetic pyrethroid metabolite in urine. Recoveries percentage of ethyl acetate is 91.4.

The effectiveness of various solid-phase extraction cartridges to clean up sample which comprise SAX/PSA, activated carbon, and octadecyl (C_{18}) were determined. From initial study, due to inability to remove pigment from sample of SAX/PSA and activated carbon, the researcher decided to stop the study on these cartridges. Further study, C_{18} has better ability to remove pigment and noticeable chromatographic interferences from sample the recoveries percentage at 78.9.

5.6 Assess the exposure of synthetic pyrethroid pesticides among school children

5.6.1 Levels of synthetic pyrethroids in vegetables and fruits

The mean level of pyrethroid pesticides in vegetable and fruit samples in June 2009. Cyfluthrin has the highest positive sample at 26 samples of vegetable. Cypermethrin has the highest positive sample at 20 samples of fruit. The result also shows the highest mean value of cypermethrin in fruits at 1.062 mg/kg and 0.907 mg/kg in vegetable samples.

The mean level of pyrethroid pesticides in vegetable and fruit samples in March 2010 present in table 4.15. Cypermethrin has the highest positive sample at 25 samples of vegetable and 21 samples of fruit. The result also shows the highest mean

value of cypermethrin in fruits at 0.655 mg/kg., while deltamethrin shows the highest mean value in vegetables at 1.382 mg/kg.

According to the Thai agricultural commodity and food standard (2006), the mean values of pesticide in most samples were generally exceed the MRLs, except cypermethrin in cabbage, fenvalerate in mango and deltamethrin in yard long bean. The highest percentage was found in cypermethrin of sugar pea while deltamethrin of yard long bean showed the lowest percentage.

Furthermore, the percentage of sample above MRLs of different international standard, samples with permethrin were not detected when compare to the Codex MRLs. Percentage of the samples above MRLs of Codex and Thailand have similar trend in all pesticides with range from 4.6 to 19.3 %. In addition, the European standards which have lower concentration limit shows higher percentage of sample exceed the MRLs in range of 22 to 55 %.

5.6.2 Levels of 3-PBA in urine

In this study, the concentration of 3-PBA in urine metabolite samples data for our study in 4 subdistricts of the studied areas, Fang district, Chiang Mai province.

In June 2009, 3-PBA was detected most frequently in Mae Ngon is 81.0%. The result also shows the highest geometric mean value of 3 PBA unadjusted in wiang at 19.78 μg/l

In March 2010, 3-PBA was detected most frequently in Mae Ngon is 88.5%. The result also shows the highest geometric mean value of 3 PBA unadjusted in wiang at 5.23 μg/l.