Pharm Methods, 2018; 9(1): 30-33
A multifaceted peer reviewed journal in the field of Pharm Analysis and Pharmaceutics
www.phmethods.net | www.journalonweb.com/phm

Original Article

Simple and Economical UV Spectrophotometric Area under Curve Method for Estimation of Eletriptan Hydro bromide

Mahesh Mukund Deshpande*[1], Veena Sanjay Kasture[2], Mahalaxmi Mohan[3]

[1]Research Scholar of Jawaharlal Nehru Technological University, Hyderabad, Telangana, India, Amrutvahini College of Pharmacy, Sangamner, Dist. Ahmadnagar, M.S. India, Savitribai Phule Pune University, Pune, Maharashtra, INDIA.
[2]Directore, Pinnacle Biomedical Reasearch Institute, Bhopal, Madhya Pradesh, INDIA.
[3]Head of Department, MGV's Pharmacy College, Nashik, Maharashtra, INDIA.

ABSTRACT

Objective: Simple and economical area under curve method was developed and validated for the estimation of Eletriptan Hydrobromide. **Method:** The solvent implemented for estimation of Eletriptan Hydrobromide was water. The λ_{max} was found to be 221 nm.The area under curve of UV spectrum between 216 to 226 nm. The method is validated according to ICH guideline Q2(R1). **Results:** The linearity range of Eletriptan HBr was found to be 5-25 µg/ml. The correlation coefficient was found to be 0.999. The values of %RSD for inter and intra-day precisions were within the acceptable limit. LOD and LOQ were found to be 5.77 µg/ml and 1.90 µg/ml respectively. % Recovery of Eletriptan HBr was 99.66 to 99.95.The values of ruggedness shows the method is rugged. **Conclusion:** The developed method can be implemented for routine analysis of Eletriptan HBr in bulk as well as formulation as per ICH Q2 (R1) guidelines.

Key words: Eletriptan HBr, Water, Area under Curve, UV-Spectrophotometer, ICH Q2(R1).

Correspondence
Mahesh M Deshpande
Research Scholar of Jawaharlal Nehru Technological University, Hydarabad, Telangana, Amrutvahini College of Pharmacy, Sangamner, Dist. Ahmadnagar, M.S. INDIA, Savitribai Phule Pune University, Pune, Maharashtra, INDIA.
Phone no: 9970363228
E-mail: maheshdeshpande83@gmail.com
DOI : 10.5530/phm.2018.1.6

INTRODUCTION

Eletriptanhydrobromide is a 5- Hydroxytryptamine1B/1D receptor agonist. Eletriptan binds with high affinity to 5-HT1B, 5-HT1D and 5-HT1F receptors, has modest affinity for 5-HT1A, 5-HT1E, 5-HT2B and 5-HT7 receptors. Eletriptan is chemically a designated as (R)-3-[(1-Methyl-2-pyrrolidinyl) methyl]-5-[2-(Phenyl sulfonyl) ethyl]-1H indol mono hydrobromide.

Its pharmacological effects include the constriction of cerebral blood vessels and neuropeptides secretion blockade which eventually relives the pain. Eletriptanhydrobromide was rapidly absorbed and extensively cleared by metabolism.[1,2] Literature survey shows that there are HPLC, UV methods available for estimation of Eletriptan HBr in bulk as well in formulation and in biological samples.[4-10] There were no Area under Curve method for estimation of Eletriptan HBr by using distilled water as a solvent. The proposed method is simple, economical and validated according to ICH guidelines for estimation of Eletriptan HBr in bulk as well as tablet dosage form.

MATERIALS AND METHODS

Active pharmaceutical ingredient (API) of Eletriptan Hydrobromide was supplied as a gift sample from Enaltec Lab. Abernathy, (Mumbai, India). Commercially available tablets (Elipran containing 20 mg of Elitriptan Hydrobromide) were obtained from local pharmacy.

