WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 8.453
Volume 14, Issue 13, 1341-1349. Research Article ISSN 2277–7105

DEVELOPMENT AND VALIDATION OF A UV SPECTROSCOPIC METHOD FOR ANALYSIS OF BILASTINE IN BULK AND TABLET FORM

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Article Received on
12 May 2025,

Revised on 02 June 2025,
Accepted on 21 June 2025

DOI: 10.20959/wjpr202513-37421

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ABSTRACT

Bilastine in bulk and pharmaceutical dose form can be estimated using a straightforward, accurate, and precise zero-order derivative UV spectroscopic approach that has been developed and validated. The absorbance of Bilastine in ethanol reaches its maximum at 278 nm, and its concentration falls between 2 and 12 µg/ml according to Beer's Law. After conducting a linearity investigation, the regression coefficient (R2) was shown to be 0.999, indicating good linearity and precision across this concentration range. The limits of detection (LOD) and quantitation (LOQ) were determined to be 0.0646 µg/ml and 0.196 µg/ml, respectively, while the percentage recovery was determined to be 98.63% and 100.10%. Additionally, the method's relative standard deviation (%RSD) values were less than 2%, indicating exceptional precision. Following ICH criteria, all validation parameters—linearity, accuracy, precision, robustness, ruggedness, LOD, and LOQ—were evaluated. Bilastine in bulk and pharmaceutical dosage forms can be routinely estimated using the designed and tested approach.

KEYWORDS: Bilastine, Zero order derivative spectroscopy , Validation , Pharmaceutical formulations.

INTRODUCTION

Bilastine is a selective histamine H1 receptor antagonist. During allergic response mast cells undergo degranulation which releases histamine and other subastances. By binding to and preventing activation of the H1 receptor, Bilastine reduces the development of allergic symptoms due to the release of histamine from mast cells. Bilastine is a novel H1 receptor antagonist indicated for the treatment of seasonal or perennial allergic rhinitis and symptomatic chronic urticaria, with little or no interactions with H2, H3 receptors, α1-adrenoceptors, β2-adrenoceptors, 5HT, bradykinin, leukotriene D4 or muscarinic M3 receptors1-2. It is a second-generation antihistamine and takes effect by selectively inhibiting the histamine H1 receptor, preventing these allergic reactions.[7] Bilastine has an effectiveness similar to cetirizine, fexofenadine, and desloratadine[8]. Bilastine was discovered by the Spanish firm FAES Farma[9] and received its first approval in the European Union in 2010 for the symptomatic treatment of allergic rhinoconjunctivitis and urticaria.[10] It is also approved in Canada and Australia. As of 2023, it remained unapproved for any use in the United States,[11] although Hikma Pharmaceuticals had agreed in 2021 to begin the FDA approval process.[12]

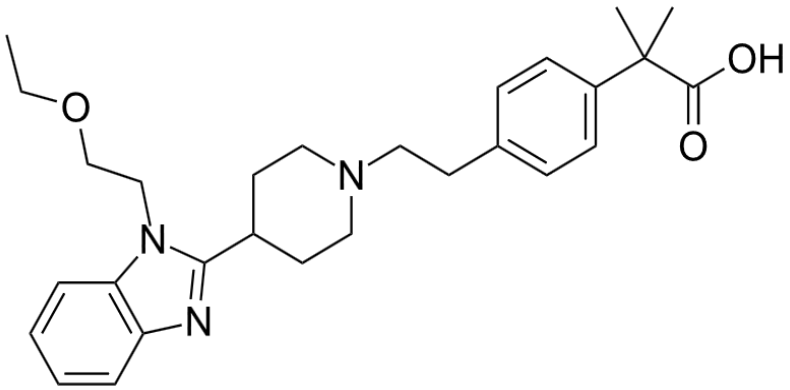

Figure 1: Chemical Structure of Bilastine.

According to a literature review, few analytical techniques have been published for determining Bilastine in pure medication and pharmaceutical dosage forms employing UV[3-7], HPLC[8-12], RP-HPLC[13-21], and HPTLC.[22-27] The current effort aims to develop and verify a new Zero order derivative UV Spectrophotometric technique for estimating Bilastine in tablet and bulk dose form that is quick, easy, accurate, and specific.

