

NOVEL UV- SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION FOR THE DETERMINATION OF DOXAZOSIN MESYLATE IN API AND TABLET DOSAGE FORM

Kavana D. C.^{*1}, Ananya B. H.², Priya R.², Rakshitha B.², Sadhana C.²

¹Assistant Professor, Department of Pharmaceutical Analysis, Bharathi College of Pharmacy, Bharathinagara, Mandya District, Karnataka, India -571422.

²UG scholar, Department of Pharmaceutical Analysis, Bharathi College of Pharmacy, Bharathinagara, Mandya, Karnataka, India -571422.

Article Received: 07 August 2026 | Article Revised: 28 August 2026 | Article Accepted: 17 September 2026

***Corresponding Author: Kavana D. C.**

Assistant Professor, Department of Pharmaceutical Analysis, Bharathi College of Pharmacy, Bharathinagara, Mandya District, Karnataka, India - 571422. DOI: <https://doi.org/10.5281/zenodo.22796431>

How to cite this Article: Kavana D. C., Ananya B. H., Priya R., Rakshitha B., Sadhana C. (2026) NOVEL UV- SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION FOR THE DETERMINATION OF DOXAZOSIN MESYLATE IN API AND TABLET DOSAGE FORM. World Journal of Pharmaceutical Science and Research, 5(9), 820-825.



Copyright © 2026 Kavana D. C. | World Journal of Pharmaceutical Science and Research.

This work is licensed under creative Commons Attribution-NonCommercial 4.0 International license (CC BY-NC 4.0).

ABSTRACT

Doxazosin Mesylate is an α_1 -adrenoceptor antagonist commonly used in the management of benign prostatic hyperplasia and hypertension. The present study describes the development and validation of a novel, simple and rapid zero-order UV-spectrophotometric method for the determination of Doxazosin Mesylate in API form. The method was developed using methanol as the solvent and the drug solution was scanned in the wavelength range of 200–400 nm. The maximum absorbance was observed at 246 nm. The method exhibited good linearity over the concentration range of 1.5–9 $\mu\text{g/mL}$, with a regression equation of $Y = 0.0656X - 0.0021$ and a correlation coefficient (R^2) of 0.9992. Precision studies demonstrated good repeatability and intermediate precision, with %RSD values below 2%. The method also showed satisfactory ruggedness between different analysts. The developed method was found to be simple, precise, sensitive and reliable and can be suitably applied for the routine quantitative determination of Doxazosin Mesylate in API form.

KEYWORDS: Doxazosin, zero-order UV-spectrophotometry, Methanol.

INTRODUCTION

A common source of troublesome lower urinary tract symptoms in males is benign prostatic hyperplasia (BPH), which is histologically characterized by prostatic stromal and epithelial hyperplasia. About 40% of men over 60 experience lower urinary tract symptoms, such as frequent urination, nocturia, decreased urine flow and intermittency, and a

feeling of incomplete bladder emptying. About 35% of men who turn 50 are thought to need treatment for BPH at some point in their lives.

Apart from the structural bladder outlet obstruction caused by prostatic enlargement, there seems to be a dynamic component as well, with sympathetic α 1-adrenoceptor activation raising smooth muscle tone in the bladder neck and prostate. Doxazosin is a well-established, safe, and effective α 1-adrenoceptor selective antagonist that was first used to treat hypertension. It is administered once daily to relieve clinical symptoms in BPH. However, a multiple-step titration strategy is necessary when using doxazosin in its conventional formulation to reduce the possibility of a first-dose hypotensive impact.^[1]

Extensive literature survey reveals that various analytical methods have been developed such as UV spectroscopy methods^[2-3], RP- HPLC method^[4-8], Stability indicating method^[9-10] HPTLC simultaneous method^[11], and Stability indicating UPLC method^[12] for the estimation of Doxazosin Mesylate present in alone or in combination with other drugs or in marketed products. Most of the reported UV Spectroscopic methods are Simultaneous estimation of Doxazosin Mesylate, along with other β -3 adrenoreceptor or agonist drugs, by using several solvents, expensive reagents and often time consuming. So, there is a need to develop novel UV Spectroscopic methods for the estimation of Doxazosin Mesylate alone in bulk and pharmaceutical formulation.

