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A Simple and an Innovative Gas Chromatography Method to Quantify Isopentane in Cosmetic Products

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Abstract: *The innovative method for quantification of Isopentane in cosmetic products. This method was developed in gas chromatography by using capillary column and flame ionisation detection. This was an innovative method for specifically quantification of Isopentane. This method was validated as per ICH guidelines. This method was simple, specific, precise, linear, accurate, robust and ruggedness for analysis of Isopentane.*

Keyword: *Isopentane, Gas chromatography, Flame ionisation detetector, Cosmetic products.*

I. Introduction

Isopentane is an organic compound with a variety of uses, ranging from being an ingredient in cosmetics including shaving gel, body wash, and specific types of toothpaste. It is also called as methylbutane or 2-methylbutane[1] structure was shown in fig.1. It has constant quality it meets all the requirements of a propellant, so it is used in shaving gel[2], which foams the gel on application. This solvent is extremely flammable. Its boiling point is 27.9°C. It is a volatile liquid at room temperature, and flammable [3][4].

Based on this volatile nature of isopentane, it is difficult to quantify in cosmetic products. For analysis of isopentane in cosmetic products, few of developed methods are available by GC/GCMS [5][6][7][8], Head-space GC[9]. As per best of my knowledge there was no specific method for the quantification of isopentane. The aim of this method was to develop specific quantification method for isopentane in cosmetic products, which is simple, selective, reproducible and accurate method.

II. Materials and Methods

2.1. Apparatus

Gas Chromatography with FID and AOC-20i Auto injector (model: GC-2010 Plus, make: Shimadzu), Column-ZB-624 (Length-30m, Diam-0.32mm, Film-1.80µm, Make: phenomenex), Sonicator (model: Soltec, Make: Sonica), Electronic Weighing Balance (Model: ML204 Ia01, Make: Mettler Toledo), Volumetric flasks (100± 0.2mL, Make: Borosil).

2.2. Chemicals and Reagents

Isopentane (99+ %) (AR grade) (Make: Alfa Aesar), Toluene (HPLC-grade) (Make: Rankem).

III. Instrumentation

3.1. GC Conditions

Column	:	ZB-624
Length	:	30m
Inner diameter	:	0.32mm
Film	:	1.80µm
Initial temperature	:	35^0C
Hold time-1	:	6.0 minutes
Rate	:	13^0C per minute
Final temperature	:	150 ^{0}C
Hold time-2	:	0.0 minutes
Rate	:	25^0C per minute
Final temperature	:	240 ^{0}C
Hold time-3	:	10.0 minutes
Injector temperature	:	150^0C
Detector temperature	:	250^0C
Nitrogen gas flow rate	:	1.0 mL/minute
Split ratio	:	1:10
Detector	:	FID
Injection Volume	:	1 µL
Hydrogen gas flow	:	40 mL/minute
Air flow	:	400mL/minute
Run time	:	28.45minutes
Retention time	:	5.3 minutes

IV. Methods

4.1. Standard Solution Preparation

Weighed accurately 1g (±0.1g) of Isopentane standard in 100mL volumetric flask and dissolved in toluene and made up to mark with toluene.

4.2. **Sample Solution Preparation**

Weighed accurately 1g (±0.1g) of sample which required to analyse in 100mL volumetric flask and dissolved in toluene and made up to mark with toluene

4.3. Sample Analysis

Toluene which was used as a diluent was taken in to GC vial and inject for blank in GC. An Iso-pentane standard solution was taken in to a GC vial and injects for standard with replicate the injections up to six times; calculate the average area for standard. Sample solution was taken in to a GC vial and injects with replicate injection in GC, calculate the average area for sample. The chromatogram was shown in Fig.2.

V. Calculation

$$\% \text{ of Isopentane} = \frac{\text{(Average Sample Area)}}{\text{(Average Standard Area)}} \text{ X } \frac{\text{(Standard weight)}}{\text{(Sample weight)}} \text{ X (Standard purity)}$$

VI. Method Validation

6.1. Specificity and Selectivity

The specificity of the method was checked by injecting blank solution and sample solution. There was no interference from blank and excipients at the retention time of analyte peak.

6.2. Linearity

Linearity was checked by preparing five concentrations of the substance ranging from 50% to 150%. A concentration of 1.00% solution was proposed in the procedure as a 100%. Hence, the test substance was prepared at concentrations of 0.50%, 0.75%, 1.00%, 1.25% and 1.50% for determining linearity. Estimations were carried out as per the procedure mentioned. Observations were recorded and a linearity curve was prepared using regression analysis. The correlation coefficient was 0.9995.Linearity graph was shown in Fig.3

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6.3. Accuracy

The accuracy of the method was determined by adding known amount of isopentane corresponding the following concentration levels of 0.5%, 1.0%and 1.5% to target analyte concentration(1%) along with the excipients in triplicate. The accuracy was calculated by the percentage of analyte recovered by the assay method. The accuracy results were shown in table1.

