



Method Development & Validation for determination of icaridin in insect repellent products by Gas chromatography

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Introduction

Icaridin functions as a potent insect repellent by effectively inhibiting the olfactory system of insects. It is characterized by low toxicity to humans, causing minimal skin and eye irritation, and is essentially odorless. It provides long-lasting insect protection. Under the Hazardous Substance Act, BE 2535 (1992), Icaridin is classified as Type 2 Hazardous Substances for public health. Although many Icaridin in insect repellent products are currently imported and distributed in Thailand, as of 2022, the Thai Food and Drug Administration (FDA) has not mandated quantitative analysis for product registration, primarily due to the absence of standardized analytical methods within the country. Therefore, this study aimed to develop and validate a method for the quantification of icaridin in insect repellents using Gas Chromatography (GC). This research is crucial for establishing quality control protocols, thereby ensuring product efficacy and safeguarding consumer health.

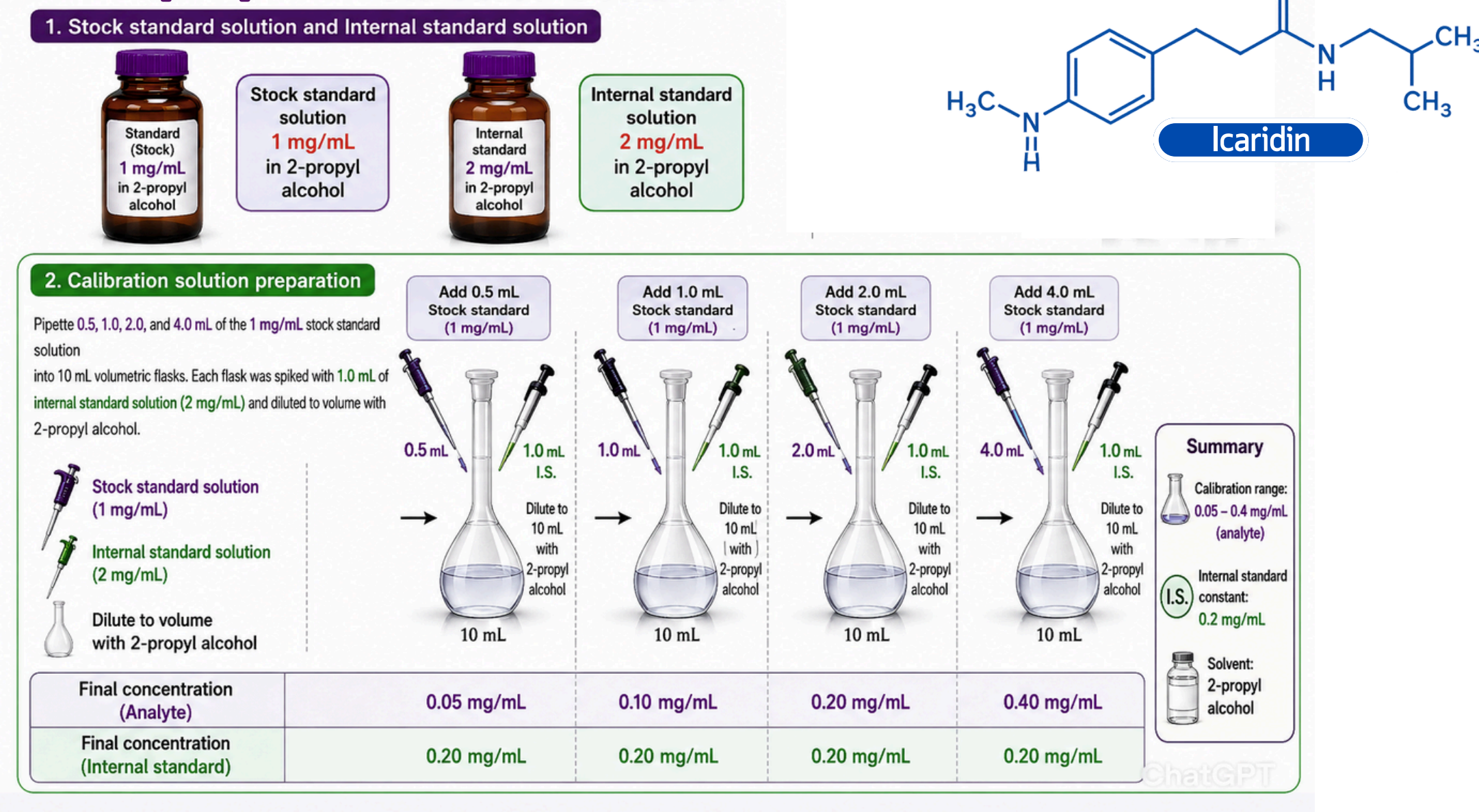


Materials and Method

Reference standard

- Icaridin from ChemService, purity 97.4 %
- Dimethyl phthalate (Internal standard) from ChemService, purity 99.6 %

