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ECO-FRIENDLY UV SPECTROPHOTOMETRIC METHOD FOR THE QUANTIFICATION OF BILASTINE IN BULK DRUG AND PHARMACEUTICAL FORMULATION BY MULTIVARIATE CALIBRATION TECHNIQUE

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ABSTRACT

This research aims to establish an UV-Visible spectroscopic technique for Bilastine by applying a multivariate regression equation that is precise, sensible, and reproducible method. Proposed technique depends on the equation of the linear regression performed by taking absorbance at five distinct wavelengths. Bilastine maximum absorbance was obtained at 274 nm using distilled water as the solvent. Graph obtained from concentration 16-24 µg mL^{-1} resulted in linear curve and the regression coefficient was obtained as 0.9995 %. RSD values for Intra-day, as well as Inter-day precision, was obtained as 0.4817 and 0.3691. The assay value determined was between 99.70 % - 101.46 % w/w.

Keywords: Bilastine, Antihistamine, UV spectrophotometry, Multivariate calibration, Assay, ICH guidelines

INTRODUCTION

Bilastine (BILA) is chemically, 2-[4-[2-[4-[1-(2- ethoxy ethyl) benzimidazole-2-yl] piperidine-1-yl] ethyl] phenyl]-2-methylpropane acid **[1]**. Chemical formula: $C_{28}H_{37}N_3O_3$. Molecular Weight is 463.622 gm./mole. White, solid, crystalline powder, MOA- suppression of immune system responses through histamine's interaction

with the H1 receptor **[2]**. Bilastine is a new H1-receptor antagonist that is nonsedative and can be used to treat the symptoms of chronic idiopathic urticaria **[3]**. Antihistamines have long been the fundamental medication used to treat allergic rhinitis **[4]**. It is a strong, effective, non-sedating antihistamine that doesn't cause cardiac toxicity. It also has great efficacy for the H1, making it equivalent to other antihistamines like cetirizine and astemizole or diphenhydramine, Tmax of 1–1.5 h, Half-life of 10-12h **[5]**. The absorption of bilastine is rapid, consistent, and directly proportionate to the dosage **[6]**. It is beneficial for individuals who have kidney or liver disease **[7]**. The UPLC method was determined to be both stable and sensitive, with no presence of degradation products **[8]**. RP-UFLC **[9]**, LC with Fluorescence detection **[10]**, Near Infrared Spectroscopy method (NIRS) **[11]**, Hydrophilic Interaction Liquid Chromatographic Method (HILIC) **[12]**, RP-HPLC **[13]**, UPLC **[14]**, Quality by Design (QbD) **[15]**. It is official in pharmacopoeias like Indian Pharmacopoeia, British Pharmacopoeia and United States Pharmacopoeia **(Figure 1)**.

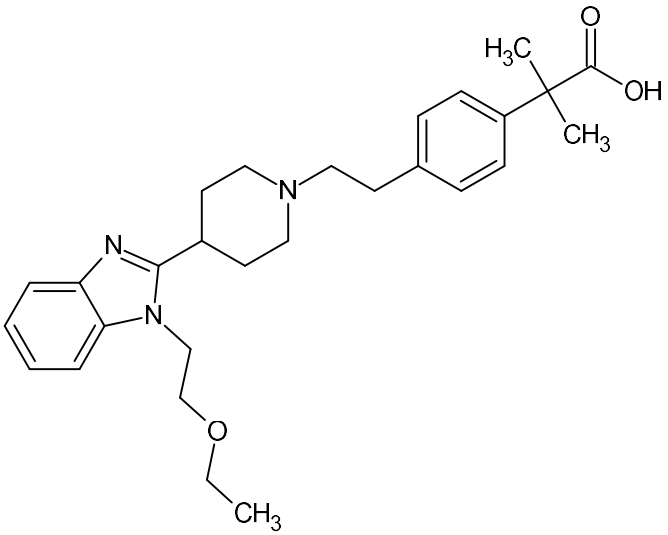

Figure 1: The chemical composition of Bilastine

The proposed approach provides a direct evaluation of Bilastine and has been validated to exhibit greater accuracy and precision compared to a conventional UV-Visible test that can be trusted more. This method can be employed for large quantities of pharmaceuticals and many forms of medication and is more easily understood, rapid, and cost-effective than earlier methods. A multivariate Calibration procedure produced a particular outcome, and the conversion of the outcome yielded a dependent variable, "m." The method delivers excellent sensitivity, resolving power, expediency, and economic analytical efficiency regards determined quantification

of BILA. BILAX (X) refers to the absorbance of an analyte. It requires scanning five distinct concentrations (λ = 240,253,262,274, and 281 nm); for any desired wavelength, the following formula can then be used.

