

## Determination of Permethrin in Pharmaceutical Product by Gas Chromatography

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**Abstract:** Gas chromatographic analysis was applied for the quantitative determination of permethrin present in some pharmaceutical products. The calibration curve of permethrin was linear with determination coefficient ( $R^2$ ) = 0.99998; over a concentration range of 1200 – 2800 mg/l; with a retention time of 31.599, 32.578 minutes for Cis-permethrin, Trans-permethrin; respectively. The validated GC method was successfully applied for the quantitative determination of the permethrin, either as a separate raw material or within the pharmaceutical product.

**Key words:** GC, Permethrin, Method Validation.

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### I. Introduction

Pyrethrum flower is the dried flower heads of *Chrysanthemum cinerariaefolium* containing not less than 1% of pyrethrins of which not less than one-half consists of pyrethrin I. It has a faint but characteristic odour (1). Pyrethrum, pyrethrins, and pyrethroids are also used as topical ectoparasiticides in veterinary practice and as agricultural, horticultural, and household insecticides (2). Permethrin is a non-systemic pyrethroid insecticide with some repellent effects. It is a viscous liquid at room temperature; it does not dissociate in water and has extremely low water solubility and volatility. It is stable to hydrolysis at pH 4–7. Technical grade permethrin is composed of 4 stereoisomers, due to the chirality at two carbon atoms in the cyclopropane ring, leading to 2 cis and 2 trans isomers. Permethrin technical grade cis:trans ratio is either 25:75 or 40:60. (6)

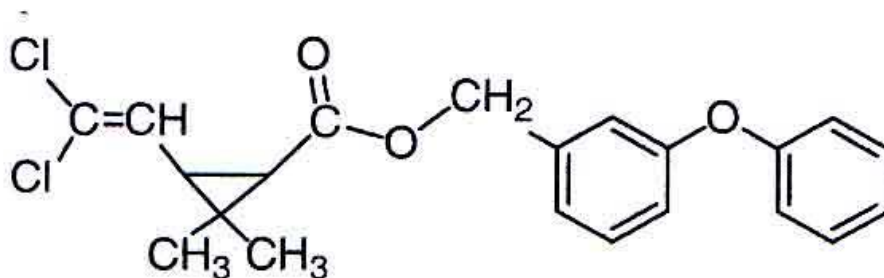


Fig (1): Chemical structure of permethrin (6)

Permethrin is commercially available in the following dosage forms: Medicated Shampoo, topical Cream, topical gel, topical Lotion, topical Spray. Permethrin is available in the following strengths: Topical lotion and cream 5 % for the treatment of scabies, topical lotion 1 % and shampoo 1 % for head lice. Also topical gel 2 % is present as well for scabies. Topical spray 0.25 and 0.5 % is also available as insect repellent, applied on clothes (5, 6). Permethrin is active against mosquitoes and is widely used for the impregnation of bednets and curtains in the control of malaria. It is also active against blackflies in the adult and larval stages and is used for the larvicidal treatment of rivers in the control of onchocerciasis. It is also active against tsetse flies. Permethrin is suitable for aircraft disinfection (4). Nowadays, due to its safety use, growing interest on its use is vastly.

### II. Materials & Methods

#### 1. Equipment

– **Gas Chromatography:** Shimadzu GC, Model 2010, connected with FID detector auto injector, Kyoto, Japan.

**Column:** DB-5 (0.25mm x 30 m), 0.25  $\mu$ m, Agilent, USA.

- **Electronic Balance:** Model AUY220, Shimadzu Instrument, Kyoto, Japan
- **Ultrasonic bath:** NSXX Sonics Model NS-A-12-7H, Germany.

**2. Chemical Reagents:**

All chemical reagents and standards used were GC grade. Pure standard of permethrin was obtained from Hetero Drugs, Hyderabad. Hexane  $\geq 97\%$  was HPLC and GC grade from Sigma Aldrich.