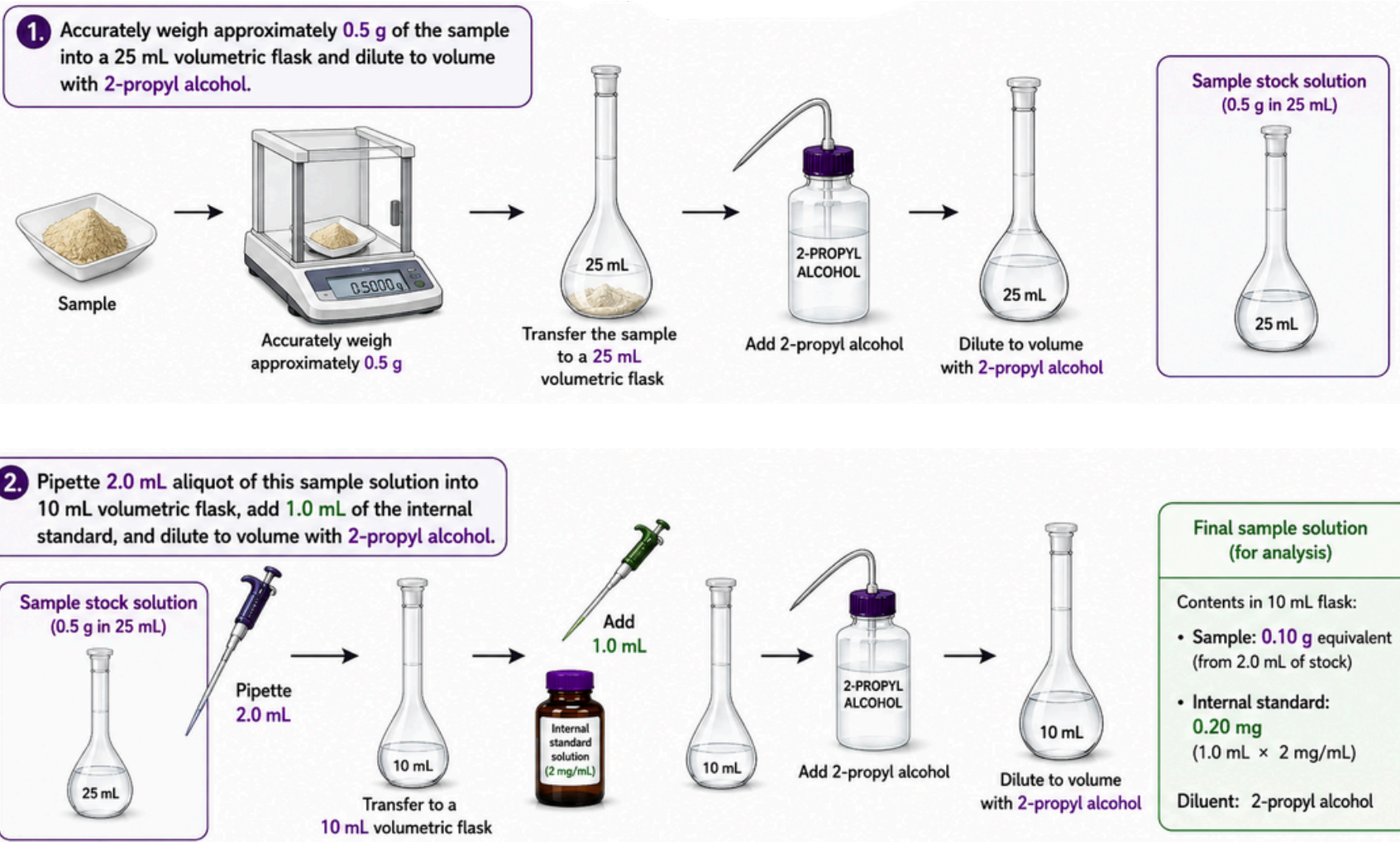
Standard solution preparation



Sample

- Liquid insect repellent contain of icaridin 7.76 %w/w
- Sample blank : Liquid repellent insect repellent without icaridin

Sample preparation



Analysis of samples

In 2023, a market survey was conducted to monitor the quality of icaridin insect repellents products in Thailand. Based on FDA registration data from 2011–2022, which comprised 53 products from 25 manufacturers and importers, 53 commercial samples were collected and analyzed for active ingredient content.