$A_{\lambda 240} = a\ X\ C_x + \ k_1$ ---------- (1)

$A_{\lambda 253} = b\ X\ C_x + \ k_2$ ---------- (2)

$A_{\lambda 262} = c\ X\ C_x + \ k_3$---------- (3)

$A_{\lambda 274} = d\ X\ C_x + \ k_4$---------- (4)

$A_{\lambda 281} = e\ X\ C_x + \ k_5$---------- (5)

The absorbance of the analyte is represented as A λ, the analyte's slope of the linear regression functions are a, b, c, d, and e; The concentration of analyte is denoted as Cx, and the k1, k2, k3, k4, and k5 indicates the intercepts at the specific wavelengths.

The selected five wavelengths equation (1-5) listed above summarised in the following

$$A_T = a\ X\ C_x + \ b\ X\ C_x + \ c\ X\ C_x + d\ X\ C_x + \ e\ X\ C_x + \ K_T$$---------- (6)

Therefore, mentioned equation can be

$A_T = C_x\ (a + b + c + d + e) + \ K_T$---- (7)

The sums of the intercepts from regression equations at a five specific wavelengths are denoted by A_T and K_T, respectively. The concentration of the analyte X is calculated using the formula given below **[16–22]**

$$C_x = \frac{A_T - K_T}{(a+b+c+d+e)}$$ ---------- (8)

Greenness Evaluation Techniques

(GHS) The Globally Harmonized System of Classification and Labelling of Chemicals introduced a series of pictograms accompanied by corresponding signal words, and the analytical environmentally friendly scale is based on the allocation of penalty points, taking into account both the quantity and the number **[23]**. The analytical eco scale technique takes into account the characteristics and amount of each reagent, as well as the potential for occupational exposure, energy consumption, and waste. Penalty points are deducted from a base score of 100.

Total penalty points-100 = Analytical Eco Scale ---------- (9)

(GAPI) The Green Analytical Procedure Index is a graphical representation consisting of five pentagons that are color-coded in a particular manner. The colour scheme in the pictogram corresponds to three degrees of assessment for each stage of an analytical process.

GAPI utilizes a colour scheme that ranges from green to yellow to red in order to indicate different levels of environmental damage associated with the analytical technique. Green represents minimal impact, yellow represents medium impact, and red represents significant impact. In 2018, J. Potka Wasylka delivered a concise summary of GAPI **[24]**.

The third assessment methodology utilizes specialized software from AGREE metrics to evaluate the environmental sustainability profile **[25]**. The software makes a circle with numbers from 1 to 12 going around the

edges. The numbers are arranged clockwise. These pictures show the 12 ideas behind green analytical chemistry. These graphs show the 12 green analytical chemistry, The 12 philosophies' outputs are given a score between 0 and 1 based on the sources and their weights. Red, yellow, and green are used to show different numbers on this collection scale. Any number between 0 and 9 is shown in yellow. A dark green number is close to 1 and to zero is shown in red.

MATERIALS AND METHODS

Chemicals and reagents

- Distilled water
- Bilastine API was ex-gratis from Ideal Analytical Laboratory, Puducherry
- The marketed tablet formulation used was bilahasta manufactured by, Exemed pharmaceuticals (Labelled to have 20 mg bilastine) purchased from a nearby market.

Instrumentation

- Double beam UV spectrophotometer (LAB INDIA 3092)
- The Ultra Sonicator
- Micro balance
- The Micropipette

Analytical method development

Solvent selection: Bilastine has been found to be readily soluble in distilled water. Therefore, both the standard and the sample were further diluted using distilled water.

Standard stock solution

A 25 mL volumetric flask containing 25 mg of the standard medication is diluted with distilled water to create the stock solution of bilastine. Aliquots of this solution with concentrations, (16-24 μg mL^{-1}) were prepared and utilized for further analysis.

