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DEVELOPMENT AND VALIDATION OF UV SPECTROPHOTOMETRIC METHOD FOR THE ESTIMATION OF BILASTINE IN BULK AND TABLET DOSAGE FORM

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Abstract:

A Simple, precise, accurate, robust and selective UV Spectrophotometric method has been developed for the estimation of Bilastine from bulk and tablet formulation. This study describes the validation of an UV Spectrophotometric method for quantitative determination of Bilastine in tablets using 0.1 NaOH as solvent. The maximum absorbance was found at 260 nm. The Linearity was found to be in the range of 0.2- 1.0 µg/mL. The % RSD for Intraday precision and Interday precision studies was found to be 0.563 and 0.625 respectively. LOD and LOQ was found to be 1.05 µg/mL and 3.18 µg/mL respectively. The method was applied to marketed formulation and Bilastine content was 99.2 % with respect to labelled claim. The results suggest that this method can be employed for routine analysis of Bilastine in bulk and commercial pharmaceutical formulations.

Key words: *Bilastine, UV Spectrophotometric method, Method development and method validation.*

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INTRODUCTION:

Bilastine is a new second generation H1-antihistamine recently approved for the symptomatic treatment of allergic rhinitis (AR) and chronic urticaria (CUIn CU, the review of the literature indicates that once-daily treatment with Bilastine 20 mg was effective in managing symptoms and improving patient's quality of life. The IUPAC name of Bilastine is 2-[4-(2-{4-[1-(2-Ethoxyethyl)-1H-benzimidazol-2-yl]-1-piperidinyl}ethyl)phenyl]-2-methylpropanoic acid.

Bilastine is an antiallergenic and acts to reduce allergic symptoms such as nasal congestion and urticaria. Bilastine is a selective histamine H1 receptor antagonist (Ki = 64nM). During allergic response mast cells undergo degranulation which releases histamine and other substances[1-2]. Spectrophotometric technique is simple, rapid, moderately specific and applicable to small quantities of compounds. The fundamental law that governs the quantitative spectrophotometric analysis is the Beer - Lambert law[3]. Literature survey reveals some UV methods[4-5], UPLC method[6], RP-HPLC method[7] were reported. [2] The aim of the present work is to develop a simple accurate, precise and economical spectrophotometric method for the estimation of Bilastine in bulk and pharmaceutical formulation and to validate the developed method as per ICH guidelines[8-10]

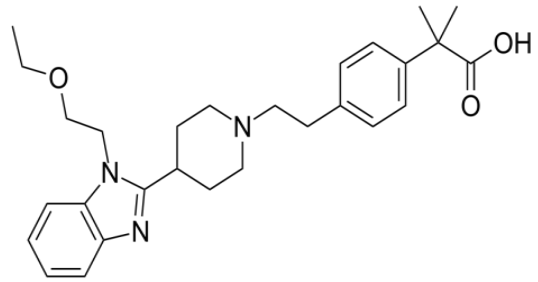

Fig No: 1 Chemical structure of Bilastine

MATERIALS AND METHODS:

Bilastine pure drug was obtained from Arlak Biotech Pharmaceuticals Private Limited, Hyderabad, India. Bilastine tablets (Bilwar 20mg) were purchased from local Pharmacy. UV Visible Spectrophotometer (T-60). Sodium Hydroxide was procured from Finar Labs.

Preparation of drug stock solution

100 mg of Bilastine was weighed and dissolved using few ml 0.1 N NaOH in 100 mL volumetric flask. The volume was made upto the mark with NaOH to produce 1mg/ml (1000µg/mL) solution.

Prepration of bilastine standard solution

Pipette out 10 mL from the above solution was further diluted to 100 mL using to produce 100µg/mL. Transfer 1ml of above solution into a 10 mL volumetric flask and made upto the mark with 0.1N NaOH to produce 10µg/mL of the solution and it was scanned 200-400 nm and absorption maxima **(λ_{max})**was found to be 260 nm.

Determination of Linearity:

Aliquots of 0.2 mL, 0.4 mL, 0.6 mL,0.8 mL and 1mL from standard bilastine solution 10 µg/mL was transferred into a 10 mL volumetric flask and volume was made up with 0.1N NaOH to give concentrations of 0.2µg/mL, 0.4 µg/mL, 0.6 µg/mL, 0.8µg/mL and 1.0 µg/mL respectively and the absorbance was measured at 260 nm against 0.1 N NaOH as blank. A calibration curve was plotted against absorbance and concentration.