Shimadzu UV 1800 (Japan) with matched quartz cells, connected to computer loaded with UV Probe Software, Single pan electronic balance, Sonication of the solutions was carried out using an Ultrasonic Cleaning Bath.

Preparation of standard stock and working standard solution:

The standard stock solution of Eletriptan Hydrobromide was prepared by dissolving accurately weighed 10mg of the drug in water and diluted to 100 ml with same solvent to obtain a final concentration of 100µg/ml.

Selection of wavelength range:

The standard solution of 10µg/ml was scanned between 400 nm to 200 nm in UV spectrophotometer against water as blank after baseline correction. Wavelength range was selected around wavelength maxima (221 nm). Different working standards were prepared between 5-25 µg/ml. Various wavelength range were tried and final range between 216-226 nm was selected based on linear relationship between area and corresponding concentration.

Method: Area Under Curve

The AUC (area under curve) method is applicable where there is no sharp peak or when broad spectra are obtained. It involves the calculation of integrated value of absorbance with respect to the wavelength between the two selected wavelengths λ1 and λ2. Area calculation processing item calculates the area bound by the curve and the horizontal axis. The horizontal axis is selected by entering the wavelength range over which area should be calculated.

VALIDATION OF THE METHOD

The method was validated in terms of linearity, precision, Sensitivity, repeatability and ruggedness, accuracy.

Linearity

The linearity was determined by using working standard solutions between 5-25 µg/ml. The spectrums of these solutions were recorded and area under curve was integrated in wavelength range 216-226 nm. Calibration curve of Area under curve vs. Concentration was plotted after suitable calculation and simple linear regression was performed (Figure 1). Regression equation and correlation coefficient were obtained. The

range of solution has been decided according to statistical parameters of generated equation.

Precision

Precision of the method was studied as intraday and inter-day variations. Intra-day precision was determined by analyzing the 5, 10, 15, 20, and 25 µg/ml of Eletriptan Hydrobromide solutions for three times in the same day. Inter-day precision was determined by analyzing the 5, 10, 15, 20, and 25 µg/ml of Eletriptan Hydrobromide solutions daily for three days over the period of week.

LOD and LOQ

The sensitivity of measurements of Eletriptan Hydrobromide by the use of the proposed method was estimated in terms of the Limit of Quantification (LOQ) and Limit of Detection (LOD). The LOQ and LOD were calculated using equation LOD = 3.3 x S.D. /m and LOQ = 10 x S.D. /m, where, 'm' is the slope of the corresponding calibration curve.

Repeatability

Repeatability was determined by analyzing 15 µg/ml concentration of Eletriptan Hydrobromide solution for six times.

Ruggedness

Ruggedness of the proposed method is determined for 15 µg/ml concentration of Eletriptan Hydrobromide by analysis of dilution from homogenous slot by two analysts using same operational and environmental conditions.

Accuracy

The accuracy for the analytical procedure was determined at 80 %, 100 % and 120 % levels of standard solution. Area under curve was measured in the range of 216-226 nm and results were expressed in terms of % recoveries. Three determinations at each level were performed and % RSD was calculated.

Determination of Eletriptan Hydrobromide In Bulk

Accurately weighed 10 mg of Eletriptan Hydro bromide was transferred to a 100-ml volumetric flask and 50 ml water was added. After shaking for 2min, the mixture was diluted up to mark with water. From stock solution, correct dilution was taken in such a way that the final concentration is 100µg/ml. The concentrations of the drug were calculated from linear regression equations. The resulting solution was scanned on a spectrophotometer in the UV range 200-400 nm. The spectrum was recorded at 221 nm.

Application of proposed method for Pharmaceutical formulation

For analysis of commercial formulation two tablet of 20 mg Elitriptan hydro bromide was transferred to 100 ml volumetric flask 50 ml water was added. After ultrasonic vibration for 15 min, the mixture was diluted up to mark with water. The whole solution filtered using whatman filter paper no. 42. From filtrate, correct dilution was taken in such a way that the final concentration is 100 µ/ml. The concentration of the drug was calculated from linear regression equations. The resulting solution was scanned on a spectrophotometer in the UV range 200-400 nm. The spectrum was recorded at 221 nm.

