Research Article

Determination of Methyl Paraben from Cosmetics by UV Spectroscopy

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ABSTRACT

This experiment is used for the determination for to estimate the concentration of methyl paraben in cosmetics. Methyl paraben is an antimicrobial agent, preservative, flavouring agent. The European Economic Community (EEC) Directive stipulates that parabens are permitted in a concentration of up to 0.8% in cosmetics, with a maximum concentration for each individual one of 0.4% (w/w), expressed as p-hyroxybenzoic acid. Effectively of parbens in high concentration causes cancer, genotoxicity and breast cancer. Specifically for methyl paraben caused contact dermatitis and drug hypersensitivity. So, need to determine the concentration of methyl paraben in cosmetics. The simple, rapid and sensitive UV spectroscopy method was developed for the determination of methyl paraben in cosmetics products. Methyl paraben maximum absorbance at 254 nm. The extraction of Methyl paraben in cosmetics may used cream, shampoo and lotion the optimal extraction solvent is methanol. The validation parameter of Methyl paraben studied the Y= 0.081x+ 0.011, R2 = 0.9940. LOD and LOQ were 0.3095 and 0.9378.

Keywords: Methyl paraben, UV spectroscopy, cosmetics, validation.

INTRODUCTION

Methyl paraben other name is a 4-hydroxybenzoate (methyl 4-hydroxybenzoate).[1] It is the most frequently used antimicrobial preservative in cosmetics and flavouring agent. Methylparaben is found in alcoholic beverages. Methylparaben odourless, small colourless crystals or white crystalline powder Freely soluble in alcohol, methanol, acetone, ether.[Error! Reference source not found.] Instance paraben is now used for preservatives in food, pharmaceuticals and cosmetics, daily used the products that continue of methyl paraben may be caused in the future breast cancer.[3] The super scale use of preservatives in cosmetics can result in potential health risked. Most of the preservatives may be harmful to the consumer due to their potency to induce allergic contact dermatitis.[4] Due to their broad antimicrobial spectra with relatively low toxicity, good stability and non-voltality, parabens are commonly used as preservatives to prevent alteration and degradation of cosmetics, pharmaceutical and food from microbial and fungal contamination and protect the consumers.[4] Several of the parabens with reported uses are used in products that can be ingested incidentally (e.g., Methyl paraben at up to 0.35% in lipstick), used near the eye (e.g., Methyl paraben at up to 0.8% in mascara), come in contact with mucous membranes (e.g., Methyl paraben at up to 0.5% in bath oils, tablets and salts), or in baby products (e.g., Methyl paraben at up to 0.4% in baby lotions, oils and creams).[5] Hygienic standard for cosmetics (2007 Edition, China) restricts the parabens preservatives limited dose. The upper limit of mass percentages of single ester was 0.4% and the upper limit of mixed esters was 0.8%.[6] The European Economic Community (EEC) Directive stipulates that parabens are permitted in a concentration of up to 0.8% in cosmetics, with a maximum concentration for each individual one of 0.4% (w/w), expressed as p-hyroxybenzoic acid.[3] Personal care product, such as hand cream, facial cleanser and moisturizer, are widely used in people's daily lives. But parabens may be added into these products during the production. It can be absorbed through the skin into the body.[6] Antimicrobial effectiveness must be demonstrated for all products for all products contain antimicrobial preservatives and for multiple dose topical dosage form and that is because the concentration of an antimicrobial preservatives may diminish during the shelf life of the product. Therefore the concentration of preservative agents must be determent as well as the probable consumption of these agents during the opening and closing of the cream must be calculated.[7] Bacterial contamination of the products through consumers use has resulted in presence of mixed and harmful microbial flora in the product.[8] Methyl paraben is an antifungal and preservative that is widely used in cosmetics. Because it is easily absorbed through the skin and is generally considered non-irritating, it is a very popular beauty product ingredient and is used to prevent fungal growth and to generally preserve formulas.[9] Despite their benefits, a controversy surrounds the discussion the effect of commonly used parabens, Methyl paraben, which can cause side effects on consumers and organoleptics alterations in the cosmetics. The studies revealed that the use of parabens may cause cancer, genotoxicity and breast cancer. Parabens are also reported to have side effect on males as it may decrease the reproduction potential, cause infertility and cause skin cancer diseases such as malignant, melanoma and contact eczema.[10]

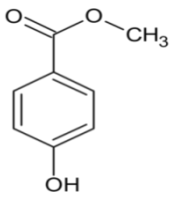

Figure 1: Methyl paraben

MATERIALS AND METHODS

Instrumentation

UV-Visible spectrophotometer (Double beam) having two matched quartz cells of light path 1 cm. shimadzu (UV-1800). Electronic analytical weighing balance (REPTECH), Ultrasonicator (LOBA life).