Determination of λ_{max}

Bilastine maximum absorbance is established using a solution made by dissolving the standard stock solution in water to a concentration of 20 μg mL^{-1}. The prepared solution was scanned in the UV-visible wavelength range 200 and 400 nm. Obtained graph plot between the concentrations against absorbance gives a linear curve. The outcomes are examined around the spectrum range 274 nm, i.e., 240, 253, 262, 274, and 281 nm, for improving correlation and diminishing the oscillations of the instrument.

Sample solution preparation

Preparation of sample solution is done by taking twenty tablets of Bilastine, precisely weighed and powdered. A 25 mL standard flask was filled with the weight equivalent to 25 mg, sonicated for 15 minutes, the sample was dissolved, and distilled water was added to make up the volume. The solution was subsequently filtered and utilized for additional testing.

Method Validation

This method's sensitivity, precision, accuracy, and linearity have been validated in accordance with the ICH Guidelines **[26]**.

Linearity

Different concentrations ranging from 16-24 µg mL^{-1} were made using standard stock solutions. To minimize instrumental variations and enhance the correlation, these solutions were assessed across a range of wavelengths: 240, 253, 262, 274, and 281 nm. **(Figure 3, Table 1).**

Table 1: UV Calibration data at five distinct wavelengths

Concentration (µg mL^{-1})	Absorbance*				
	240 nm	253 nm	262 nm	274 nm	281 nm
16	0.261	0.326	0.258	0.336	0.315
18	0.307	0.375	0.301	0.382	0.360
20	0.352	0.423	0.343	0.428	0.405
22	0.407	0.472	0.386	0.474	0.450
24	0.451	0.521	0.428	0.520	0.494

#Average of 5 determinations; UV= Ultra violet

The graph is plotted as concentration against absorbance and standardizations were achieved. The technique's sensitivity was determined by LOD and LOQ using the following formula.

$LOD = 3.3\ \sigma/S$ …….... (8)

$LOQ = 10\ \sigma\ /S$..... (9)

Hereby, lowermost concentration of standard deviation (SD) and the standard curve of the slope is denoted as S.

Precision

In order to determine the intra-day and inter-day precision, a 20 µg mL^{-1}. The solution was replicated and digitized six times. Six different days were used to measure the intra-day precision and within the same day to measure the inter-day precision.

Accuracy

The recovery study for recommended technique were determined at 80%, 100%, and 120% by the standard addition technique, and using this % recovery was calculated. The solutions for the recovery study were prepared from both standard and sample stock solutions.

Assay

By measuring the absorbance at 274 nm from the extracted tablet solution, the amount of bilastine present in the tablet was determined.

RESULTS AND DISCUSSION

As seen in **Figure 2**, the Bilastine maximum absorbance was measured at 274 nm using water as solvent.

Within the concentration range between 16-24 µg mL^{-1} this technique was found to be linear. An excellent linear correlation is obtained from the calibration plots with R^2 - 0.9988- 0.9999. The % relative standard deviation for precision obtained as 0.48171 and 0.3691. The LOD and LOQ were at 0.18148 and 0.54996 µg mL^{-1}. Hence the

values come under validation parameters accordance to the ICH guidelines limitations.

Linearity

The linearity spectra are depicted in **Figure 3,** and the corresponding calibration curves are shown from **Figure 4 to 8.** The approach is deemed accurate and dependable for every wavelength, as indicated by the low values of the RSD. The calculation of detection and quantification of limit has been done and results were depicted in **Table 2**.

Precision

The suggested technique is distinct, dependable, and accurate, as evidenced from the low standard deviation values. The intra-day precision and inter-day precision values are, 0.48171 and 0.3691respectively. It is within tolerances of less than 2% at every wavelength (**Figure 9, 10**).

Recovery

The Bilastine % recovery was found between 99.70% to 101.46% w/w, according to the ICH guidelines. The acceptable range of % recovery was from 97-103% w/w **(Figure 11, Table 3)**.

Assay:

The maximum absorbance of bilastine was measured at 274 nm for the tablet formulation by UV-Visible spectroscopy. The amount and assay percentages were obtained as 20 mg and 99.88 % w/w, further % Relative standard deviation value is depicted in **Table 4**.

Evaluation of Greenness Profile

The final output of the greenness profile for the suggested methods was done. Analytical scale of result is shown in **Table 5**.