MATERIALS AND METHODS

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

Chemicals: Bilastine pure drug was obtained from Astitva chemicals and its pharmaceutical dosage Bilastine 20 tablets obtained from retail pharmacy.

Solvent: Acetonitrile and 0.1N HCL(50:50) is used as a solvent

Selection of analytical wavelength: Appropriate dilutions of Bilastine were prepared from standard stock solution and using spectrophotometer solution was scanned in the wavelength range 200-400 nm. The absorption spectra obtained and show maximum absorbance at 278 nm, as the wavelength for detection.

Preparation of standard stock solution: 100mg of Bilastine was weighed accurately transferred into 100 ml of volumetric flask and diluted in Acetonitrile and 0.1N HCL(50:50) upto the mark. From this, the solution was further diluted into 100µg/ml and pipetted out 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 ml into 10 ml individual volumetric flask and diluted in Acetonitrile and 0.1N HCL up to the mark, this gives 2, 4, 6, 8, 10 and 12µg/ml concentration.

Preparation of sample solution: 20 tablets of Bilastine marketed formulations was weighed and powdered. A quantity of tablet powder equivalent to 100mg of Bilastine was transferred into 100ml volumetric flask then it was diluted with Acetonitrile and 0.1N HCL and make upto the mark.

METHOD AND VALIDATION

The method was validated according to the ICH guidelines.[26-28]

RESULT AND DISCUSSION

Method: Zero order derivative spectroscopy

Linearity: The linearity of an analytical method is its capacity to show the test results that are directly proportional to the concentration to the analyte in the sample within the range. The linearity was established in the range of 2-12µg/ml was measured at 278m and absorbance values are shown in table 1. The calibration curve was prepared by plotting graph against the concentration and absorbance and therefore the graph shown in Fig-3 statistical variables like slope, intercept, regression equation, correlation coefficient and sandell's sensitivity were determined and shown in table-2.

Precision: The precision of an analytical method express the closeness of series of individual analytical measurement obtained from the multiple sampling of equivalent sample. Precision was established by intra-day and inter-day was determined by analysing the same concentration for six times in a same day. Inter-day precision was analysing the same concentration daily for six days shown in table-3.

Accuracy: The accuracy of an analytical method says that closeness of test results obtained by that method of the true value. To assess the accuracy of the developed method, recovery studies were carried out at three different levels at 50%, 100% and 150%. In which the formulation concentration holds it constant and varied pure drug concentration. Shown in table -4.

Ruggedness: The ruggesdness is defined as the reliability of results when the method is performed under the variation in condition. This includes distinct analyst, laboratories, instruments, temperature etc. Ruggedness was determined between disteinct analyst, the value of %RSD was found to be less than 2.(Table-5)

LOD and LOQ: The limit of detection is an individual analytical method is the smallest amount of analyte in the sample whuch can be reliably detected by the analytical method. The limit of quantification is a descrete analytical procedure is the smallest amount of analyte in the sample which can be quantitatively determined. LOD and LOQ were calculated by using following formula

LOD = 3.3(SD)/S and LOQ = 3(LOD)

LOD and LOQ value of Bilastine were found be 0.0646 µg/mL and 0.196 µg/mL.

Table 1: Results of calibration curve at 273nm by zero order derivative spectroscopy.

Sl No	Concentration in µg|ml	Absorbance ± Standard deviation
1	0	0
2	2	0.115±0.0017
3	4	0.235±0.0034
4	6	0.370±0.0027
5	8	0.513±0.0019
6	10	0.664±0.0030
7	12	0.771±0.0028

**Average of six determinations*

Table 2: Regression parameters of Bilastine by Zero order spectroscopy.

Regression Parameter	Results
Range	2-12 µg/ml
λ_{max}	273nm
Regression equation	Y=0.063x+0.0141
Slope(b)	0.063
Intercept (a)	0.0141
Correlation coefficient	0.999
Sandell's sensitivity	0.016
LOD(µg/ml)	0.0646
LOQ(µg/ml)	0.196

Y=bx+a**

Table 3: Determination of Precision results for Bilastine at 278nm by Zero order spectroscopy.

