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A novel method for evaluation of 2-ethylhexanol in octyl stearate by using gas chromatography technique

N.K.Sarthchandra Prakash*[1], Jampala Balaji[2], Krishan Manaral[3].
[1,2]R&D Department, The Himalaya Drug Company, Makali, Bangalore, India-562162.
*** Corresponding author: N.K.Sarthchandra Prakash**
E-mail id: sarathchandra@himalayawellness.com

ABSTRACT
A new innovative method developed for determination of 2-Ethylhexanol in Octyl Stearate by gas chromatography using capillary column and flame ionization detection. This method may also be applicable for determination of 2-Ethylhexanol in cosmetic products. The determination of 2-Ethylhexanol was developed first time in Octyl Stearate. This method was validated as per ICH guidelines. This method was simple, specific, precise, linear, accurate, robust and ruggedness for analysis of 2-ethylhexanol in Octyl Stearate.

Keywords: 2-Ethylhexanol, Octyl Stearate, Gas chromatography, Flame ionisation detector, HP-5MS capillary column.

INTRODUCTION
Octyl Stearate is an ester of Stearic acid and 2-ethyl hexanol. Octyl Stearate is also named as 2-ethylhexyloctadecanoate; cetiol868; Octadecanoicacid, 2-ethylhexyl ester; stearicacid, 2-ethylhexylester; wickenol156; 2-Ethylhexyl Stearate; and Stearic acid, octyl ester [1]. Octyl Stearate is commonly colourless or slightly yellow oily liquid. Its boiling point was 431.862ºC at 760mmHg, so it is a very low volatile liquid. The structure of octyl stearate was shown in figure1.

It is used widely in the Cosmetic products as a base, a thickening agent, a pigment wetting agent, a dispersant, a solvent and an emollient in skin and eye make-up and in lipstick [2]. 2-Ethylhexyl Stearate gives skin a soft and smooth appearance. It has a dry-slip feel that is similar to silicone's texture. For this reason, it is often used as organic substitute for silicones in cosmetic products. They are often found in many skin care products as an ingredient used to adjust the consistency of the products. 2-Ethylhexanol is generally used in foundations and mineral make-up powders as a binder, as well as lipsticks and lotions or creams. They are often used as a solvent for sunscreens as they do not interfere with the active ingredients. When added into hair care products, 2-Ethylhexanol contribute to the shininess of hair and reduce frizziness after product application. They are also used in spray, lotion bar or balm to increase the silky feeling and reduce the feeling of products [3]. Octyl stearate is prepare from esterification of Stearic acid with 2-Ethylhexanol. So, 2-Ethylhexanol is a one of the major impurities in Octyl Stearate [4]. The structures of Stearic acid in figure2 and 2-Ethylhexanol in figure3 and are shown.

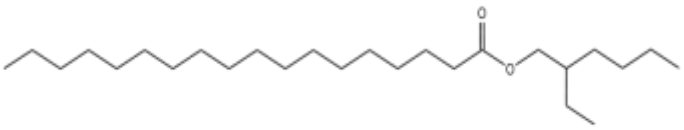

Figure1: Octyl stearate

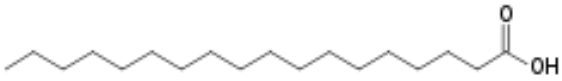

Figure2: Stearic acid

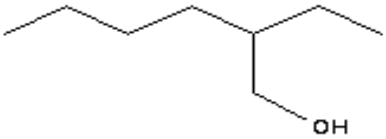

Figure 3: 2-Ethylhexanol

In the atmosphere, 2-Ethylhexanol occurs as a vapour [5]. It is a colourless liquid, soluble in organic solvents [6] but moderately soluble in water [7]. This compound is combustible and will react violently with oxidizing materials and strong acids [8].