The accuracy and precision were monitored using strict quality control criteria. Duplicate measurements were performed for all samples with RPD < 3%, while spiked samples were analyzed every 10 samples or per analytical batch with recovery 90–110%. If any quality control result failed to meet these criteria, the entire batch or affected samples were re-analyzed.

Total samples analyzed

53

Total samples analyzed

Compliant samples

37 (70%)

Out of specification

16 (30%)

% of non-compliance

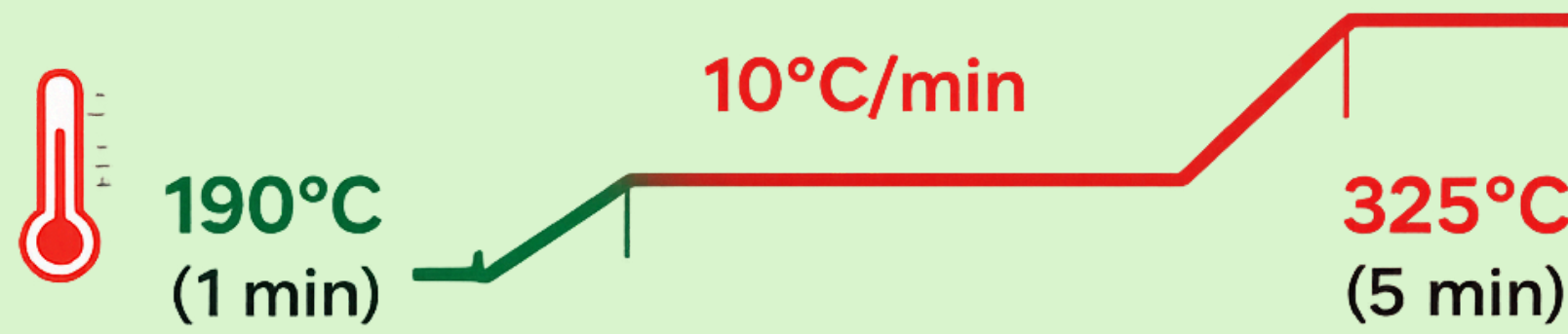
30%



GC-FID Conditions



Instrument : Agilent 7890A GC with FID
Column : DB-1
: 30 m x 0.25 mm ID x 0.25 μ m film
Temperature Program :