6.4. Precision

The method precision, system precision and intermediate precision were calculated. The results were within the limits**.** Method precision results are shown in table2 .The system precision and intermediate precision results were shown in table3.

6.5. Robustness of the Method

Changing with the flow rate (±0.2mL/minute) and temperature program, no change in area of Isopentane and RSD was within limits. It indicates robustness of the method.

6.6. Ruggedness of the Method

Changing with different column (ZB-624 and DB-624) and different analyst the results were within limits. It indicates ruggedness of the method.

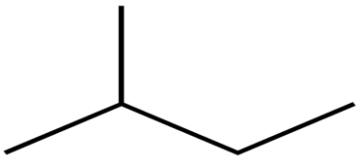
6.7. LOD and LOQ

Method limit of detection was 0.01ppm and limit of quantification was 0.04ppm.

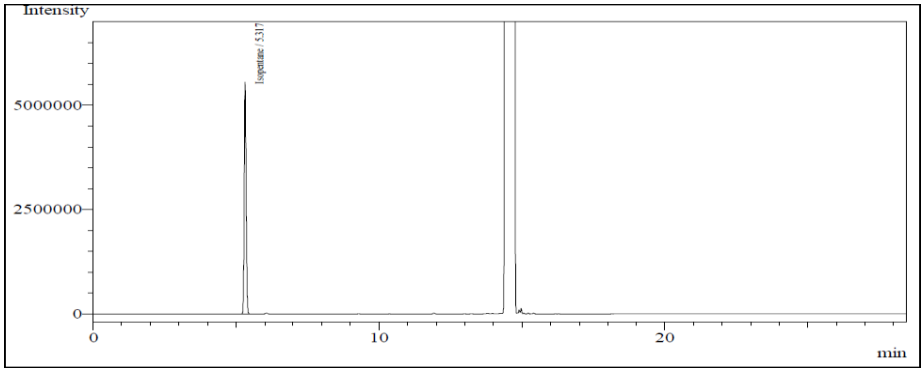
VII. Results and Discussions

The method was optimized based on using ZB-624 capillary column in gas chromatography. This method was validated as per ICH guidelines for specificity, linearity, accuracy, precision, robustness and ruggedness. The results were within limits.

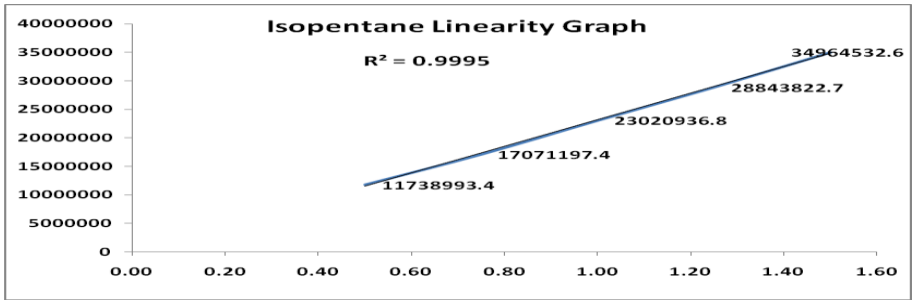
VIII. Figures and Tables



(Fig.1: Structure of Isopentane)



(**Fig.2:** GC Chromatogram of Isopentane)



(Fig.3: Linearity graph for Isopentane)

Table 1: Accuracy

Added amount (%)	**Recovery (%)**
0.5	96.40
1.0	96.01
1.5	95.21

Table 2: Method Precision

Preparation	**Assay (%)**
Preparation-1	102.41
Preparation-2	102.55
Preparation-3	103.18
Preparation-4	104.33
Preparation-5	108.30
Preparation-6	102.89
Average	103.94
Standard deviation	2.24
%RSD	2.16%

Table3: System precision and Intermediate precision

Precision	**%RSD**
System Precision	1.41%
Intermediate Precision	1.64%

IX. Conclusion

This developed and validated GC method was very simple, selective and reproducible method for the quantification of Isopentane in cosmetic products. Solution preparation was very simple on this method. This method was first time developed for specific quantification of Isopentane. This method was very good suitable for especially shaving gel, shaving foam samples. This method was having very low detection limits and low quantification limits. This method was specific, linear, precise, accurate and robust. So as per best of my knowledge this method is very good suitable for analysis of Isopentane in cosmetic products.

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