The boiling point of the 2-Ethylhexanol is 180ºC to 186ºC. The International Programme on Chemical Safety (IPCS) has classified 2-Ethylhexanol as acutely irritant to the eyes, skin, and respiratory tract. Both the IPCS and the U.S. EPA have indicated a potential for neurotoxicity associated with acute 2-Ethylhexanol exposure [7]. Based on safety concerns, want to measure the 2-Ethylhexanol in Octyl Stearate. As per literature few of methods are found for determination of 2-Ethylhexanol by GC [9] and HPLC [10][11]. As per best of my knowledge there is no specific method for the determination of 2-Ethylhexanol. The difference between the boiling points of 2-Ethylhexanol and Octyl Stearate is very high. Due to this reason the development of the method is very challenging. The method was developed by GC-FID using capillary HP-5MS column. This method was simple, accurate, precession, linear and repeatable.

EXPERIMENTAL

Apparatus

Gas Chromatography with FID and AOC-20i Auto injector (model: GC-2010 Plus, make: Shimadzu), Column-HP-5MS (Length-30m, Diam-0.25mm, Film-0.25µ, Make: Agilent technologies), Sonicator (model: Soltec, Make: Sonica), Electronic Weighing Balance (Model: ML204 Ia01, Make: Mettler Toledo), Volumetric flasks (100± 0.2mL, Make: Borosil).

Chemicals and Reagents

2-Ethylhexanol (99.6+ %) (AR grade) (Make: Sigma Aldrich), Ethyl Acetate (Laboratory reagent grade) (Make: Rankem), Octyl Stearate (AR grade) (make: Subhash chemicals), Octyl Stearate (AR grade) (make: CRODA).

INSTRUMENTATION

GC Conditions

Column	HP-5MS
Length	30m
Inner diameter	0.25mm
Film	0.25µm
Initial temperature	50^0C
Hold time-1	3.0 minutes
Rate	12^0C per minute
Final temperature	200 ^{0}C
Hold time-2	3.0 minutes
Rate	25^0C per minute
Final temperature	320 ^{0}C
Hold time-3	12.0 minutes
Injector temperature	320^0C
Detector temperature	330^0C
Carrier gas	Nitrogen
Gas flow rate	1.2 ml/ minute
Split ratio	1:10
Detector	FID
Injection Volume	1 µl
Hydrogen gas flow	40 mL/minute
Air flow	400mL/minute
Run time	35.30minutes
Retention time	8.8 (±0.1) minutes

METHODS

Standard Stock Solution Preparation

Weighed accurately 1g (±0.1g) of 2-Ethylhexanol standard in 100mL volumetric flask and dissolved in Ethyl acetate and made up to mark with Ethyl acetate.

Standard Solution Preparation

1mL of 2-Ethylhexanol stock solution was transferred in to 100mL volumetric flask and made up to the mark with Ethyl acetate.

Sample Solution Preparation

Weighed accurately 1g (±0.1g) of Octyl Stearate sample in 100mL volumetric flask and dissolved in Ethyl acetate and made up to mark with Ethyl acetate.

Sample Analysis

Ethyl acetate which was used as a diluent was taken in to GC vial and injected for blank in GC. A 2-Ethylhexanol standard solution was taken in to a GC vial and injected for standard with replicate the injections up to six times; calculated the average area of 2-Ethylhexanol for standard. Octyl Stearate sample solution was taken in to a GC vial and injected with replicates injection in GC; calculated the average area of 2-Ethylhexanol for sample.

Based on calculation which was mentioned below, calculated the % of 2-Ethylhexanol.

Calculation

$$\% \text{ of 2-Ethylhexanol} = \frac{\text{(Average Sample Area)}}{\text{(Average Standard Area)}} \times \frac{\text{(Standard weight)}}{\text{(Sample weight)}} \times \frac{1}{100} \times \text{Standard purity.}$$

