Original Article

Application of UV spectrophotometric method for easy and rapid estimation of lafutidine in bulk and pharmaceutical formulation

Abstract

Introduction: The present research work discusses the development of a UV estimation method for lafutidine. Simple, accurate, cost efficient, and reproducible spectrophotometric method has been developed for the estimation of Lafutidine in bulk and pharmaceutical dosage form. **Materials and Methods:** The Stock solution was prepared in a mixture of water and methanol (1:1). Further dilutions were made in water. **Results:** The drug was determined at maximum wavelength (λ_{max}) 279 nm. Beers law was obeyed in the concentration range of 10–50 μg/ml having line equation y = 0.0100x + 0.035 with correlation coefficient of 0.999. Results of the analysis were validated statistically and by recovery study. **Conclusion:** The result of analysis was validated as per ICH guidelines and this method can be used for the routine analysis of lafutidine formulation.

Key words: Lafutidine, UV spectrophotometry, validation

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INTRODUCTION

Chemically, lafutidine is 2-[(2-furyl methyl) sulfinyl]-*N*-(2z-4-{[4-(piperridin-1yl methyl) pyridin-2-yl] oxy} but-2-en-1-yl) acetamide [Figure 1].[1] It is a H_2 receptor antagonist and is reported to show potent and long lasting antagonisms of histamine H_2 receptor mediated effect. It is effective agonist the esophageal lesions induced by acid reflux through inhibition of acid secretion.[2] Analysis is an important component in the formulation development of any drug molecule. It becomes essential to develop a simple, sensitive, accurate, precise, reproducible method for the estimation of drug samples. Our main concern is development and validation[1] of UV spectrophotometric method as per ICH guideline.[3]

Earlier publications have described HPLC[4] and LC–MS[5] methods for quantification of lafutidine in human plasma and pharmaceutical dosage form. However, these methods involve arduous sample preparation and long chromatographic rum times for biological samples.

So far to our present knowledge, no UV spectrophotometric analytical method is available in literature for analyzing lafutidine in pharmaceutical dosage form or as bulk drug sample. It was felt necessary to develop a simple, precise and rapid method for quantitative determination of lafutidine. The current research work deals with the development of spectrophotometric method and its validation as per International Conference on Harmonisation (ICH) guidelines.[3]

MATERIALS AND METHODS

Experimental

Instrument and materials

Instrument used were Schimadzu 1800 double beam UV/Visible Spectrophotometer and schimadzu 1600 analytical balance. Lafutidine pure drug was obtain from

Ajanta Pharmaceuticals Ltd., Mumbai, India as gift sample with 99.9% (w/w) assay value and was used without further purification. All chemicals and reagents used were of analytical grade.

Methodology

Preparation of standard stock solution

Standard drug solution of lafutidine was prepared by dissolving 10 mg lafutidine in 5 ml methanol this solution was transferred it to 10 ml volumetric flask and volume was made up to mark with distilled water to obtain stock solution of 1 mg/ml concentration.

Preparation of calibration curve

Aliquots of 1–5 ml portion of stock solutions were transferred to series of 100 ml volumetric flasks, and volume made up to mark with distilled water. Solutions were scanned in the range of 200–400 nm against blank. The absorption maxima were found to be at 279 nm against blank. The calibration curve was plotted. The optical characteristics are summarized in Table 1.

Preparation of sample solution

The proposed method was applied to analyze commercially available lafutidine tablet. Ten tablets were weighed and powdered. The amount of tablet powder equivalent to 10 mg of lafutidine was weighed accurately and transfer to 10 ml volumetric flask then 5 ml methanol was added and kept for 15 min with frequent shaking and volume was made up to mark with distilled water. The solution was then filtered through Whattman filter paper #41. This filtrate was diluted suitably with distilled water to get the solution of 10 µg/ ml concentration. The absorbance was measured against solution blank. The drug content of the preparation was calculated using standard calibration curve. Amount of drug estimated by this method in Table 2.

