Development and Validation of UV Spectrophotometric Method for Determination of Eletriptan HBr

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ABSTRACT: A rapid and sensitive UV Spectrophotometric method for routine evaluation of eletriptan in tablet was developed and validated as per ICH guidelines. UV Spectrophotometric method was performed at 270nm and samples were prepared with a solution of phosphate buffer. The linearity demonstrated a correlation coefficient of 0.999951 various validation parameters like accuracy, precision, LOD, LOQ, recovery study and range were determined. The proposed method was simple, rapid, precise, accurate and sensitive and can be used for routine analysis of eletriptan hydro bromide in bulk and combined dosage form. © 2020 iGlobal Research and Publishing Foundation. All rights reserved.

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INTRODUCTION

Eletriptan chemically designated as 3-[[(R)-1-Methyl-2-pyrrolidinyl] methyl]-5-[2-(phenylsulfonyl) ethyl] indole, monohydrobromide, is selective 5-hydroxytryptamine 1B/1D (5-HT1B/1D) structure as depicted in **figure 1** is a 5-HT 1 receptor agonist. Eletriptan HBr is generally used in the treatment of the migraine headache, because the eletriptan HBr activate the 5-HT1 receptors located on intracranial blood vessels, including those on the arteriovenous anastomoses, leads to vasoconstriction, which is correlated with the relief of migraine headache [1-2].

The UV spectrophotometric method is very simple, rapid, economical, and it allows the determination in pharmaceuticals with enough reliability. For the UV spectrophotometric method, the survey of literature revealed very complex methods, using bands of the visible which have used complexometry, derivative or chemometric assistance and interpolation on the calibration curve [2-3]. The aim of this work was the development and fully validation of a new UV spectrophotometric method, which can be more economical and simpler than the official methods and with other methods published. The UV spectrophotometric method

is simpler than the others studied because it does not need derivative and chemometric assistance. Moreover, this method can be used in dissolution studies because it uses its own dissolution medium as diluent [2-3].
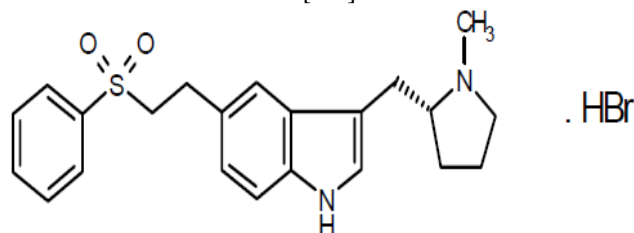

Figure 1. Structure of Eletriptan HBr

Analysis is the most important aspect of any drug development whether in bulk or in combination, a suitable method must be developed so as to ensure that any drug either in dosage form or bulk form can be pointed out. The method development ensures that amount of particular drug can be easily determined. The validation parameters confirm that the developed method is precise, accurate and reproducible and can be used for routine evaluation of eletriptan in bulk and combined dosage form [2-4].

MATERIALS AND METHODS

UV Spectroscopic Method

Instrumentation

A UV-Visible Spectrophotometer (UV-1700 SHIMADZU) with 10mm matched quartz cells was used for Spectrophotometric method. All weighing were done on electronic balance (Model Shimadzu AUW-220D).

Reagents & chemicals

Eletriptan HBr was received as gift sample from WOKHARDT Research Centre, Aurangabad. Tablet formulation manufactured by Pfizer limited was purchased from local market RELPAX containing eletriptan hydro bromide 40mg per tablet [5].

Preparation of standard stock solution

Standard drug solution of metoprolol succinate was prepared by accurately weighing 10 mg of the drug, and dissolved in phosphate buffer pH 6.8 and the volume was made up to 100ml to obtain stock solution (100 µg/ml) [5-6].

Determination of Analytical Wavelength

From the standard stock solution 1ml was pipette out into 10ml volumetric flask. The volume was made up to 10ml with phosphate buffer pH 6.8. The resulting solution containing 10µg/ml was scanned between 200-400 nm [5-6].

Preparation of Calibration Curve

Aliquots of 1 to 6ml portions of stock solutions were transferred to a series of 10ml volumetric flasks, and volume made up to the mark with phosphate buffer pH 6.8. The serial dilutions in the range of 10, 20, 30, 40, 50, and 60µg/ml were prepared. The absorbance was measured at λmax 270nm [5-9].

UV Method Validation

Linearity & Range

The linearity of the response of the drug was verified at 10 to 60µg/ml concentrations. The calibration curve was obtained by plotting the absorbance versus the concentration data and was treated by linear regression analysis. The equation of the calibration curve for metoprolol was obtained [5-9].

Precision

The accuracy of the method was determined by recovery experiments. Each solution was repeated in triplicate and the percentage recovery was calculated. The precision of the method was demonstrated by intra-day and inter-day variation studies [5-9].

Limit of Detection (LOD) and Limit of Quantification (LOQ)

LOD and LOQ were calculated by the equations;

LOD = 3.3σ/ S and LOQ = 10σ/S

Where S is the slope of the calibration curve and σ is the residual standard deviation.

Recovery Study

Accuracy of the method was studied by recovery experiments. The recovery was performed at three levels, 80, 100, and 120% of metoprolol standard concentration. The recovery samples were prepared in a before mentioned procedure for each recovery level. The solutions were then analyzed and the percentage recoveries were calculated from the calibration curve [5-11].