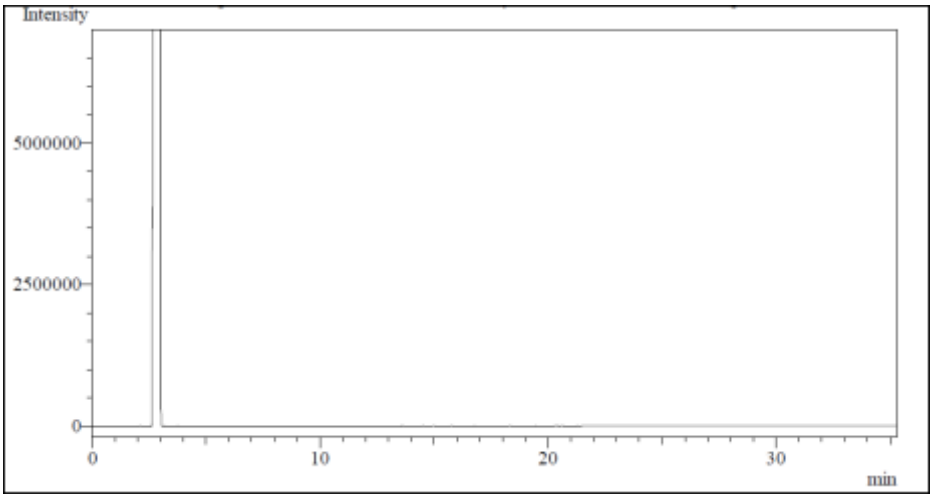

Figure 4: GC Chromatogram of Ethyl acetate blank

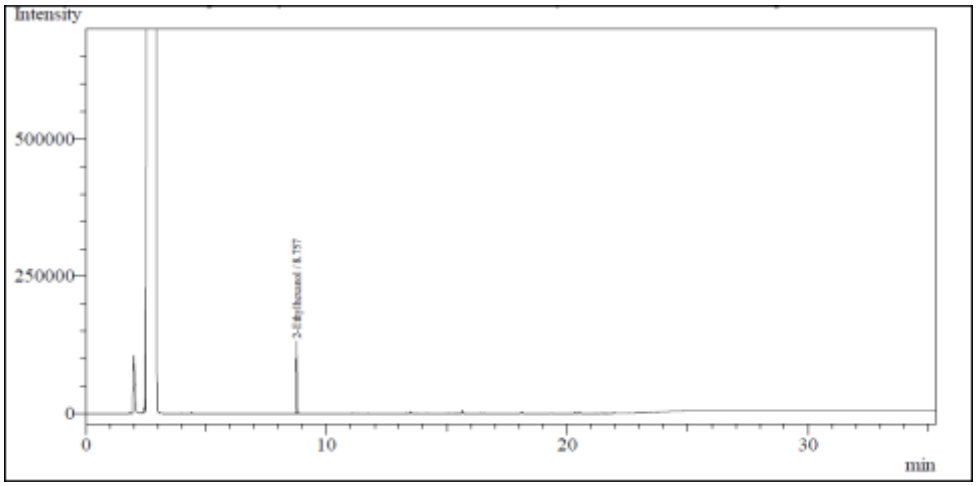

Figure 5: GC Chromatogram of 2-Ethylhexanol Standard

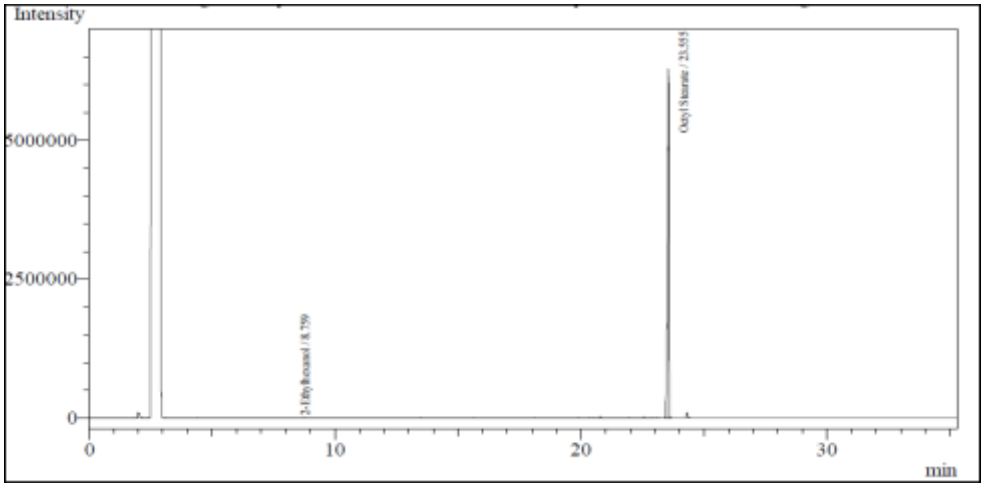

Figure 6: GC Chromatogram of Octyl Stearate

Method Validation

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.