Concentration (µg|ml)	Intra-day Absorbance ± Standard deviation*	%RSD**	Inter-day Absorbance ± Standard deviation*	%RSD**
2	0.111±0.0017	1.531	0.110±0.0026	1.562
4	0.251±0.0034	1.446	0.239±0.0027	1.129
6	0.370±0.0027	0.729	0.372±0.0032	0.860
8	0.513±0.0019	0.370	0.519±0.0036	0.693
10	0.664±0.0030	0.451	0.640±0.0027	0.421
12	0.771±0.0028	0.363	0.765±0.0021	0.274

Average of six determinations, Percentage relative standard deviation.*

Table 4: Determination of accuracy results for Bilastine at 278nm by Zero order spectroscopy.

Spiked levels	Amount of sample (µg|ml)	Amount of standard (µg|ml)	Amount recovered	%Recovery± Standard deviation*	%RSD**
50	6	3	9.01	100.10%±0.218	0.2182
100	6	6	11.83	98.63%±0.206	0.2095
150	6	9	14.92	99.46%±0.127	0.1277

Average of six determinations, Percentage relative standard deviation.*

Table 5: Determination of ruggedness results of Bilastine at 278nm by Zero order spectroscopy.

Analysts	Analyst 1	Analyst 2
Mean absorbance	0.3701	0.3721
±Standard deviation*	0.0027	0.0027
%RSD**	0.729	0.725

Average of six determinations, Percentage relative standard deviation.*

FIGURES

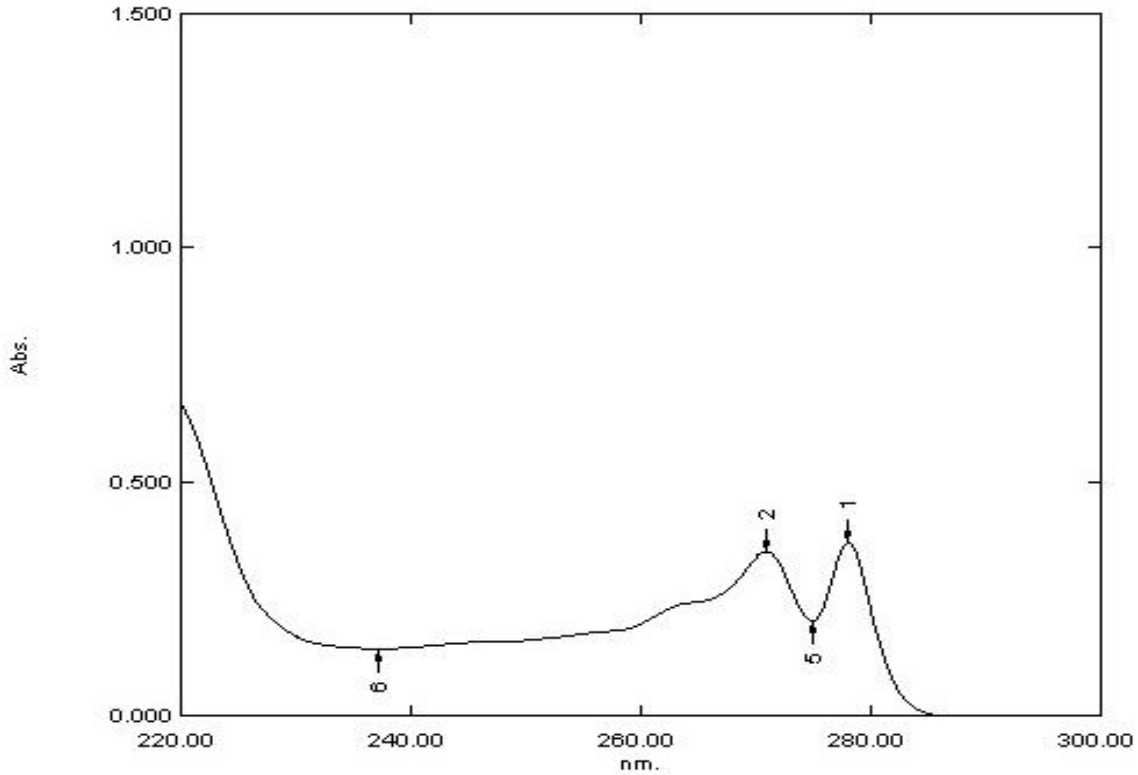

Fig. 2: Zero order spectrum of Bilastine at 278nm.