The outcome results of AGREE and GAPI are shown in **Figure 12 and Figure 13**.

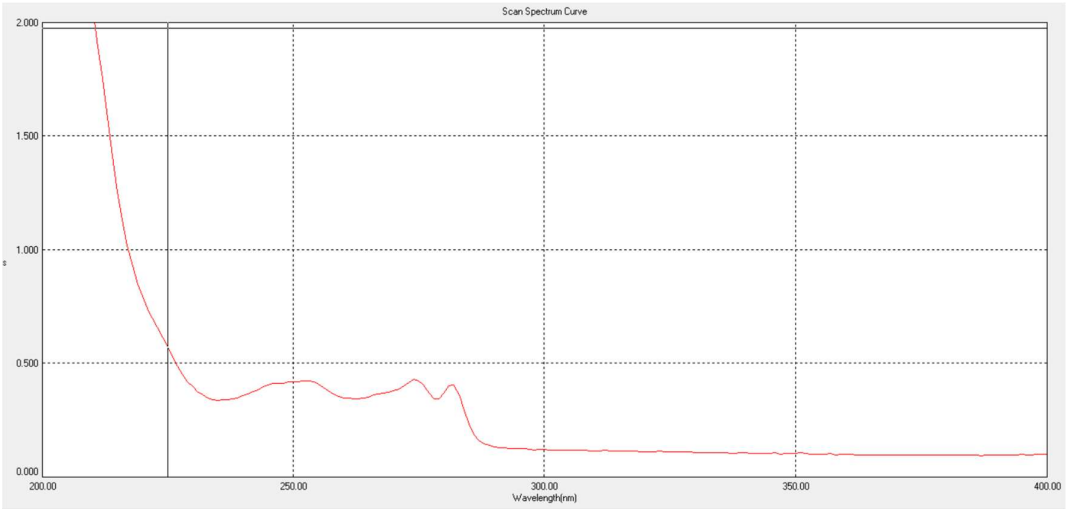

Figure 2: UV spectrum of Bilastine (20 µg mL^{-1}), λ_{max} at 274 nm

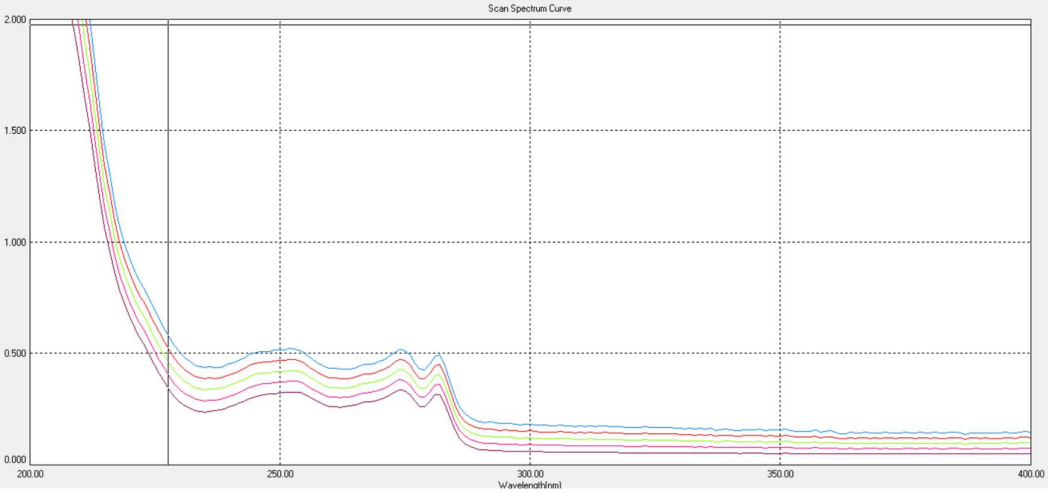

Figure 3: UV Spectrum of Bilastine showing linearity at 281 nm

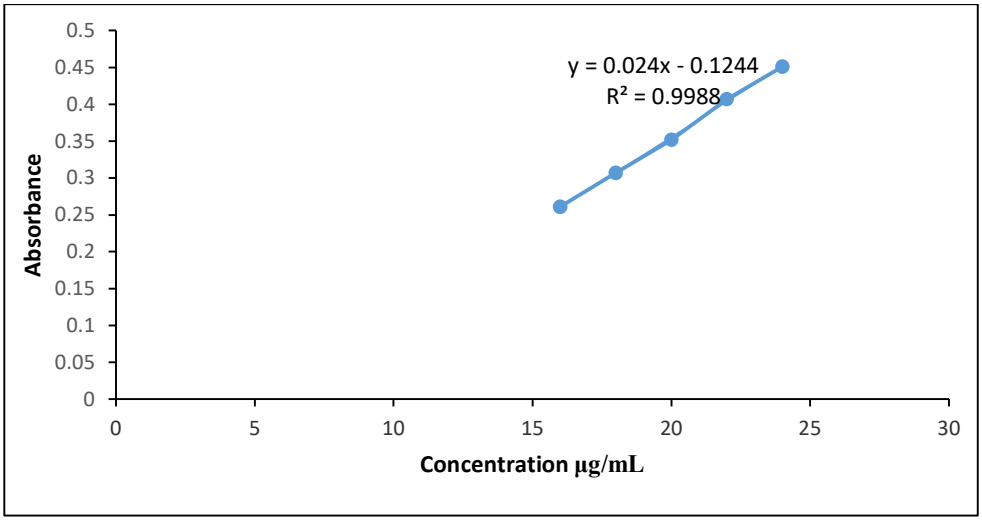

Figure 4: Calibration curve at 240 nm