Material and Reagents

Methyl paraben (ozone international Mumbai), Methanol.

Method Preparation of Standard and Sample Solution

Stock solution

100mg methyl paraben accurately weighed and transferred into a 100ml volumetric flask and diluted up to the mark with methanol (1000ppm). Further 10 ml from methyl paraben stock solution was transferred into a 100 ml volumetric flask and diluted up to the mark with methanol (100ppm), then a series of solutions were prepared by 2,4,6,8,10 ppm of diluted solution in to 10 ml flasks and diluted up to the mark with methanol.

Sample (cream, lotion, shampoo) solution

The tested cosmetic products, including shampoo, body lotion body creams, sun creams, face creams, make up removals, were obtained at local markets. A 5.0 ml volume of methanol was added to the cosmetic samples (0.50gm). The emulsions were sonicated for 10 min, diluted to 10 ml and filtered through 0.22 µm Milipore membrane filters.[6]

List of Cosmetic Products:

1. Alograce cream
2. Lakme cream
3. Ponds cream
4. Nivea cream
5. Vaseline lotion
6. Neem Aloevera face pack
7. Dove shampoo
8. Clinic plus shampoo
9. Pantene shampoo
10. Almond shampoo

Method Validation[15,16]

The method was validated according to ICH Q2 (R1) Guideline. Validation is a process of establishing documented evidence, which provides degree of assurance that a specific process will consistently produce a desired product meeting its predetermined specifications and quality attributes.

Linearity (n=5)

The solution was prepared by pipetting 0.2, 0.4, 0.6, 0.8, and 1 ml from working stock solution into 10ml volumetric flask and the volume was adjusted to mark with methanol to produce 2-10µg/ml respectively. The absorbance of solutions was measured at 254 nm. Calibration curve was generated by taking the absorbance verses concentration. (Fig. 2,3 and Table 1)

Precision

Repeatability (n=6)

Aliquots of 0.6 ml of working standard solution of Methyl paraben (100µg/ml) were transferred to 10ml volumetric flask and volume was adjusted to methanol to get concentration of 6µg/ml. The absorbance of solution was measured spectrophotometry six times and % RSD was calculated. (Table no. 2)

Intraday precision (n=3)

Aliquots of 0.4, 0.6 and 0.8ml of working standard solution of Methyl paraben (100µg/ml) were transferred to 10ml volumetric flask and volume was adjusted to methanol to get concentration of 4, 6 and 8µg/ml. The absorbance of solution was measured spectrophotometry three times and % RSD was calculated. For intraday, the analysis was carried out at different intervals on the same day. (Table no. 3)

Interday precision (n=3)

Aliquots of 0.4, 0.6 and 0.8ml of working standard solution of Methyl paraben (100µg/ml) were transferred to 10ml volumetric flask and volume was adjusted to methanol to get concentration of 4, 6 and 8µg/ml. The absorbance of solution was measured spectrophotometry three times on the different days and % RSD was calculated. (Table no.4)

Limit of detection (LOD)

The LOD is estimated from the set of 3 calibration curves used to determine method linearity. The LOD may be calculated as,

LOD = 3.3 × (S.D./Slope)

Where, SD = the standard deviation of Y- intercept of 3 calibration curves.

Slope = Mean slope of the 5 calibration curves.

LOD= 0.3095004

Limit of Quantitation (LOQ)

The LOD is estimated from the set of 3 calibration curves used to determine method linearity. The LOD may be calculated as,

LOQ = 10 × (S.D./Slope)

Where, SD= the standard deviation

Y= intercept of 3 calibration curves

Slope = Mean slope of the 3 calibration curves.

LOQ = O.93788

Robustness

Robustness of a method is a study of the effect of small variation of the experimental conditions on reproducibility of the measurements. In the present investigation a study of robustness was carried out by making a small change in wavelength (±2) of measurements. (Table no.5)

Calculation

Concentration of methyl paraben

IN PPM (µg/ml):

Y= mx+c

Y = 0.081x+0.011 put the value of y= absorbance of sample in this formula.

The value obtained x is your concentration in ppm (µg/ml)

IN MILIGRAM (mg)

Conc. Of methyl paraben (mg) = Concentration in ppm* dilution factor

Here, dilution factor: 10/1*10/1*10/1 (1000) or, 10/1*10/1(100)

RESULTS AND DISCUSSION

Table 1: Linearity study of Methyl paraben

Concentration (µg/ml)	Absorbance (254nm)
2	0.1893
4	0.3194
6	0.4823
8	0.6887
10	0.8177

In most of selected cosmetics products the contents of Methyl paraben were found to be within specified range i.e. Not more than 0.4 % w/w. The products like Aloe grace (0.420), Lakme (0.4064), Ponds (0.479), Vaseline (0.4041) shows content of Methyl paraben more than 0.4 % w/w and *Aloe vera* (0.1528) which contain amount less than 0.4% w/w and other products persist negligible amount of Methyl paraben in range 0.04 - 0.07% w/w in shampoos which may be considered as safer for daily use. (Table 6).