Table 1: Calibration curve parameter

Concentration (µg/ml)	Absorbance (average)
10	0.139
20	0.231
30	0.335
40	0.438
50	0.537
60	0.636

RESULT AND DISCUSSION

Linearity

The linearity of the response of the drug was verified at 10–50 µg/ml concentrations. The calibration curve was obtained by plotting the absorbance versus the concentration data and was treated by linear regression analysis [Table 3]. The equation of the calibration curve for lafutidine obtained was y = 0.0100x + 0.035, the calibration curve was found to be linear in the aforementioned concentrations (the correlation coefficient (r^2) of determination was 0.999) [Figure 2 and 3].

Precision

Assay of method precision (intraday precision) was evaluated by carrying out six independent assays of test samples of lafutidine. The intermediate precision (interday precision) of the method was also evaluated using two different analysts, systems and different days in the same laboratory. The relative standard deviation (RSD) and assay values obtained by two analysts were 0.28, 99.67, and 0.26, 99.68, respectively [Table 4].

Accuracy (recovery test)

Accuracy of the method was studied by recovery experiments. The recovery experiments were performed by adding known amounts of the drugs in powdered tablets. The recovery was performed at three levels, 80, 100, and 120% of lafutidine standard concentration. The recovery samples were prepared in afore mentioned procedure. Three samples were prepared for each recovery level. The solutions were then analyzed, and the percentage recoveries were calculated from the calibration curve. The recovery values for lafutidine ranged from 100.1 ± 0.07611% [Table 2, Figure 4].

Limit of detection and limit of quantification

The Limit of detection (LOD) and limit of quantification (LOQ) of lafutidine were determined by using standard deviation of the response and slope approach as defined in ICH guidelines. The LOD and LOQ for lafutidine are described in Table 3.

Table 2: Determination of accuracy by percentage recovery method

Ingredient	Tablet amount[a] (µg/ml)	Level of addition (%)	Amount added[a] (µg/ml)	Total amount taken from tablet[a] (µg/ml)	Amount recovered (µg/ml)	%Recovery	Average %recovery
Lafutidine	10.00	80	8.4	18.4	18.34	99.67	100.1 ± 0.07611
	10.00	100	10.2	20.20	20.13	99.65	
	10.00	120	12.23	22.23	22.45	100.98	