Linearity

Linearity was checked by preparing five concentrations of the substance ranging from 50% to 150%. A concentration of 100ppm solution was proposed in the procedure as a 100%. Hence, the test substance was prepared at concentrations of 50ppm, 75ppm, 100ppm, 125ppm, and 150ppm 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 0.9991. The results are shown in table1.

Accuracy

The accuracy of the method was determined by adding known amount of 2-Ethylhexanol corresponding to following concentration levels of 50ppm, 100ppm and 150ppm target analyte concentration(100ppm). The accuracy was calculated by the percentage of analyte recovered by the assay method. The accuracy results are shown in table2.

Precision

The method precession, system precession and intermediate precession were calculated. The results were within the limits**.** Method precession results are shown in table 3. The system precession and intermediate precession results are shown in table4.

Robustness of the method

Changing with the flow rate (±0.2mL/min), and temperature program, no change in area of 2-Ethyl hexanol and RSD was within limits. It indicates robustness of the method.

Ruggedness of the method

Changing with different analyst the results are within limits. It indicates ruggedness of the method.

LOD and LOQ

Method limit of detection is 0.02ppm and limit of quantification is 0.05ppm.

EXPERIMENTAL RESULTS

The method was optimized based on using HP-5MS capillary column in gas chromatography. This method was validated as per ICH guidelines for specificity, linearity, accuracy, precession, robustness and ruggedness. The results were within limits.

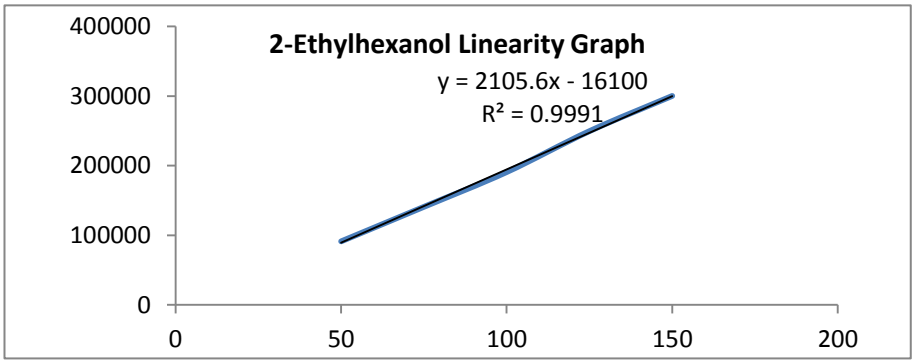

Figure 7: Linearity graph for 2-Ethylhexanol

Table1: Linearity

Parameter	Value
Concentration range(ppm)	50 ppm to 150 ppm
Slope of regression	2105.6
Intercept	-16100
Correlation coefficient	0.9991

Table 2: Accuracy

Added amount (ppm)	Recovery (%)
50	105.07
100	105.01
150	106.96

Table 3: Method Precession

Preparation	Assay (%)
Preparation-1	101.28
Preparation-2	100.16
Preparation-3	100.68
Preparation-4	100.55
Preparation-5	101.63
Preparation-6	103.82
Average	101.35
Standardeviation	1.32
% RSD	1.30%

Table 4: System precession and Intermediate precession

Precession	%RSD
System Precession	0.59%
Intermediate Precession	1.56%

CONCLUSION

This developed and validated GC method was very simple, selective and reproducible method for the quantification of 2-Ethylhexanol in Octyl Stearate. Solution preparation was very simple on this method. As per best of my knowledge, this method was first time developed for evaluation of 2-Ethylhexanol in Octyl Stearate. This method was having very low detection limits and low quantification limits. This method was suitable for determination of 2-Ethylhexanol in cosmetic products. This method was specific, linear, precise, accurate and robust. So, this method is very good suitable for analysis of 2-Ethylhexanol in cosmetic products.

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