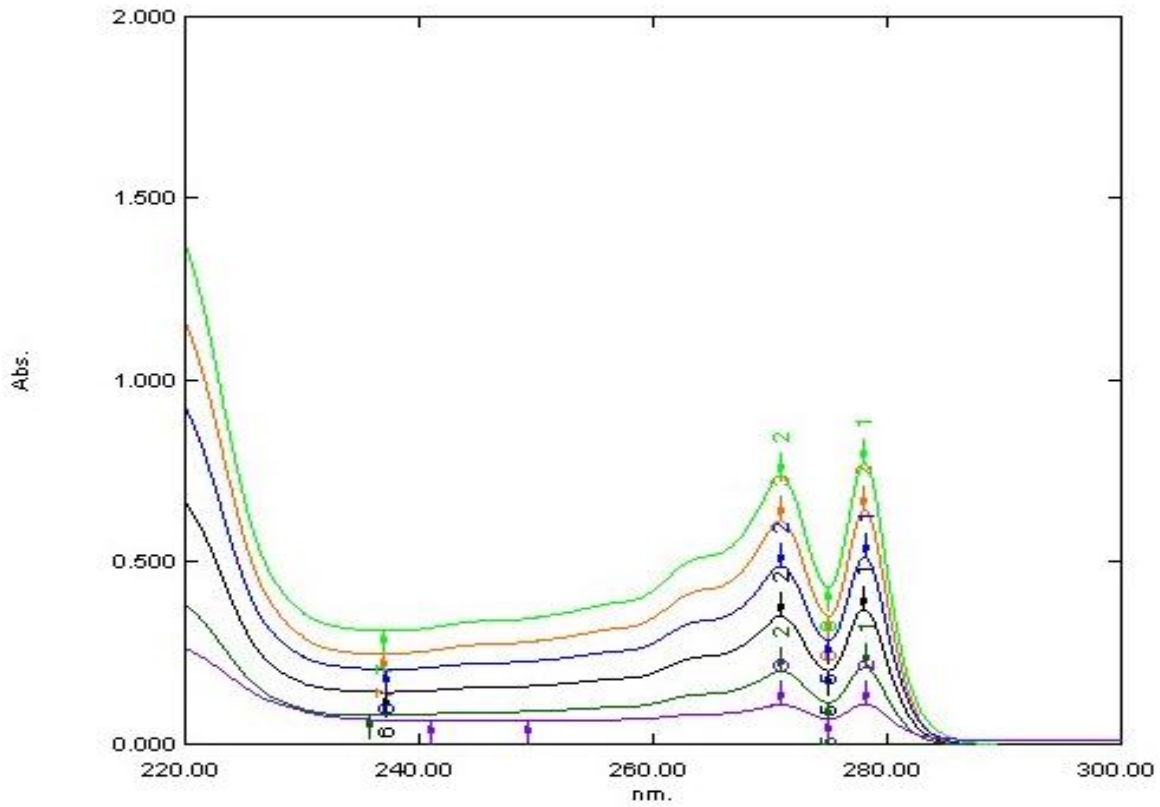

Figure 3: Zero Order Overlain Spectra of Bilastine Showing Absorbance At 278nm.