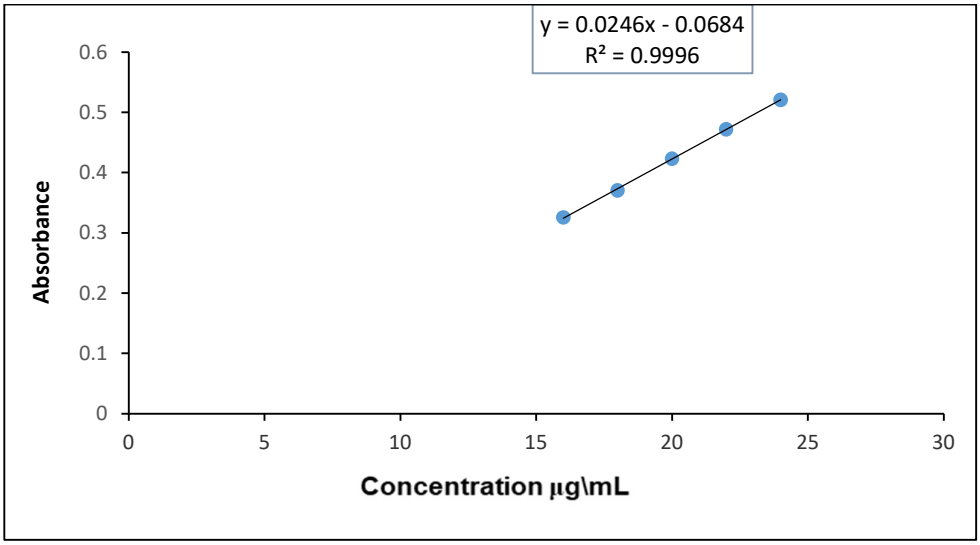

Figure 5: Calibration curve at 253 nm

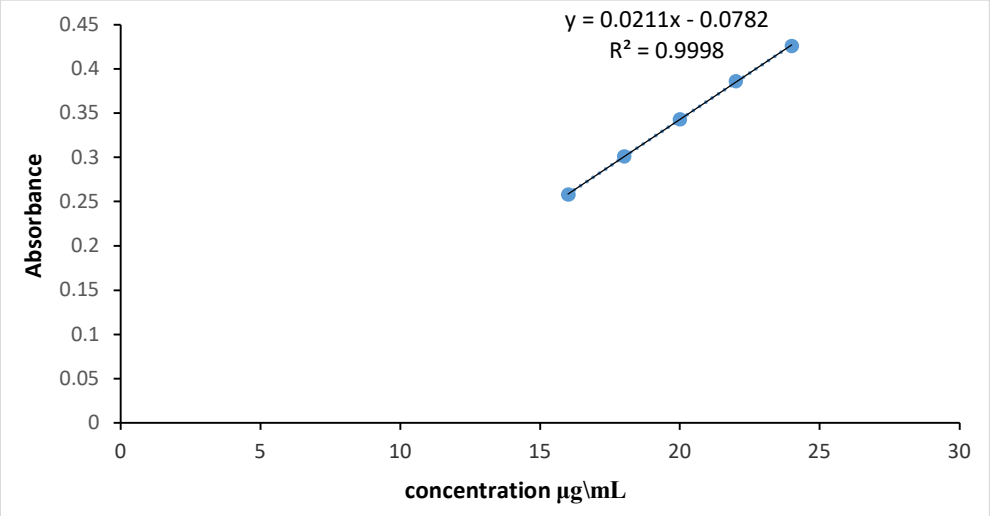

Figure 6: Calibration curve at 262 nm

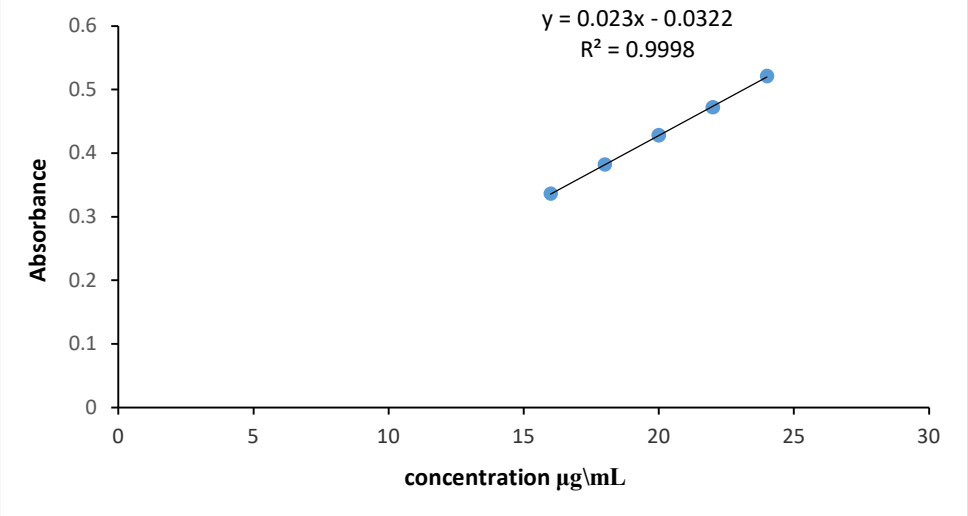

Figure 7: Calibration curve at 274 nm

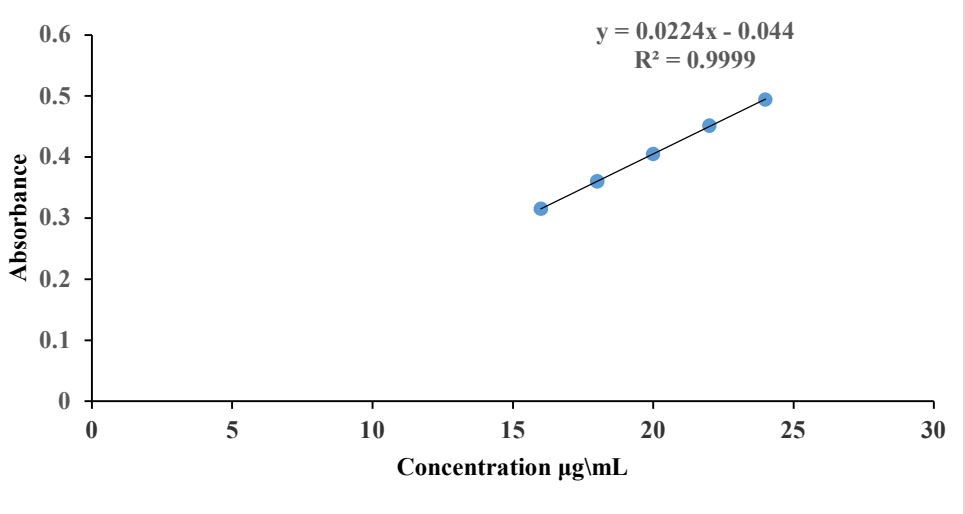

Figure 8: Calibration curve at 281 nm

Table 2: Linearity data with LOD and LOQ at selected five wavelengths

Wavelength (nm)	Regression equation	R^2	LOD (µg mL^{-1})	LOQ (µg mL^{-1})	% RSD
240	y = 0.024x - 0.1244	0.9988	0.4140	1.2546	0.8467
253	y = 0.0246x - 0.0684	0.9996	0.2341	0.7094	0.4121
262	y =0.0211x - 0.0782	0.9998	0.1487	0.4506	0.2767
274	y =0.023x - 0.0322	0.9998	0.1814	0.5499	0.2956
281	y =0.0224x - 0.044	0.9999	0.1169	0.3544	0.1964