Method validation:

Accuracy:

Accuracy of the methods was analyzed by the percentage recovery of the standard drug that was added to the fixed concentration of sample solutions. The study was carried out by adding three different percentage levels of standard drug i.e. 50%, 100%, 150% to the sample concentration. Each solution was prepared in triplicate and the absorbance was noted to find out the percentage recovery.

Precision:

Precision means repeatability of results for a particular method. The absorbance of the 0.6 µg/mL of the bilastine drug solution was measured individually six times within a day and the absorbance was noted (Intraday precision). Similarly, the absorbance of the solutions were individually measured in 6 different days and recorded (Inter day precision). % RSD were reported for repeatability (intraday) and intermediate precision (inter-day).

Robustness:

The robustness study was performed to evaluate the influence of small but deliberate variation in the parameters. The robustness was checked by changing the λ_{max} of the drug.

LOD and LOQ:

The detection limit of an individual's analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. Quantification limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be quantitatively determined with suitable precision and accuracy.

LOD =3.3 Sa/b

LOQ=10 Sa/b

Sa = the standard deviation of the intercept
b = Slope of the calibration curve

Assay: 10 tablets of Bilwar 20 mg were randomly selected and analyzed using the newly developed and validated method. Take the tablets in mortar and pestle to crush in powder. Weight of the tablet powder equivalent to 100 mg was taken and dissolved in 100 mL of 0.1 N NaOH in 100 mL volumetric flask. The volume was made upto the mark with NaOH to produce 1mg/ml (1000µg/mL) solution. Filter the solution using Whatmann filter paper to get clear solution. The above solution was further diluted to give 0.6 µg/mL .Measure the absorbance of the solution six replicates at 260 nm.

RESULTS AND DISCUSSION:

Linearity:

From the graph it was found that Bilastine obeys Beers law and the concentrations lies between 0.2-1.0 µg/mL. The correlation coefficient, intercept and slope were calculated for Bilastine and the result was shown. The linearity data and calibration curve were shown in Table 1.

Table 1: Linearity data of Bilastine.

S.No	Concentration (µg/mL)	Absorbance
1	0.2	0.119
2	0.4	0.289
3	0.6	0.408
4	0.8	0.589
5	1.0	0.783
	Mean	0.7552
	Slope	0.814
	R^2	0.993