**III. Experimental**

**3.1 Chromatographic conditions:**

The GC analysis was carried out using Shimadzu GC, Model 2010, connected with FID detector auto injector, under the following condition:

**Column:** DB-5 (0.25mm x 30 m), 0.25  $\mu$ mf, Agilent, USA.

**Injection volume:** 1  $\mu$ l.

**Flow rate:** 13.5 ml/min.

**Injection Temp.:** 300°C.

**Injection Mode:** Splitless

**Split ratio:** 10

**Carrier gas:** Helium.

**Oven Temperature Program:**

Rate (°C/min)	Temperature (°C)	Hold Time (min)
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7	277	2

**4.1. Method Validation:**

**4.1.1. Calibration curves:**

The stock standard solution of pure standard was prepared as follows:

100 mg of the compound was accurately weighed and placed into a 50 ml volumetric flask. n-Hexane was added and the solution was diluted to volume with the same solvent, then it was sonicated for 15 min.

Calibration curve was established on nine data points covering the concentration range of 1200– 2800  $\mu$ g/ml.

One microliter aliquots of each standard solution were used for GC analysis. Triplicate injections were made for each standard solution. The calibration curve was obtained by plotting the peak area of the permethrin at each level prepared versus the concentration of the sample.

**Table (1):** Statistical analysis for the calibration curve of the permethrin standards

Compound	Linearity range ( $\mu$ g/ml)	Slope, a	Intercept, b	R <sup>2</sup>
Permethrin	1200-2800	6,756.8578	-28,260.699	0.99998

S.D. values are given in parenthesis.

a for each curve the equation is  $y = ax + b$ , where y is the peak area, x is the concentration of the analyte ( $\mu$ g/ml), a is the slope, b is the intercept and R<sup>2</sup> is the determination coefficient.

**4.1.2. Recovery**

The accuracy of the method was evaluated with the recovery test. This involved the addition of known quantities of permethrin standards to known amounts of sample. The percentage recovery was determined by subtracting the values obtained for the control matrix preparation from those samples that were prepared with the added standards, divided by the amount added; then multiplied by 100.

Table (2): Results of the recovery test for the used essential oil & extract

Compound	Spiked amount (mg)	Recovery (%)	Mean (n = 5)	R.S.D. (%)
Permethrin	0.530	99.88-101.42	100.82	1.60

R.S.D. (%) = (standard deviation/mean)  $\times$  100.

**4.1.3. Limits of detection and quantification**

Limits of detection (LOD) were calculated according to the expression  $3.3\sigma/S$ , where  $\sigma$  is the standard deviation of the response and S is the slope of the calibration curve. Limits of quantification (LOQ) were established by using the expression  $10\sigma/S$ . LOD and LOQ were experimentally verified by injections of pure standard at the LOD and LOQ concentrations.

Table (3): Limit of detection (LOD) and limit of quantification (LOQ)