RESULTS AND DISCUSSION

Analytical Wavelength

The maximum absorption was found to be at the wavelength of 270nm hence the wavelength for Eletriptan was found to be 270 nm as shown in **figure 2**.
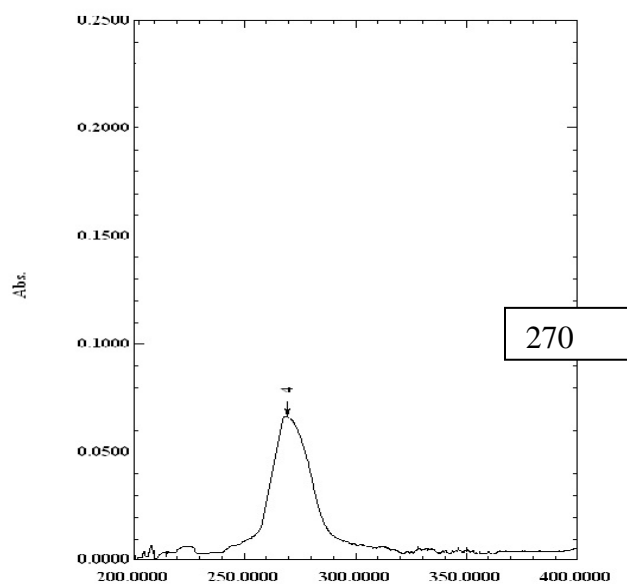

Figure 2. A typical UV Spectrum of Eletriptan 270 nm

Calibration Curve

The results of absorbance for all the prepared concentrations were plotted i.e. Concentration vs. Absorbance the method was found to be linear over the prepared concentration range with the standard equation y=0.0082x-0.0037 and Regression value was found to be 0.9999, as shown in **figure 3**. From the calibration data obtained it was found that the regression coefficient was less than 1 which is within the limits of Beer lamberts' law.
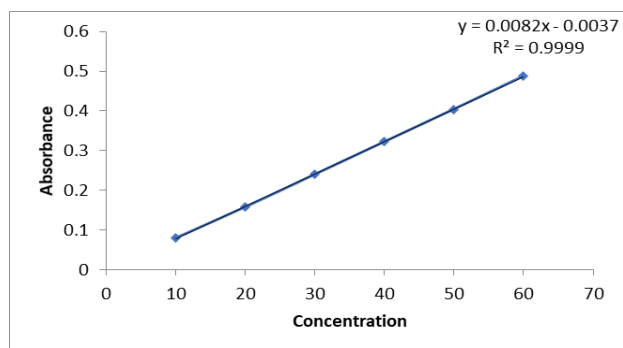
Figure 3. Calibration Curve of Eletriptan HBr

Precision

Precision of the method was evaluated for Eletriptan HBr. The reproducibility (inter-day precision) of the method and repeatability (intra-day precision) was evaluated in the same laboratory. The values obtained were 0.341124 and 0.329457 respectively (**Table 1**). From the data obtained in Table 01 the method was found to be precise in respect of reproducibility as well as repeatability.

Table 1. Precision determinations

Precision	Intra-Day Precision*	Inter-Day Precision*
Result	0.329457	0.341124

Accuracy (Recovery Study)

Accuracy of the method was studied by recovery experiments. The recovery was performed at three levels 80, 100 and 120% of eletriptan standard concentration. 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 value for eletriptan was 99.9995 ± 0.052 and RSD was 0.05221 which is less than 2, which shows that the method has good reproducibility (**Table 2**).

Limit of detection (LOD) and limit of quantification (LOQ)

Limit of detection is the lowest amount of analyte which can be detected but not necessarily quantified, and limit of quantification is the lowest possible concentration that can be quantified LOD and LOQ were found to be 0.246089 mcg/ ml & 0.745723 mcg/ml respectively, lower value indicate that the 0.246089 mcg/ ml quantity can be detected in bulk or combine form for Eletriptan & 0.745723 mcg/ml can be quantified in bulk or combine dosage form.

Specificity

Specificity is the ability of the method to accurately measure the analyte response in the presence of all potential sample components (excipients). The results were compared with the analysis of a standard Eletriptan and tablet formulations. Excipients of the solid dosage form did not interfere with the analyte, which shows that the method has good specificity.

Validation parameters

All the validation parameters as reported in **table 3** were found to be within the desired range which depicts that the method was found to be reproducible with respect to all the validation parameters and can be a useful tool for routine evaluation of eletriptan in bulk and combined dosage form.

Table 2: Recovery Study

Formulation stock	Total conc.	Drug Recovered	% Recovery	Mean % Recovery	SD	%RSD
32	72	72	100	100.041	0.08448	0.084447
32	72	72.1	100.138			
32	72	71.99	99.986			
40	80	80	100	99.995	0.007216	0.007217
40	80	79.99	99.987			
40	80	80	100			
48	88	87.90	99.9887	99.9625	0.0649519	0.0649763
48	88	88	100			
48	88	88	100			
			Mean	99.9995	0.0522159	0.05221343

Table 03: Validation parameters

Parameter	Result
Linearity range	10-60mcg/ml
Regression eq.	y=0.0082x-0.0037
Correlation coeficient	0.999951
Slope (m)	0.00812
Avg % RSD	0.364392
SD Average	0.000606
λmax	270 nm
LOD	0.246089
LOQ	0.745723
Molar absorptivity	2379.32
Sandells sensitivity	0.125
Interday precision	0.341124
Intraday precision	0.329457

CONCLUSION

A suitable UV Spectrophotometric method was developed and validated as per ICH guidelines for the determination of Eletriptan HBr in dosage formulations. It was shown above that, the proposed method was linear, accurate, reproducible, repeatable, precise, selective, specific and cost effective proving the reliability of the method. More over same solvent was used throughout the experimental work and it was found that no interference from any excipients was observed in method. Hence, the proposed method was successfully applied to routine analysis of Eletriptan HBr in bulk and combined formulations.

ACKNOWLEDGEMENT

DATA AVAILABILITY

We declare that data supporting in the findings is present in the manuscript can be made available to the public.

CONFLICT OF INTEREST

The authors have no conflicts of interest.

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