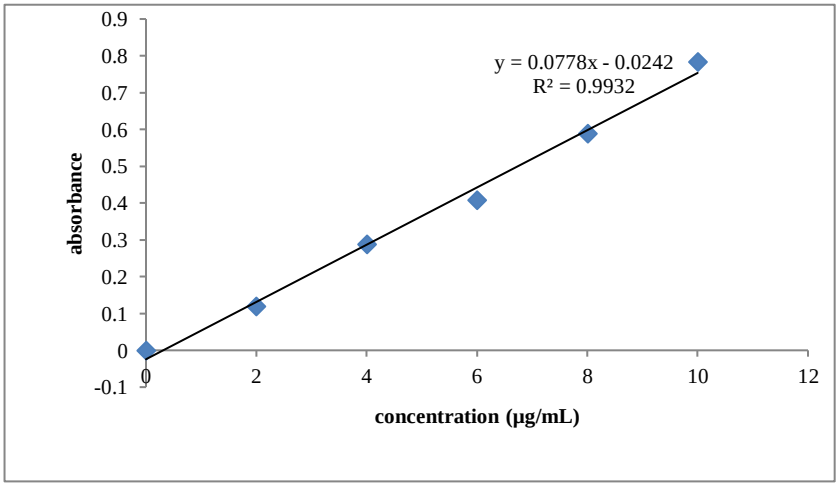

Fig No 2: Calibration Curve of Bilastine

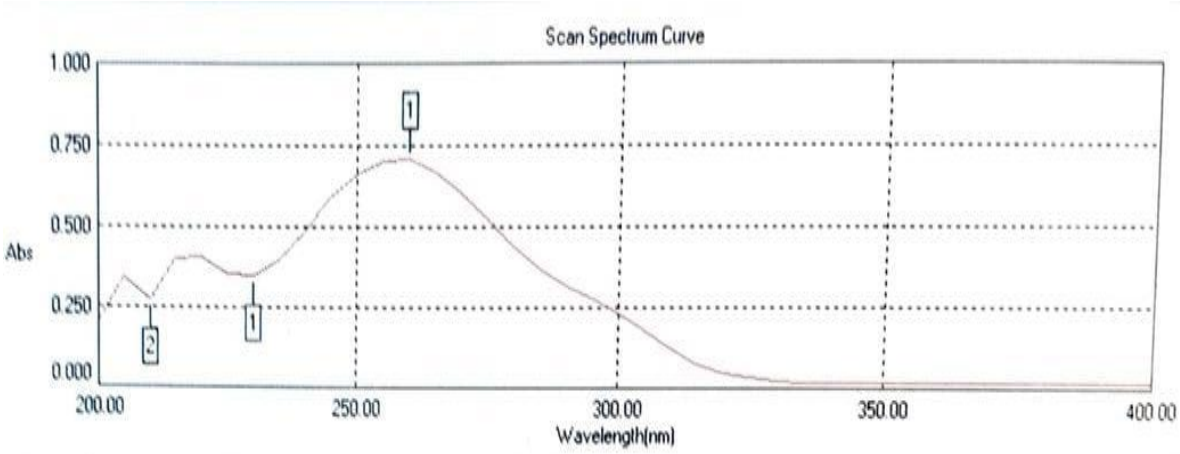

Fig -3: Spectrum Curve of Bilastine

Discussion: Calibration curve was plotted and correlation coefficient was found to be 0.9932. So, there was a good correlation between absorbance and concentration.

PRECISION

Intraday and Interday precision data was shown in Table 2 and 3 respectively.

INTRA-DAY PRECISION:

Table 2: Intra-Day precision of Bilastine

S.N0.	Concentration (µg/mL)	Absorbance-1	Absorbance-2
1	0.6	0.438	0.432
2	0.6	0.435	0.433
3	0.6	0.435	0.41
4	0.6	0.436	0.434
5	0.6	0.434	0.431
6	0.6	0.433	0.431
	Mean	0.435167	0.4285
	STD	0.001722	0.009138
	%RSD	0.395803	2.132517

INTER-DAY PRECISION:

Table 3: Inter-Day precision of Bilastine

S.No	Concentration	Absorbance (Day 1)	Absorbance (Day2)	Absorbance (Day3)
1	6	0.49	0.437	0.43
2	6	0.487	0.439	0.432
3	6	0.485	0.434	0.434
4	6	0.484	0.43	0.436
5	6	0.483	0.431	0.437
6	6	0.483	0.433	0.436
	MEAN	0.4853	0.434	0.4341
	STD	0.0027	0.00346	0.00271
	%RSD	0.563	0.798	0.625

Discussion: The % RSD for Intraday and Interday precision was found to be < 2%. It indicates that the method was precise.

ACCURACY

Recovery studies: Recovery studies were carried out by spiking the samples solution with standard solution at 50%, 100%, and 150% at 3 replicates and data was shown in Table 4.

Table 4: Accuracy data of Bilastine

Sample (%level)	Amount taken (µg/mL)	Amount added (µg/mL)	Amount recovered (µg/mL)	% Recovery	Mean
50	10	3	3.82	99	99
50	10	3	3.93	100	
50	10	3	3.99	98	
100	10	6	6.86	101	100
100	10	6	6.89	100	
100	10	6	6.90	99	
150	10	12	11.76	101.2	100.2
150	10	12	12.74	100	
150	10	12	12.77	99.8	

DISCUSSION: The average % recovery of Bilastine was found to be in between 99-100.2%.

Limit of Detection and Limit of Quantification

LOD and LOQ value for Bilastine was shown in Table 5.

Table 5: LOD and LOQ of Bilastine

Parameters	Bilastine (µg/ml)
LOD	1.05
LOQ	3.18

Discussion: LOD and LOQ value for Bilastine was found to be 1.05 µg/mL and 3.18µg/mL. It indicates that the method was sensitive.

ROBUSTNESS:

Robustness data was shown in Table 6.

Table 6: Robustness data of Bilastine

S.NO	WAVELENGTH(nm)	ABSORBANCE
1	262	0.513
2	260	0.524
3	258	0.555

Discussion: There was no much variation in the absorbance with change in wavelength.

Table- 8: Assay of Bilastine (n=6)

Label claim	Amount found	Assay% ± SD
20 mg	99.2 mg	99.2 % ± 0.05

Discussion: Bilastine was 99.2%, which was comparable with the label claim amount. It shows that UV Visible method developed was successful in determining Bilastine from tablet dosage form.

CONCLUSION:

A new method was developed and validated for the determination of Bilastine using UV spectroscopy. The proposed method was found to be accurate, precise, simple, economic, and rapid. The developed method can be applied for the assay of commercial tablets containing Bilastine in routine quality control analysis. Therefore, the methods can be successfully employed for routine analysis of Bilastine in QC laboratories. So can be used for routine analysis of Bilastine in bulk and its dosage form.

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