Development method for determination of transfluthrin in insecticide products by GC

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INTRODUCTION

Mosquitoes are vector of many diseases such as dengue fever, malaria etc. Self-protection against mosquito bites can be done in several ways including sleeping in nets, wearing clothes to cover the body and use insecticide or repellent products which has various forms such as spray , liquid or sheets for apply to the skin etc. Liquid insecticides and mosquito coil which are popular products in Thailand. The synthetic chemicals that used as active substances are pyrethroids such as d-allethrin, s-bioallethrin, metofluthrin, prallethrin and transfluthrin etc., which are classified as hazardous substances used in public health type 3 , according to the Hazardous Substance Act BE 2535. These products must be approved and registered with Thai FDA before production and import. The labeling must be conducted in according to the law. The concentration of active substances are corresponding to the label specified with the acceptance criteria according to the announcement of the Ministry of Public Health. At present , liquid insecticides and mosquito coil products which were registered with Thai FDA are found the using of transfluthrin as active substances in many products. Therefore , the method for determination of transfluthrin in liquid insecticides and mosquito coil products by GC has been developed by the Bureau of Cosmetics and Hazardous substances , Department of medical sciences. The developed analysis method can be applied in the analysis of transfluthrin in liquid insecticides and mosquito coil products by prepared the document of standard procedure to provide analysis services and surveillance of product quality as well as safety protection for consumers.

MATERIALS AND METHOD

Reference standard : Transfluthrin from ChemService , purity = 99.5 %

Working standard solution : 0.05 , 0.1 , 0.2 , 0.4 mg/mL in acetone

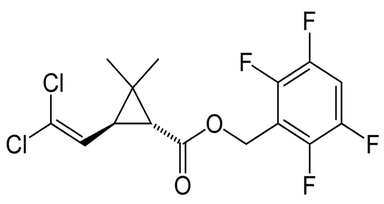
Sample : Mosquito coil products contain of transfluthrin 0.05 %w/w,
Liquid insecticide (electric evaporizer insecticides) contain of transfluthrin 0.88 %w/w

sample preparation

- **Mosquito coil** : Blend sample with blender , weigh blended sample in cellulose thimble to contain transfluthrin 10-20 mg and extract with acetone for 4 hrs by soxhlet extraction. Evaporate, dissolve and make up to volume in 25 mL volumetric flask with acetone
- **Liquid insecticide** : Weigh sample to contain transfluthrin 10-20 mg. Dissolve and make up to volume in 25 mL volumetric flask with acetone.


Gas chromatography (GC) with FID detector ; Agilent 7890 A

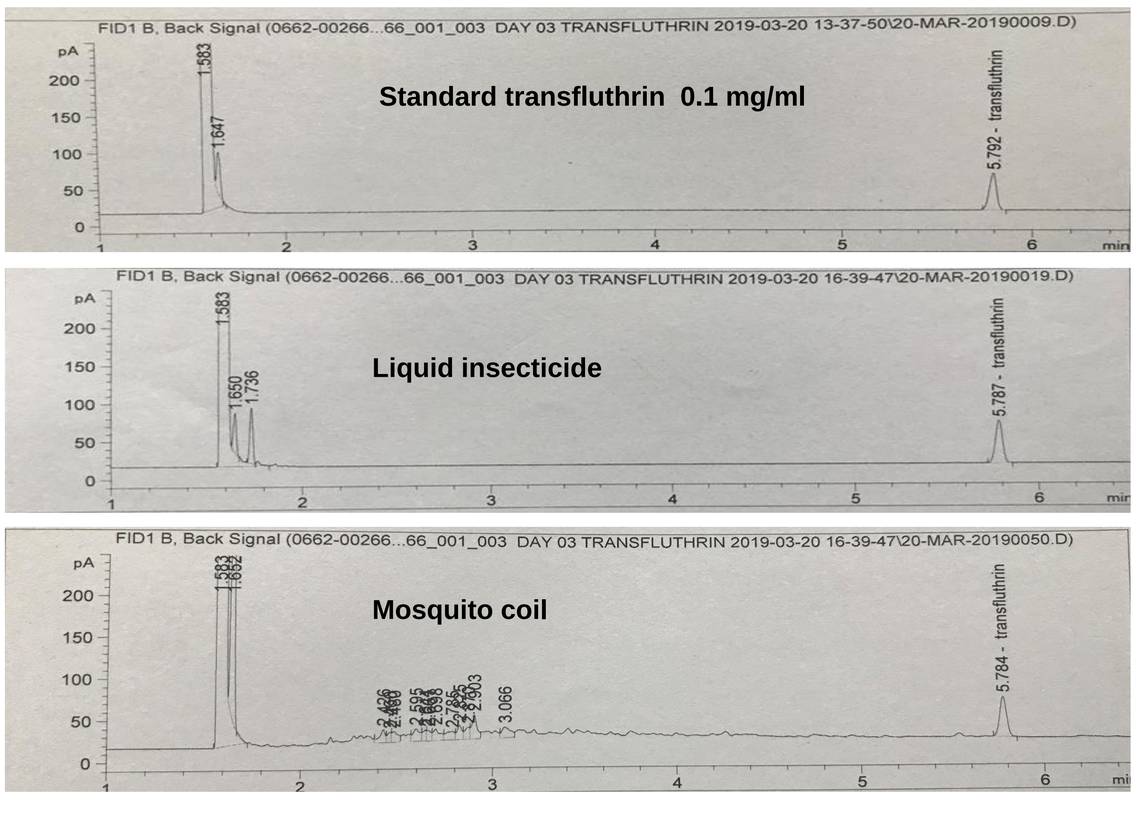
Capillary Column : HP -5 , 30 m × 0.32 (i.d.) mm, film thickness : 0.25 µm