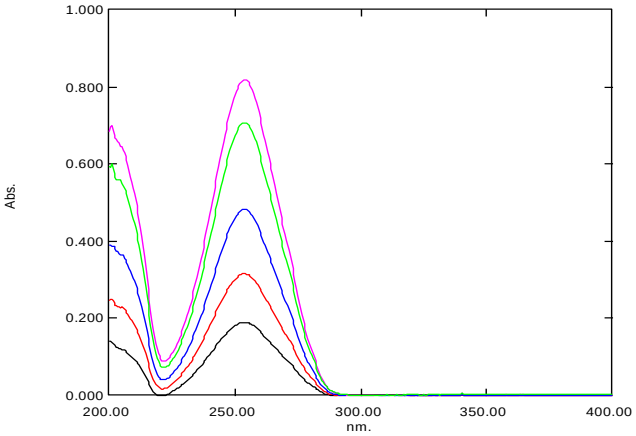

Figure 2: Linearity of Methyl paraben

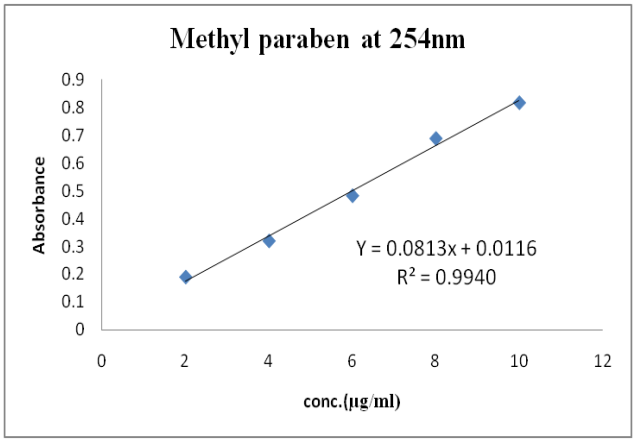

Figure 3: Calibration curve of Methyl paraben.

Table 2: Repeatability

Sr. No.	Absorbance
1	0.4823
2	0.4802
3	0.4828
4	0.4822
5	0.4835
6	0.4932
MEAN	0.484033
SD	0.004624
%RSD	0.955246

Table 3: Intraday precision

Sr. No.	Conc.(µg/ml)	Abs 1	Abs 2	Abs 3	Mean	SD	%RSD
1	4	0.3194	0.3261	0.3245	0.3233	0.0034	1.0821
2	6	0.4823	0.4891	0.4923	0.4879	0.0051	1.0467
3	8	0.6889	0.6811	0.6959	0.6886	0.0074	1.0751

Table 4: Interday precision

Sr. No.	Conc.(µg/ml)	Abs 1	Abs 2	Abs 3	Mean	SD	%RSD
1	4	0.3194	0.3251	0.3295	0.3246	0.0050	1.5597
2	6	0.4823	0.4821	0.4933	0.4892	0.0060	1.2334
3	8	0.6889	0.6801	0.6989	0.6893	0.0094	1.3626

Table 5: Robustness

Wavelength (nm)	Absorbance
252	0.4951
254	0.4823
256	0.4745

Table 6: Absorbance and % of Methyl paraben found in cosmetic products

Sr. No.	Cosmetic product	Absorbance	% of methyl paraben found
1	Alograce cream	0.1815	0.4200
2	Lakme cream	0.1756	0.4064
3	Ponds cream	0.2050	0.4790
4	Nivea cream	0.1919	0.0440
5	Vaseline lotion	0.1747	0.4041
6	Neem aloe vera face pack	0.6302	0.1528
7	Dove shampoo	0.2034	0.0475
8	Clinic plus shampoo	0.3048	0.0725
9	Pantene shampoo	0.2181	0.0511
10	Almond shampoo	0.3048	0.0475

CONCLUSION

The method of detected and validated parabens in daily cosmetics by UV- spectroscopy was established. The result obtained confirm that the proposed method is simple, rapid, selective and a good sensitivity, for the determination of Methyl paraben. Which did not need any complex extraction procedure. The method of spectroscopy which shows maximum absorption at 254 nm. It can be applied successfully for determination of methyl paraben from cosmetic in range of 0.4 %. So, it is suitable for the analysis of parabens in daily cosmetics.

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