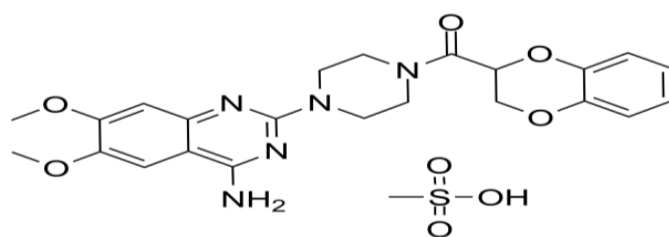


Figure 1: Chemical structure of Doxazosin Mesylate.

MATERIALS AND METHOD

Instrument

UV-Visible double beam spectrophotometer, SHIMADZU (model UV-1800) with UV probe software. All weights were taken on analytical balance.

Chemicals: Doxazosin

Solvent: Methanol

Selection of analytical wavelength

Appropriate dilutions were prepared for drug from the standard stock solution and the solution was scanned in the wavelength range of 200-400nm. The absorption spectra thus obtained were derivative from Zero order method. It shows maximum absorbance at 246 nm were shown in Fig.2.

Preparation of Standard stock solution:

Accurately weigh 100mg of Doxazosin Mesylate was transferred into 100ml volumetric flask and diluted with Methanol up to the mark. From this pipette out 10ml into 100ml volumetric flask and diluted with Methanol up to the

mark, from this solution pipette out 0.15, 0.3, 0.45, 0.6, 0.75 and 0.9ml in 10ml individual volumetric flask and add Methanol up to the mark, this gives 1.5, 3, 4.5, 6, 7.5 and 9 $\mu\text{g/ml}$ concentrations.

Preparation of Sample solution

The powder equivalent to 100 mg of Doxazosin Mesylate was transferred into 100 ml volumetric flask then it was diluted with the Methanol and made up to the mark. From the above solution 10 ml was pipetted out into 100 ml volumetric flask and the volume was made up to the mark with Methanol. The final concentration of Doxazosin mesylate was brought to 2.25, 4.5 and 6.75 $\mu\text{g/ml}$.

Method validation

The method is validated according to the ICH guidelines.^[13-15]

RESULTS AND DISCUSSION

Method: Zero order derivative spectroscopy

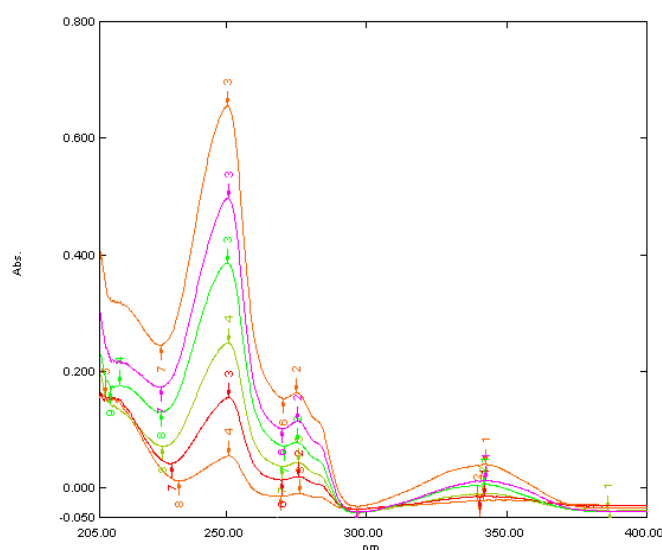


Fig. 2: Zero order spectra of Doxazosin Mesylate showing absorbance at 246 nm.

Linearity

The working standard solution were diluted serially with Doxazosin Mesylate to obtain the range of 1.5-9 $\mu\text{g/ml}$. A calibration curve for Methanol was obtained by measuring the absorbance at the λ max of 246nm and absorbance values are shown in Table.1 and Calibration graph were presented in Fig.3. Statistical parameters like slope, intercept, coefficient of correlation, and Sandel's sensitivity were determined and presented in Table.2.

Table 1: Results of calibration curve for Doxazosin Mesylate at 246nm by zero order Spectroscopy.

