



**International Journal of Biology, Pharmacy
and Allied Sciences (IJBPAS)**

'A Bridge Between Laboratory and Reader'

www.ijbpas.com

DETERMINATION OF METHYL PARABEN FROM MARKETED COSMETIC PRODUCTS BY UV SPECTROSCOPY

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Received 15th April 2024; Revised 20th Aug. 2024; Accepted 15th Sept. 2024; Available online 1st Sept. 2025

<https://doi.org/10.31032/IJBPAS/2025/14.9.9314>

ABSTRACT

This investigation aims to quantify the amount of methyl paraben found in cosmetic products. Methyl paraben serves as preservative, and antibacterial. According to the European Economic Community (EEC) Directive, parabens, also known as p-hydroxybenzoic acid, are allowed in cosmetics at a maximum concentration of 0.4% (w/w) per individual paraben. This allows for a concentration of up to 0.8% in cosmetics. High concentrations of parabens effectively cause genotoxicity, breast cancer. Therefore, it is necessary to ascertain the methyl paraben content of cosmetics. The detection of methyl paraben in cosmetic items was made possible by the development of a straightforward, quick, and sensitive UV spectroscopy approach. Maximum absorbance of methyl paraben at 254 nm. Methanol is the best extraction solvent for the methyl paraben removal process, which may be applied to cream. The $Y = 0.805$, was the validation parameter for methyl paraben. The LOD and LOQ values found to be 0.1237 and 0.3750, respectively.

Keywords: Methyl paraben, UV spectroscopy and LOD, LOQ & Methanol

INTRODUCTION:

Methyl paraben chemically named as utilized flavouring and antibacterial (methyl 4- hydroxybenzoate) [1]. Widely preservative in cosmetics. For example,

paraben is currently utilized as a preservative in food, medications, and cosmetics; regular usage of these goods containing methyl paraben may contribute to the development of breast cancer in the future [2]. Beverages with alcohol include methylparaben. White crystalline powder or tiny colourless crystals devoid of odour are methylparaben. Solubility without restriction in acetone, methanol, alcohol, and ether. Parabens are frequently used as preservatives in food, pharmaceutical, and cosmetic products to prevent microbial and fungal contamination and to preserve the products quality while also protecting consumers. This is because of their broad antibacterial spectrum, relatively low toxicity, good stability, and non-volatility [3]. The extensive usage of preservatives in cosmetics may pose a harm to one's health. Due to their ability to cause allergic contact dermatitis, the majority of preservatives may be dangerous for consumers. Due to microbial flora that is both mixed and dangerous, products that have been contaminated by bacteria as a result of consumer use²Antimicrobial efficacy needs to be demonstrated for all products containing antimicrobial preservatives as well as topical dosage forms with numerous doses, as the concentration of these preservatives may decline over the duration of the product's shelf life. As a result, it is necessary to determine both the

concentration of preservation agents that should be avoided and the likelihood that these compounds will be consumed when the cream is opened and closed [4]. Some of the parabens that have been used in products are those that are intended to be inadvertently swallowed (such as lipstick containing up to 0.35% paraben), used in close proximity to the eyes (such as mascara containing up to 0.8% paraben), enter into contact with the mucosal surfaces (such as bath oils, tablets, and salts containing up to 0.5% paraben), or are used in baby products (such as lotions, oils, and creams containing up to 0.4% paraben) [5]. In daily life, many people use personal care products including hand cream, face cleanser, and moisturizer. Nevertheless, during production, parabens could be added to these items. It is capable of entering the body through the skin. A single ester's maximum mass percentage was 0.4%, and combined esters' maximum mass percentage was 0.8%. The European Economic Community (EEC) Directive states that parabens, also known as p-hydroxybenzoic acid, may be included in cosmetics at a maximum of 0.4% (w/w) per individual paraben. This allows for a concentration of up to 0.8% [2]. It has been observed that parabens can have adverse effects on males, including reduced fertility, infertility, and skin cancers such malignant melanoma and contact eczema. Methyl paraben, a prevalent paraben that can affect

the organoleptic properties of cosmetics and have negative effects on consumers, is a topic of dispute despite its many benefits. According to the studies, paraben consumption may result in genotoxicity, cancer, and breast cancer [6].