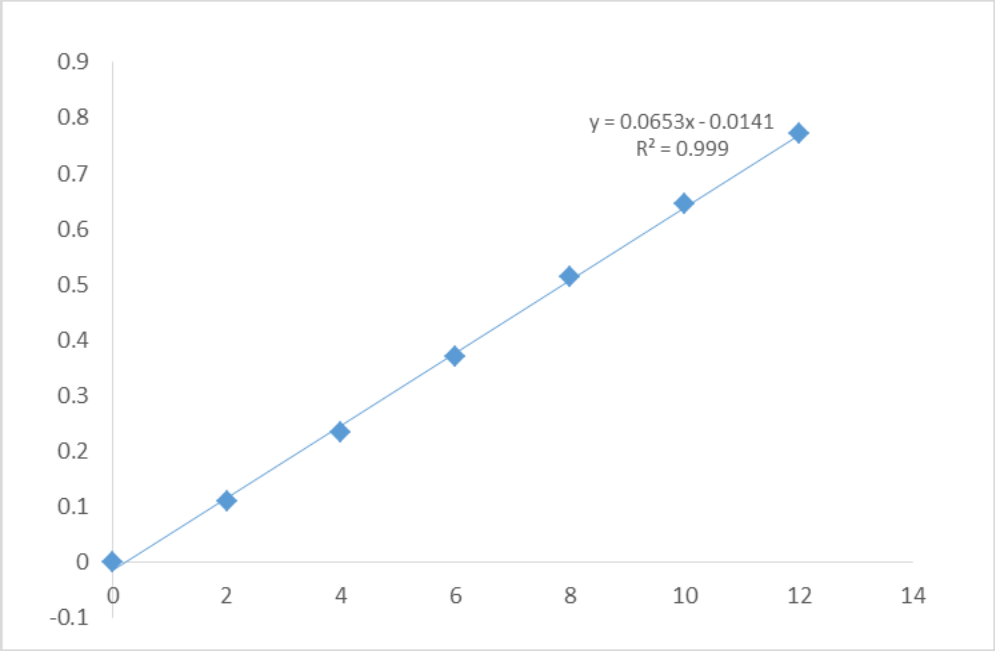

Figure 4: Calibration curve of Bilastine by Zero order derivative spectroscopy

CONCLUSION

The analytical method developed for Bilastine was validated as per ICH guidelines demonstrating simplicity, specificity, accuracy, economy, and sensitivity. This method is suitable for regular analysis of Bilastine in both bulk form and pharmaceutical preparations.

REFERENCE

1. https://en.wikipedia.org/wiki/Bilastine
2. https://www.sciencedirect.com/topics/pharmacology-toxicology-and-pharmaceutical science/bilastine
3. da Silva AT, Brabo GR, Marques ID, Bajerski L, Malesuik MD, Paim CS. UV spectrophotometric method for quantitative determination of Bilastine using experimental design for robustness. Drug Analytical Research, 2017 Dec 28; 1(2): 38-43.
4. Prathyusha P, Sundararajan R. UV spectrophotometric method for determination of Bilastine in bulk and pharmaceutical formulation. Research Journal of Pharmacy and Technology, 2020; 13(2): 933-8.
5. Prajapati P, Tamboli J, Surati P, Mishra A. Risk assessment-based enhanced analytical quality-by-design approach to eco-friendly and economical multicomponent spectrophotometric methods for simultaneous estimation of montelukast sodium and bilastine. Journal of AOAC International, 2021 Sep 1; 104(5): 1453-63.