*nm = nanometre; µg mL^{-1}= Microgram per millilitre

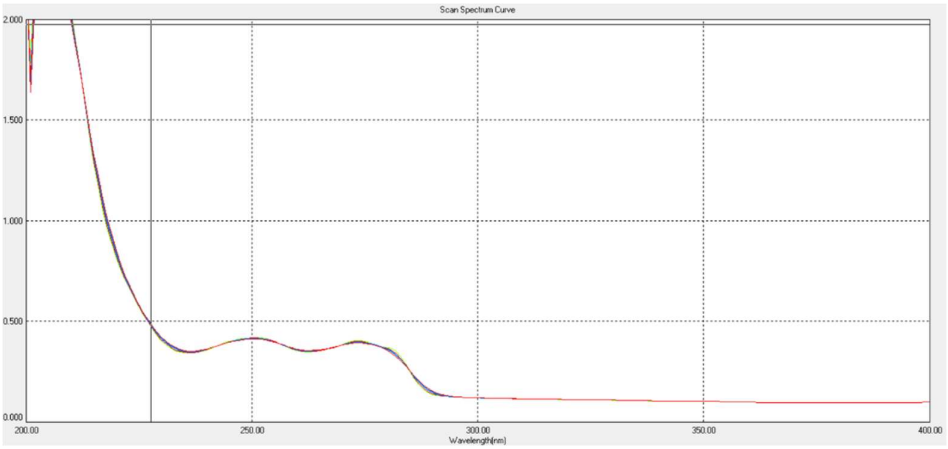

Figure 9: UV spectra showing intraday precision

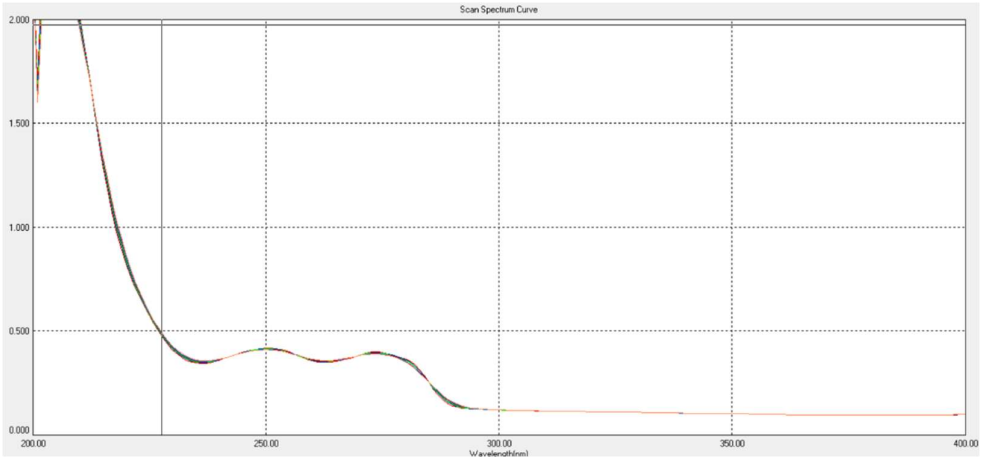

Figure 10: UV spectra showing interday precision

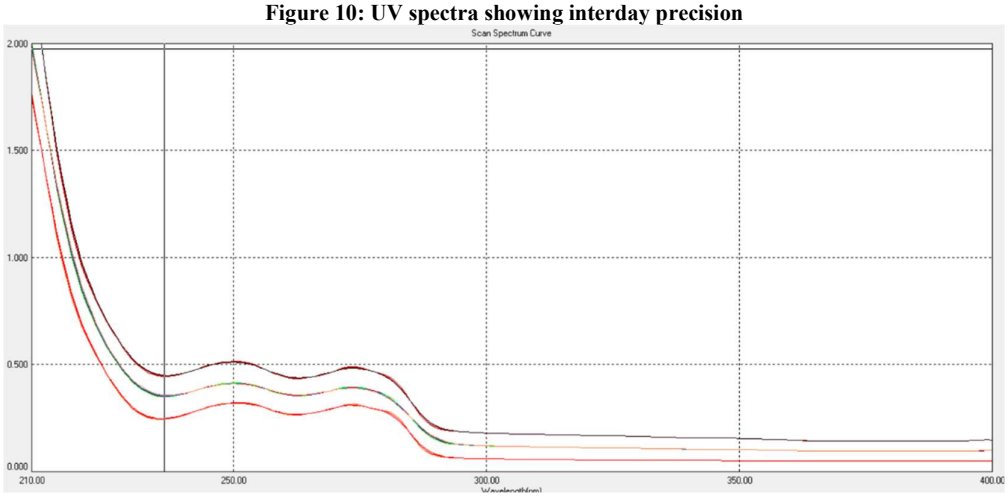

Figure 11: UV spectra showing recovery studies

Table 3: Recovery Studies

Wavelength (nm)	Amount present (µg mL^{-1})	Amount added (µg mL^{-1})	Absorbance	Amount recovered (µg mL^{-1})	% Recovery
240 nm	10	6	0.336	16.01	100.06
		10	0.428	19.95	99.75
		14	0.521	24.35	101.46
253 nm	10	6	0.338	16.02	100.13
		10	0.426	19.96	99.80
		14	0.523	24.34	101.42
262 nm	10	6	0.339	16.03	100.19
		10	0.429	19.97	99.85
		14	0.525	24.31	101.29
274 nm	10	6	0.334	16.05	100.31
		10	0.426	19.95	99.75
		14	0.519	24.35	101.46
281 nm	10	6	0.335	16.04	100.25
		10	0.427	19.94	99.70
		14	0.518	24.341	101.42

Table 4: Assay of Bilastine

Label claim (mg)	Amount obtained (mg)	% Assay
20	19.98	99.90
20	19.81	99.05
20	20.14	100.70
Average	19.98	99.88
SD		0.8251
% RSD		0.8261

Table 5: Summary of Eco scale penalty points for the proposed method

Description	Penalty points	Total penalty Points	Score
Distilled water	0	0	100
Instrument	0		