transfluthrin

	GC condition
Column	**190°C**
Injection port	**290°C**
Detector	**300°C**
Injector	**split injection**
Split flow	**approximately50 mL/min**
Injection volume	**1 µL**
Detector	**flame ionization**
Carrier gas	**Helium**
Velocity	**35 cm / sec (constant flow 16.7 psi)**
Flow rate	**Nitrogen 45 mL/min**
	hydrogen 40 mL/min
	Air 400 mL/min

GC chromatogram



METHOD VALIDATION

Parameter	Criteria	Result	
		Mosquito coil	Liquid insecticide
1. System suitability			
- Theoretical plate	> 50,000	105149	101575
- Tailing factor	0.8 - 2.0	1.18	1.12
- K prime	> 2.0	3.02	3.04
2. Linearity and Range		0.05 – 0.4 mg/mL	0.05 – 0.4 mg/mL
- working range	r ≥ 0.995	r = 1.000	r = 0.9999
- linearity		r = 1.000	r = 1.000
3. Precision			
3.1 Repeatability (n=7)	%RSD ≤ 5	%RSD = 3.17	%RSD = 1.05
3.2 Intermediate precision			
- Between days (n=5)	*p-value* ≥ 0.05	*p-value* = 0.22	*p-value* = 0.37
- Between analysts (n=3)	*p-value* ≥ 0.05	*p-value* = 0.44	*p-value* = 0.48
4. Accuracy (recovery)	90 - 110	92.64 – 101.19	97.79 – 105.68
5. Limit of Detection (LOD)	-	0.005 %w/w	0.005 %w/w
6. Limit of Quantitation (LOQ)	-	0.01 %w/w	0.01 %w/w

RESULTS

The method for determination of transfluthrin in liquid insecticides and mosquito coil products by GC has been developed and carried out on HP-5 column(30 m x 0.32 mm x 0.25 µm) with temperature program of oven , injection port and detector are 190°C , 290°C and 300°C , respectively. The chromatogram showed a peak of transfluthrin at retention time about 5.7 minutes. Method validation has also been performed. System linearity was found in concentration range of 0.05 - 0.4 mg/mL with the correlation coefficient (r) of 0.99998. The precision of analysis including repeatability of replicate analysis of transfluthrin in liquid insecticides and mosquito coil products provided the relative standard deviation (%RSD) are 1.05 and 3.17 , respectively. There is no significant different of the test results from different days and different analysts (p-value > 0.05). Percent recovery of transfluthrin in both of 2 products were found in range of 90-110. Limit of quantification (LOQ) was 0.01 %w/w and limit of detection (LOD) was 0.005 %w/w.

CONCLUSIONS

❖The development method for determination of transfluthrin in liquid insecticides and mosquito coil products by GC, provided the validation data within the specification of the analytical method and showed the validity of the method.

❖The developed method can be applied in the analysis of transfluthrin in liquid insecticides and mosquito coil products by prepared the document of standard procedure to provide analysis services and surveillance of product quality as well as safety protection for consumers.

❖The Bureau of Cosmetics and Hazardous substances intend to expand the scope of analysis by validate the method in aerosol insecticide to cover all products of transfluthrin which sold in Thailand.

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