Sl. no	Concentration in $\mu\text{g/ml}$	Absorbance at 249nm $\pm$ SD
1	1.5	0.097 $\pm$ 0.00094
2	3	0.197 $\pm$ 0.00082
3	4.5	0.284 $\pm$ 0.00222
4	6	0.396 $\pm$ 0.00227
5	7.5	0.4835 $\pm$ 0.00169
6	9	0.595 $\pm$ 0.00249

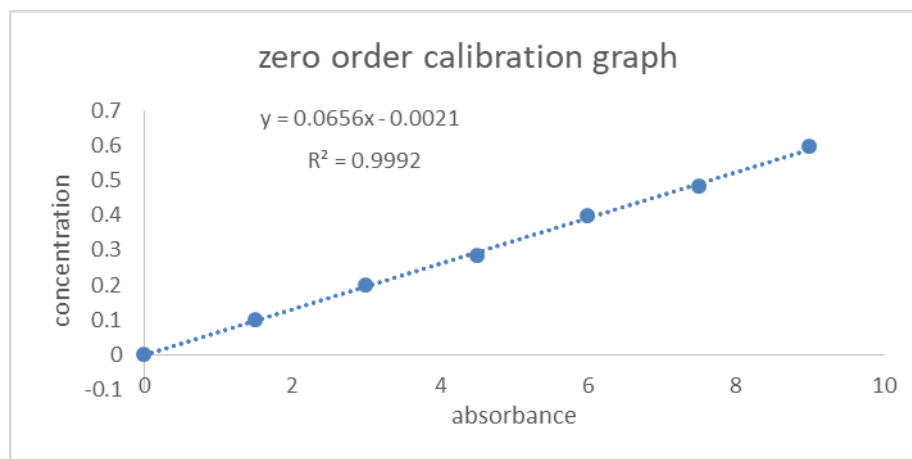


Fig. 3: Calibration curve for Doxazosin Mesylate at 246 nm by Zero order Spectroscopy.

Table no. 2: Regression parameters for Doxazosin Mesylate by zero order spectroscopy.

Optimum Condition	UV Method
$\lambda_{\text{max}}(\text{nm})$	246 nm
Beer's law limits ($\mu\text{g/ml}$)	1.5-9 $\mu\text{g/ml}$
Sandell's sensitivity	0.0158 mcg / cm^2
Regression equation (Y^*)	$Y=0.0656x - 0.0021$
Slope (b)	0.0656
Intercept (a)	- 0.0021
Co-relation coefficient(R^2)	0.9992

Precision

Precision of the method was studied as intra-day and inter-day precision. Intra-day precision was determined by analyzing the 1.5, 3, 4.5, 6, 7.5, and 9 $\mu\text{g/ml}$ concentration for three times in same day. Inter-day precision was determined by analyzing the same concentration of solution daily for three days. Precision results are shown in Table.3.

Table 3: Determination of precision results for Doxazosin Mesylate at 246nm by Zero order derivative spectroscopy.

Concentration ($\mu\text{g/ml}$)	Intra-day Absorbance $\pm\text{SD}^{**}$	%RSD	Inter-day Absorbance $\pm\text{SD}^{**}$	%RSD
1.5	0.097 $\pm$ 0.00094	0.9624	0.0975 $\pm$ 0.00096	0.9846
3	0.197 $\pm$ 0.00082	0.4162	0.19683 $\pm$ 0.00107	0.5436
4.5	0.284 $\pm$ 0.00222	0.7803	0.2845 $\pm$ 0.00171	0.6010
6	0.396 $\pm$ 0.00227	0.5732	0.3965 $\pm$ 0.00171	0.4312
7.5	0.4835 $\pm$ 0.00169	0.3495	0.4835 $\pm$ 0.00171	0.3536
9	0.595 $\pm$ 0.00249	0.41845	0.5955 $\pm$ 0.00170	0.2854

Accuracy

To assess the accuracy of the proposed method, recovery studies were carried out at three different levels i. e, 50%, 100% and 150%. In which the formulation concentration was kept constant and varied pure drug concentration. Accuracy results were shown in Table.4.

Table.4: Determination of accuracy results for Doxazosin Mesylate by Zero order derivative spectroscopy.

Spiked Levels	Amount of sample (µg/ml)	Amount of standard (µg/ml)	Amount Recovered (µg/ml)	%Recovery ±SD**	%RSD
50%	4.50	2.25	6.75	99.56± 0.4404	0.4404
100%	4.50	4.50	9.05	100.56±0.5883	0.5883
150%	4.50	6.75	11.33	100.71±0.7359	0.7359

Ruggedness

Ruggedness was determined between different analysts. The value of %RSD was found to be less than 2 were shown in Table.5.