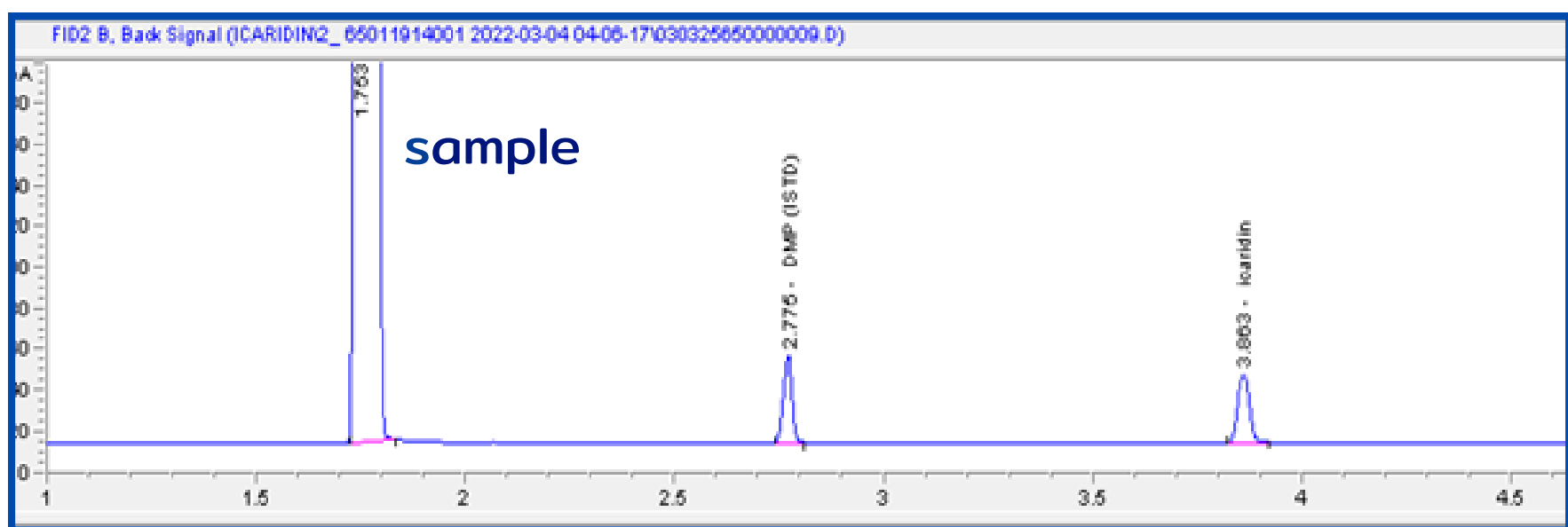
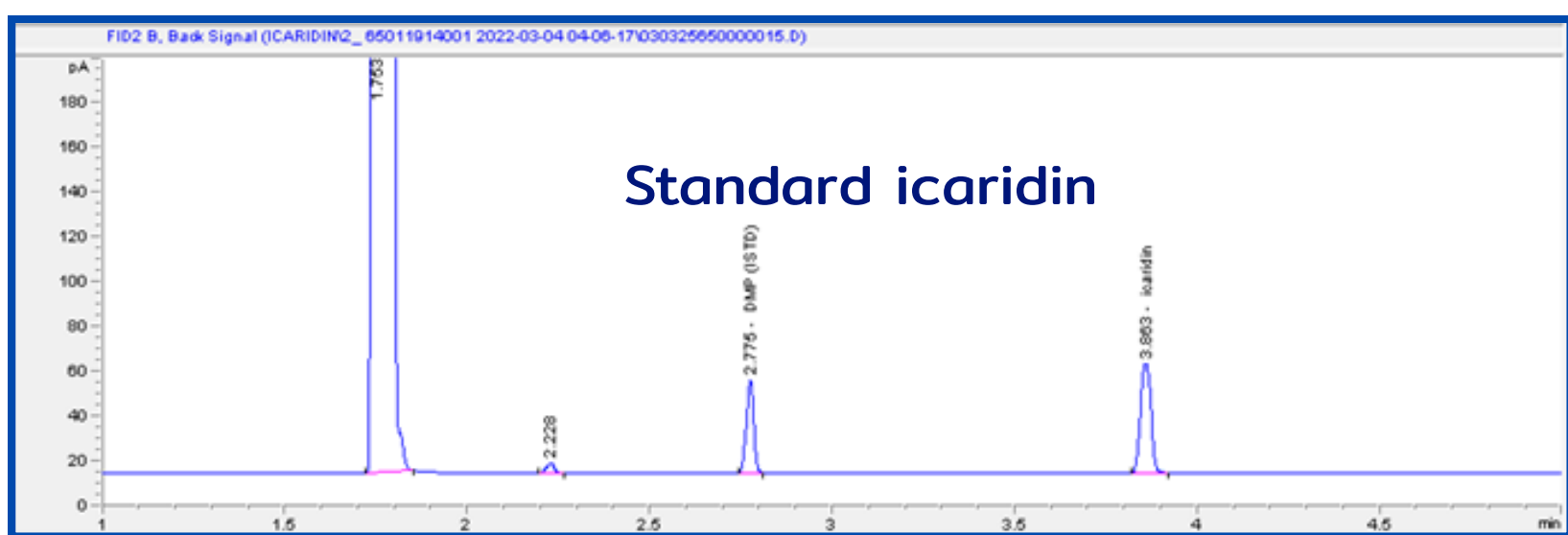
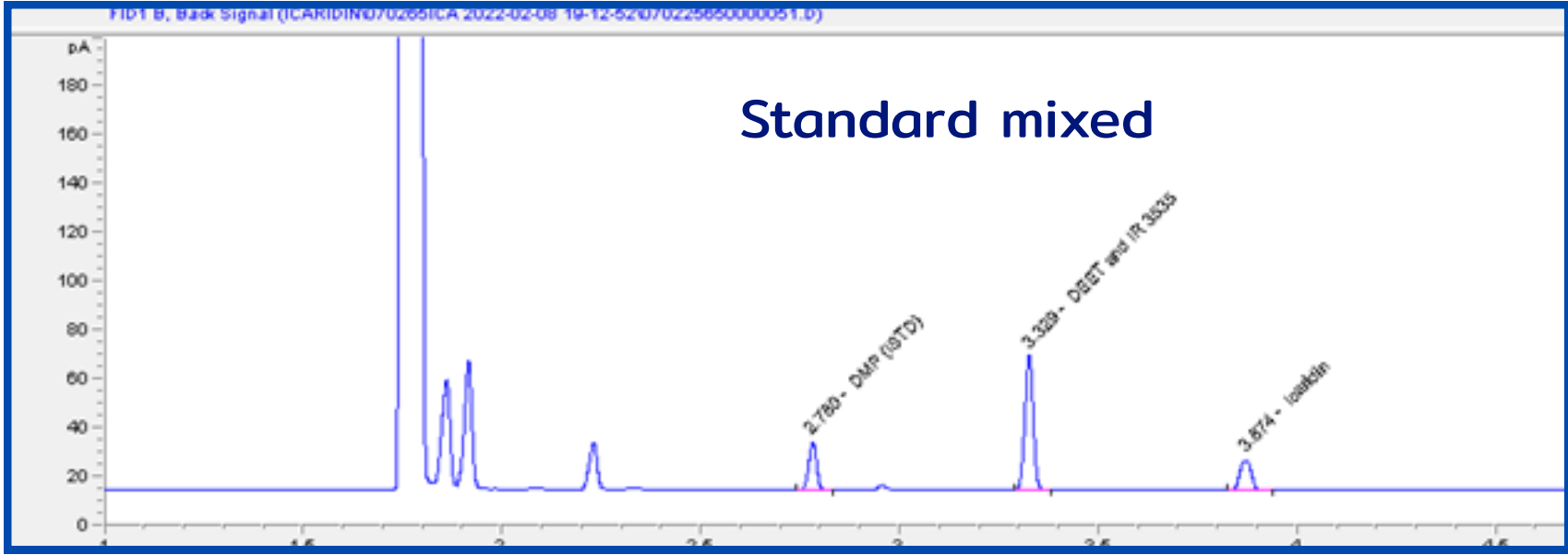
Injection Port : 240°C
Detector : 330°C
Injection : Split Injection
Split Flow : ~40 mL/min
Injection Volume : 1.0 μ L
Carrier Gass : Helium