Occupational hazard	0		
Waste	0		

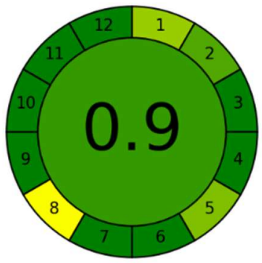

Figure 12: Agree metrics output for the proposed method

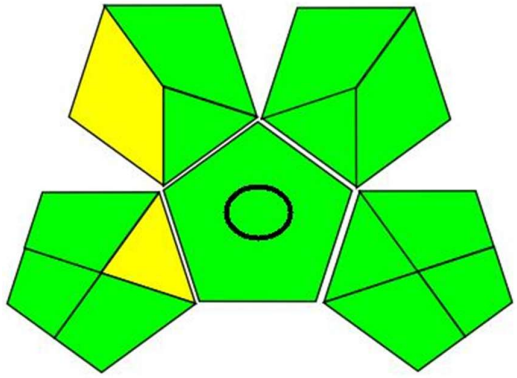

Figure 13: GAPI Pictogram for the proposed method

CONCLUSION:

Comparing the suggested method against the traditional UV-Visible Spectrophotometry for Bilastine assay, it is more exact, accurate, repeatable, economical. It is evident from the greenness assessment tools that the method is ecofriendly. Bilastine standard drug and tablet dosage form of Bilastine quantified by the multivariate regression equation. According to the Quality Guidelines of ICH, this method has been validated and they are inside the range of the limits. This method was found simple and ecofriendly than complicated HPLC and HPTLC methods and it is used for the analysis of the sample of Bilastine bulk drugs and pharmaceutical dosage forms.

ETHICAL STATEMENT

This study does not involve experiments on animals or human subjects.

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CONFLICT OF INTEREST

No potential conflict of interest relevant to this article exists.

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