Compound	LOD ( $\mu\text{g/ml}$ )	LOQ ( $\mu\text{g/ml}$ )
Permethrin	33.33	100

**4.2. Standard Preparation:**

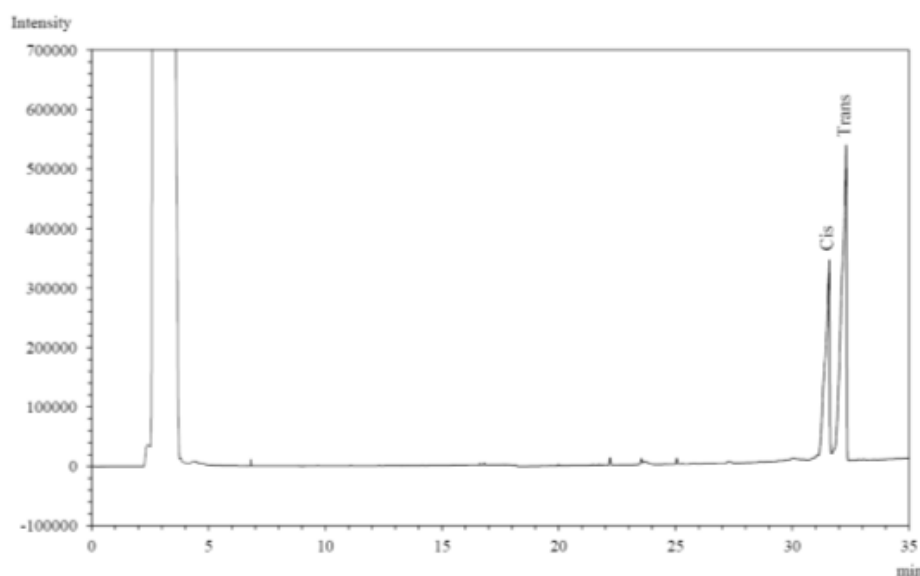
100 mg of the following standard was accurately weighed and transferred into 50 ml volumetric flask; then dissolved in hexane and sonicated for 15 minutes. The volume was completed to 50 ml with hexane.

- (Dilutions were done case related).

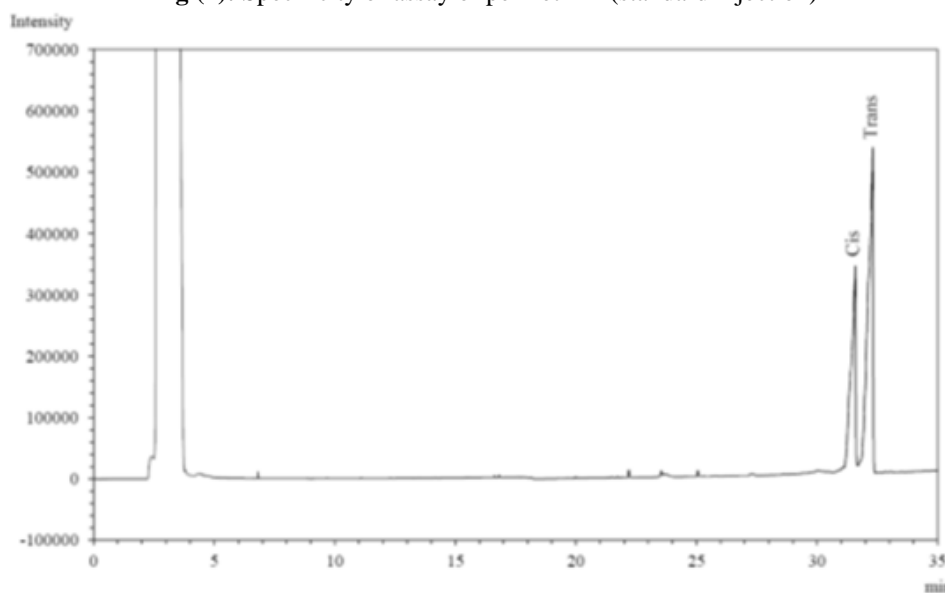
**IV. Results and Discussion**

The used GC method for the analysis of pharmaceutical product proved that it is an accurate, precise, fast and easy method for the quality control of complex pharmaceutical products containing mixture of cis and trans-permethrin. This fact is clear and can be deduced from the obtained result of calibration curve table (1); where the linearity was found to be (1200-2800  $\mu\text{g/ml}$ ), slope (6,756.8578), intercept (-28,260.699) and the determination coefficient (0.99998).

The percentage recoveries table (2) range form (99.88-101.42%). Limit of detection (LOD) and limit of quantification (LOQ) are shown in table (3); where we can value of LOD (33.33  $\mu\text{g/ml}$ ) and LOQ (100  $\mu\text{g/ml}$ ).



**Fig (2):** Specificity of assay of permethrin (standard injection)



**Fig (3):** Specificity of assay of permethrin (Test injection)

#### **IV. Conclusion**

The applied GC method is of great value for the quality control of the pharmaceutical products containing mixture of cis and trans-permethrin.

#### **References**

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