6. VAIRAGI KP, DESAI DS. Development And Validation Of UV Spectrophotometric Method For The Simultaneous Estimation Of Montelukast Sodium And Bilastine In Their Combined Dosage Form.
7. CHANDOLA A, BHATT M. Method Development And Validation Of Bilastine In Api And Pharmaceutical Dosage Form By Uv Spectrophotometer. International Journal of Pharmaceutical Research (09752366). 2021 Oct 1; 13(4).
8. Roshdy A, Salam RA, Hadad G, Belal F, Elmansi H. Green quality by design HPLC approach for the simultaneous determination of Bilastine and Montelukast. BMC chemistry, 2023 May 2; 17(1): 43.
9. Beltagi AM, Lashin IA, Essa WA, Hathoot AA, Azzem MA. Evolution and effectiveness of HPLC Technique for rapid estimation of an Antiallergenic agent Bilastine. Asian Journal of Pharmaceutical Analysis, 2021; 11(2): 57-62.
10. Heera K. Stability and Degradation Kinetic study of Bilastine in Solution State by RP-HPLC Method. Journal of Drug Delivery & Therapeutics, 2021 Sep 2; 11.
11. Shinde A, Gajeli GB, Ubale S, Matole V. Simultaneous HPLC method development and validation of Bilastine and Montelukast in bulk and formulation. Research Journal of Pharmacy and Technology, 2021; 14(11): 6061-5.
12. Patel DS, Pasha DT. Stability Indicating Isocratic HPLC Method for Bilastine and Characterization of Forced Degradation Products by LC-MS/MS.(2023). Int. J. Life Sci. Pharma Res., 2022; 13(1): P83-93.
13. Pardeshi PJ, Gaware VM, Dhamak KB. Development and Validation of RP-HPLC Method for the Estimation of Bilastine from bulk and Formulation. Asian Journal of Pharmaceutical Analysis, 2020; 10(2): 109-11.
14. Prathyusha P, Sundararajan R, Bhanu P, Mukthinuthalapati MA. A new stability indicating RP-HPLC method for determination of Bilastine in bulk and pharmaceutical formulation. Research Journal of Pharmacy and Technology, 2020; 13(6): 2849-53.
15. Patil CV, Khan S, Sharma R, Patel J, Patel R. Stability indicating rp-hplc method development and validation for simultaneous estimation of bilastine and montelukast in bulk and tablet dosage form. Inter J Pharma Sci Med., 2023; 8(3): 132-49.
16. Martini PR, dos Santos Porto D, Motta PR, Bajerski L, Malesuik MD, Paim CS. Bilastine: stability-indicating a method using environmentally friendly by reversed-phase high-performance liquid chromatography (RP-HPLC). Drug Analytical Research, 2022 Jul 28; 6(1): 13-20.

17. Beltagi AM, Lashin IA, Essa WA, Hathoot AA, Azzem MA. Evolution and effectiveness of HPLC Technique for rapid estimation of an Antiallergenic agent Bilastine. Asian Journal of Pharmaceutical Analysis, 2021; 11(2): 57-62.
18. Pachauri AD, Bhangare SM, Ghode PD, Sayare AS, Deshpande TC, Kakad VD, Aher SM, Khandelwal KR, Ghode SP. RP-HPLC method development and validation for the simultaneous estimation of Bilastine and Montelukast sodium in tablet dosage form. African Journal of Biomedical Research, 2024 Nov 28; 27(3): 302-9.
19. Ahmed A, Manjra Mehfuza U, Mubina L, Nazifa S, Seema P, Khan GJ, Ahamad QM. Development of RP-HPLC Method for Simultaneous Determination of Bilastine and Montelukast by Qbd Approach and Its Validation.(2023). Int. J. Life Sci. Pharma Res., 2023; 13(5): P199-220.
20. Dasari V, Krantikumar P, Godasu SK, Raju P, Vuyyala G. Development and validation of anti-allergic drugs: RP-HPLC. Dogo Rangsang Res J., 2023; 13(3): 97-107.
21. Vyas P, Thakur VS, Basedia D, Dubey B. Development and validation of RP-HPLC based analytical method for simultaneous estimation of montelukast and bilastine in tablet dosage form. Current Research in Pharmaceutical Sciences, 2022 Apr 6: 68-73.
22. Balar YN, Dedania ZR, Dedania RR. A Validated Stability Indicating HPTLC Method for Bilastine and Montelukast in Pharmaceutical Dosage Form. Research Journal of Pharmacy and Technology, 2023; 16(10): 4498-504.
23. Deshpande PB, Pawane AG. Development and Validation of Stability-indicating HPTLC Method for Simultaneous estimation of Montelukast Sodium and Bilastine as Bulk Drugs and in Combined Tablet Formulation.
24. Bhandari D, Modi V, Desai S. Comparison of HPLC-PDA and HPTLC Methods for Simultaneous Estimation of Bilastine and Montelukast Sodium in Marketed Formulation. Pharmaceutical Chemistry Journal., 2025 Feb 24: 1-7.
25. ICHQ2A Text on validation of analytical procedures,https://database.ich.org/sites/default/files/Q2%28R1%29%20Guideline.pdf
26. ICH Q2 (R1) Validation of analytical procedure: Text and methodology, 2005.https://database.ich.org/sites/default/files/Q2%28R1%29%20Guideline.pdf.
27. ICH Q2B: Guidance for Industry: Validation of Analytical Procedures: Methodology U.S Department of Health and Human Services Food and Drug Administration, (CBER), 1996.