Table 5: Determination of Ruggedness results for Doxazosin Mesylate at 246 nm by Zero order Spectroscopy.

Analysts	Analyst-1	Analyst-2
Mean absorbance	0.284	0.286
Standard deviation	0.0022	0.0025
%RSD	0.7803	0.7810

Limit of detection and Limit of Quantitation

The LOD and LOQ of the present method were calculated based on standard deviation of the Response and slope of linearity curve. LOD and LOQ values of Acarbose were found to be 0.03418µg/ml and 0.10357µg/ml.

CONCLUSION

In the present investigation, we have developed Novel, simple, accurate and Precise UV- spectrophotometric methods like Zero order derivative spectroscopy for the routine determination of Doxazosin Mesylate in API form and the methods were validated in terms of linearity, accuracy, precision, ruggedness and robustness.

ACKNOWLEDGEMENT

We would like to thanks Management, Director, Principal, Project Guide and non-teaching staff of Bharathi college of pharmacy for their continuous co-operation and support.

REFERENCES

1. Goldsmith DR, Plosker GL. Doxazosin gastrointestinal therapeutic system: a review of its use in benign prostatic hyperplasia. *Drugs*, 2005 Oct; 65(14): 2037-47.
2. Neeraj R, Mahesh M. Development and validation of UV spectrophotometric method for determination of Doxazosin Mesylate in tablet formulation. *Der Pharmacia Sinica*, 2012.
3. P S Yadav, Hajare AA, Patil KS. Development and validation of UV spectrophotometric method for doxazosin mesylate in bulk and tablets. *Res J of Pharm and Tech*, 2022 Jun 1; 15(6): 2675-80
4. Kim YJ, Lee Y, Kang MJ, Huh JS, Yoon M, Lee J, Choi YW. High-performance liquid chromatographic determination of doxazosin in human plasma for bioequivalence study of controlled release doxazosin tablets. *Biomed Chromatography*, 2006 Nov; 20(11): 1172-7.
5. Bakshi M, Ojha T, Singh S. Validated specific HPLC methods for determination of prazosin, terazosin and doxazosin in the presence of degradation products formed under ICH-recommended stress conditions. *J pharm and biomed analysis*, 2004 Jan 27; 34(1): 19-26.
6. Dhanya B, Suganthi A, Sen AK, Sahoo U, Seth AK. Determination of doxazosin mesylate in tablets by RP-HPLC. *Indian J Pharm Sci*, 2011 Jan; 73(1): 120.

7. Maslarskr V Development and Validation of simple RP-HPLC method for determination of doxazosin mesylate Sci of Europe, 2023(132): 13-6.
8. Rao KS, Deb KB, Rao ME, Patro SS, Patnaik AK. Development and validation of stability indicating analytical method for doxazosin mesylate and its application to kinetic studies. Chemi Sci J., 2012 Jan 1.
9. Shrivastava A, Gupta VB. Stability-indicating RP-HPLC method for the simultaneous determination of prazosin, terazosin, and doxazosin in pharmaceutical formulations. Sci pharm, 2012 May 22; 80(3): 619.
10. Bebawy LI, Moustafa AA, Abo-Talib NF. Stability-indicating methods for the determination of doxazosin mesylate and celecoxib. J of pharm and biomed analysis, 2002 Feb 1; 27(5): 779-93.
11. Belal TS, Mahrous MS, Abdel-Khalek MM, Daabees HG, Khamis MM. Validated HPTLC method for the simultaneous determination of alfuzosin, terazosin, prazosin, doxazosin and finasteride in pharmaceutical formulations. Analytical Chemistry Res, 2014 Aug 1; 1: 23-31.
12. Sushma BL, Madhusudhan G, Jayashree A. Stability indicating UPLC method for determination of related substances in doxazosin mesylate. Asian J Res Chemistry, 2017 Mar 1; 10(2): 95.
13. ICH, Q2A Text on Validation of Analytical Procedures, 1994.
14. ICH, Q2B Validation of Analytical Methodology, 1996.
15. ICH, Q2B Validation of Analytical Methodology, 1996.