[a]Amount equivalent to pure drug

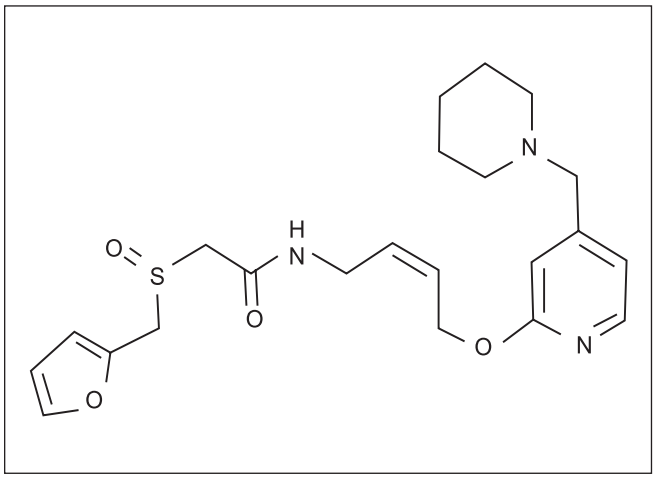

Figure 1: Chemical structure of lafutidine

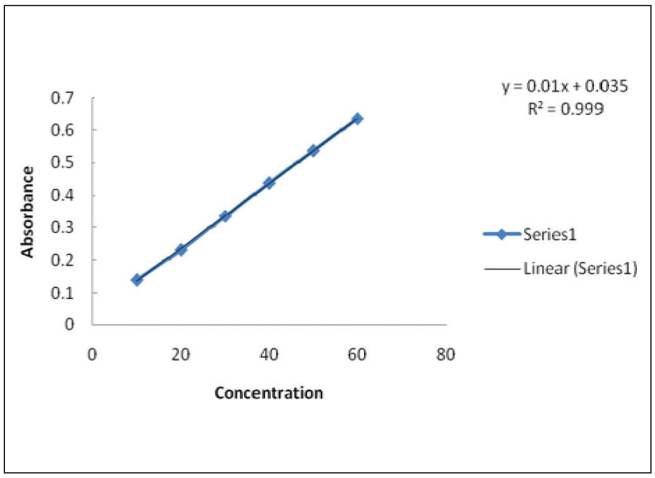

Figure 2: Calibration curve

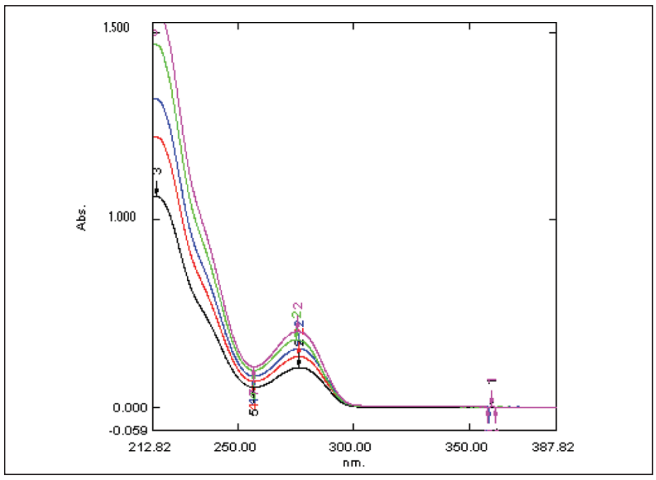

Figure 3: Overlay figure of linearity ranges of lafutidine (10–50 µg/ml)

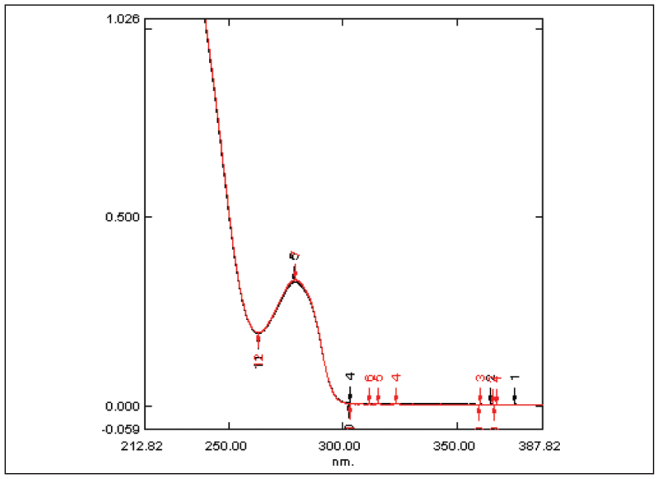

Figure 4: Overlay UV spectra of lafutidine standard and test tablet from 200-400 nm

Table 3: Validation parameters

Parameter	Result
Absorption maxima(nm)	279
Linearity range (µg/ml)	10–50
Standard regression equation	y = 0.0100x + 0.035
Correlation coefficient (r^2)	0. 999
Molar absorptivity	80000 M^{-1} cm^{-1}
A (1%, 1 cm)	1856.14
Accuracy (% recovery ± SD)	99.998
Precision (% CV)	0.5684
Limit of quantitation (µg/ml)	9.95
Limit of detection (µg/ml)	3.28

Table 4: Determination of precision

Sample number	Assay of lafutidine as % of labeled amount	
	Analyst-I (intraday precision)	Analyst-II (interday precision)
1	99.72	99.77
2	99.93	99.97
3	99.78	99.71
4	99.90	99.88
5	99.48	99.51
6	99.20	99.25
Mean	99.67	99.68
SD	0.28	0.26

Determination of active ingredients in tablets

The validated method was applied for the determination of lafutidine in tablets (six tablets were assayed and the results in Table 2 indicate that the amount of drug in tablet samples met with requirements (98%–102% of the label claim).

CONCLUSION

The developed UV spectrophotometric method was found to be rapid, simple, inexpensive, reproducible, and applicable over a wide concentration range with high precision and accuracy. The method was validated as per the guidelines laid by ICH. The results of the validated tests were found to be satisfactory and therefore this method can be applied successfully for routine quality control analysis of lafutidine in bulk and pharmaceutical formulation.

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