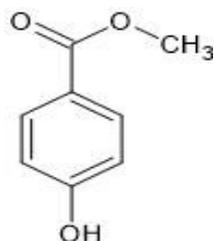


Figure 1: Methyl paraben

MATERIALS AND METHOD [8, 9]:

Instrumentation:

Instrument name: UV/vis Spectrophotometer, Company: SHIMADZU & Model No: 1800.

Materials and reagents:

Methyl paraben and methanol.

Method of preparation standard and sample solution [10, 11]:

- **Stock solution:**

Precisely weigh out 50 mg of methyl paraben, then transfer it into a 50 ml volumetric flask and dilute it to the appropriate level using a concentration of 1000 µg/ml methanol stock 1. Take 0.5 ml of the methyl paraben stock solution from stock 1 and dilute it with 10 µg/ml of methanol stock 2 in a 50 ml volumetric flask. Next, produce a series of solutions containing 0.2, 0.4, 0.6, 0.8, and 1.0 µg/ml and dilute them with methanol to the

appropriate level.

- **Sample solution:**

Local markets provided the tested cosmetic products. A capacity of 5.0 ml The cosmetic samples received 0.50 grams of methanol added to them. The emulsions underwent a 10-minute sonication, were diluted to 10 milliliters, and then passed through whattman's filters paper to filter.

Method validation:

The ICH Q2 (R1) Guideline was followed in the validation of the procedure. Establishing documented proof, also known as validation, offers some assurance that a particular procedure will consistently yield the desired outcome that satisfies its predetermined requirements and quality standards.

Linearity:

A 10-ml volumetric flask may be used to manufacture the solution by pipetting 0.2, 0.4, 0.6, 0.8, and 1 ml of the working stock solution. The volume was then marked with methanol to produce 0.2–1.0 µg/ml, in that order. At 254 nm, the absorbance of the solutions was measured. The absorbance versus concentration was used to create the calibration curve.

Precision:

- **Repeatability:**

To obtain a concentration of 0.6µg/ml, aliquots with 0.6 ml of the methyl paraben (100µg/ml) working standard solution were

put into a 10 ml volumetric flask, and the volume was changed to methanol. Using spectrophotometry, the solution's absorbance was measured six times, and the % RSD was computed.

- **Intraday precision:**

To get 0.4, 0.6, and 0.8 µg/ml concentrations, aliquots of the methyl paraben working standard solution

(100µg/ml) were measured in 0.4, 0.6, and 0.8 ml. were transferred to a 10-milliliter volumetric flask, and the volume was changed to methanol. The solution's absorbance was measured three times using spectrophotometry, and the percentage RSD was calculated. The study was conducted multiple times on the same day for intraday.

Table 1: Intraday precision value

S. No.	Conc.(µg/ml)	Abs 1	Abs 2	Abs 3	Mean	SD	%RSD
1	0.4	0.321	0.343	0.334	0.3326	0.011452	3.443328
2	0.6	0.498	0.523	0.509	0.51	0.014370	2.817668
3	0.8	0.729	0.789	0.753	0.757	0.030199	3.989344

- **Interday precision:**

A 10 ml volumetric flask was filled with aliquots of 0.4, 0.6, and 0.8 ml of the methyl paraben working standard solution (100µg/ml); the volume was then adjusted to

methanol to get concentrations of 0.4, 0.6, and 0.8µg/ml. Three separate days of spectrophotometric measurements of the solution's absorbance were made, and the % RSD was computed.

Table 2: Inter day precision value

S. No.	Conc.(µg/ml)	Abs 1	Abs 2	Abs 3	Mean	SD	%RSD
1	0.4	0.327	0.312	0.329	0.3206	0.008505	2.652929
2	0.6	0.498	0.489	0.501	0.496	0.006244	1.259072
3	0.8	0.729	0.706	0.723	0.7193	0.011930	1.658615

Limit of detection (LOD):

The three calibration curves that were used to assess the linearity of the procedure are used to estimate the LOD. One way to compute the LOD is,

$$\text{LOD} = 3.3 \times (\text{S.D} / \text{slope})$$

Where, SD = the standard deviation

y- intercept of 3 calibration curve.