Validation Results

- Specificity
Dimethyl phthalate (ISTD), icaridin, DEET, and IR3535 were successfully resolved under the optimized GC conditions. Quantitative analysis of the insect repellent lotion samples showed well-separated peaks with no matrix interference.

Gas Chromatography with Flame Ionization Detector (GC-FID)



Parameter	Criteria	Result
1. System suitability		
• Theoretical plate	50,000	76,544
• Tailing factor	0.8 - 1.2	0.931
• K prime	> 2.0	22.61
2. Linearity and Range		
• Working range	-	0.05 - 0.4 mg/mL
• Linearity	r ≥ 0.995	r = 1.000
3. Precision		
3.1 Repeatability (n=7)	%RSD ≤ 3	%RSD = 2.08
• Intermediate precision		
3.2 Between days (n=5)	P-value ≥ 0.05	P-value = 0.19
• Between analysts (n=3)	P-value ≥ 0.05	P-value = 0.16
4. Accuracy (recovery)	90 - 110	99.5 - 102.4
5. Limit of detection (LOD)	-	0.04% w/w



Conclusion

- The development method for determination of icaridin in liquid insect repellent 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 icaridin in liquid insect repellent products by prepared the document of standard procedure to provide analysis services and surveillance of product quality as well as safety protection for consumers. In 2025, the FDA designated icaridin as a hazardous substance requiring the submission of active ingredient assay results to support product registration applications.
- The Bureau of Cosmetics and Hazardous substances intend to expand the scope of analysis by validate the method in aerosol insect repellent to cover all products of icaridin which sold in Thailand.



References

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- Eurachem Guide. The Fitness for purpose of analytical methods: a laboratory guide to method validation and related topics. 2nded. [online]. 2014; [cited 2022 Jan 1]; [70 screens].