Slope = mean slope of the 5 calibration curves.

$$\text{LOD} = 0.123760$$

Limit of quantitation (LOQ):

The three calibration curves that were used to assess the linearity of the procedure are used to estimate the LOQ. One way to compute the LOQ is,

$$\text{LOQ} = 10 \times (\text{S.D} / \text{slope})$$

Where, SD = the standard deviation

y- intercept of 3 calibration curve.

Slope = mean slope of the 5 calibration curves.

LOQ = 0.375031

Robustness:

An analysis of a method's robustness looks at how minor changes to the experimental setup affect the measurements' repeatability.

A study of resilience was conducted in the current examination by slightly varying the wavelength (± 2) of observations.

RESULTS AND DISCUSSION:

Table 3: Linearity values

Concentration ($\mu\text{g/ml}$)	Absorbance (254nm)
0.2	0.274
0.4	0.375
0.6	0.577
0.8	0.738
1.0	0.896

Methyl paraben levels in the majority of the chosen cosmetics goods were determined to

be within the acceptable range, or less than 0.4% w/w.

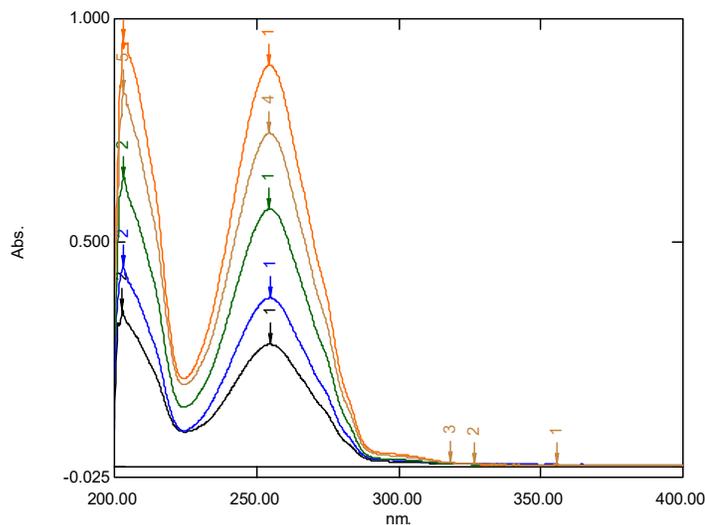


Figure 2: Spectrum of standard methyl paraben

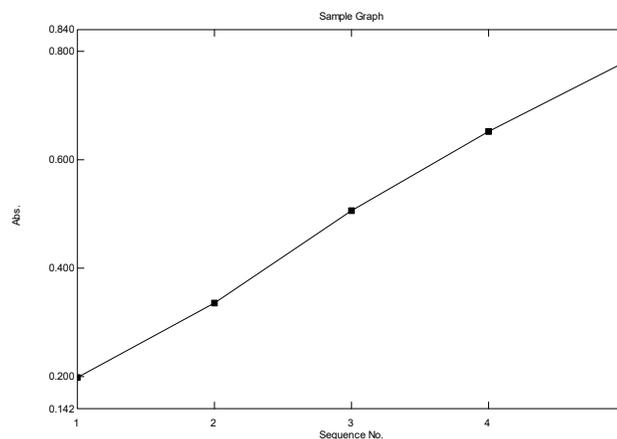


Figure 3: Calibration curve of Methyl paraben

Repeatability:**Table 4: Repeatability value with absorbance**

S. No.	Absorbance
1	0.375
2	0.375
3	0.375
4	0.372
5	0.372
6	0.377
MEAN	0.37433
SD	0.001966
%RSD	0.525308

Table 5: The Absorbance and % of methyl paraben present in cosmetic products

S. No.	Samples	Absorbances	% of methyl paraben
1	Samples 1	0.271	0.3366
2	Samples 2	0.332	0.4124
3	Samples 3	0.312	0.3875

CONCLUSION:

It was determined how to use UV spectroscopy to identify and confirm the presence of parabens in everyday cosmetics. The outcomes validate that the suggested approach for determining methyl paraben is easy to use, quick, accurate, and has a high level of sensitivity. It required no elaborate extraction process. The spectroscopic approach that exhibits the highest absorption at 254 nm. In the range of 0.4%, It works well for figuring out how much methyl paraben is in makeup. As such, it is suitable for examining parabens in regular cosmetics.

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