RESULTS AND DISCUSSION

Method Validation

The proposed method was validated as per ICH Guidelines. The solution of drugs was prepared as per the earlier adopted procedure given in the experiment.

Linearity studies

The linear regression data for the calibration curves showed good linear relationship over the concentration range 5-25 µg/ml for Eletriptan Hydrobromide (Figure 1). Linear regression equation was found to be Y=0.7532x + 1.262 (r2 = 0.999). The result is expressed in Table 1.

Precision

The precision of the developed method was expressed in terms of % relative standard deviation (% RSD). These result shows reproducibility of the assay. The % R.S.D. values found to be less than 2, so that indicate this method precise for the determination of both the drugs in formulation. The results are given in Table 2.

Sensitivity

The linearity equation was found to be Y= 0.7532x + 1.262 (r2 = 0.999). The LOQ and LOD for Eletriptan Hydrobromide were found to be 5.77 µg and 1.90 µg, respectively. The results are given in Table 3.

Repeatability

Repeatability was determined by analyzing 15 µg/ml concentration of Eletriptan Hydrobromide solution for six times and the % amount found was between 97% to 101% with % R.S.D. less than 2. The results are given in Table 4.

Ruggedness

The peak area was measured for same concentration solutions, six times. The results are in the acceptable range for both the drugs. The results are given in Table 6. The results showed that the % R.S.D. was less than 2%. The results are given in Table 5.

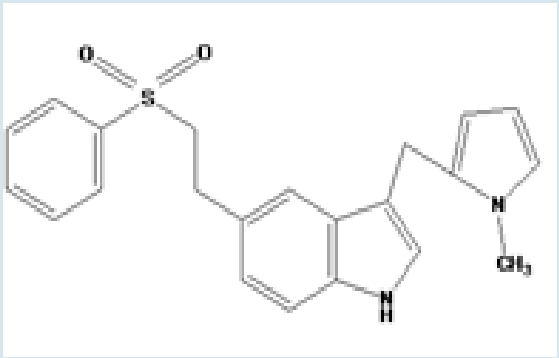


Figure 1: Structure of EletriptanHydrobromide.

Table 1: Linearity study of Eletriptanhydrobromide

Conc. (µg/ml)	Absorbance Mean (n=3)	% R.S.D.
5	0.467	0.4393
10	0.832	0.4298
15	1.203	0.4358
20	1.548	0.4315
25	1.910	0.4353

Table 2: Results of Precision studies

Conc.(µg/ml)	Intra-day		Inter-day	
	Amt.found	% R.S.D.	Amt.found	% R.S.D.
5	100.06	0.2437%	101.4	0.2469%
10	98.97	0.2410%	99.06	0.2412%
15	99.50	0.2423%	100.17	0.2440%

Average of three estimations.

Table 3: Sensitivity studies

L.O.D. (µg/ml)	L.O.Q. (µg/ml)
1.90	5.77

Table 4: Repeatability studies

Component	Amount taken (µg/ml) (n=6)	Amount found %	% R.S.D.
Eletriptanhydrobromide	15	15.12	0.2441%

Table 5: Ruggedness studies

Component	Amount taken (µg/ml) (n=3)	Amount found (%)	
		Analyst-I ±SD	Analyst-II ±SD
Eletriptanhydrobromide	15	99.4210±0.2437	99.6802±0.2430

Table 6: Accuracy studies

Drug	Initial amount (µg/ml)	Amount added (µg/ml)	% Recovered	% R.S.D.
Eletriptanhydrobromide	10	8	98.01%	0.2387%
	10	10	100.05%	0.2437%
	10	12	99.72%	0.2429%

Table 7: Analysis of Eletriptanhydrobromide in Formulation

Conc.(µg/ml)	Amount found (%)	Mean Amount found (%)	(%) R.S.D.
10	99.5 99.8 99.1	99.4	0.2973(%)

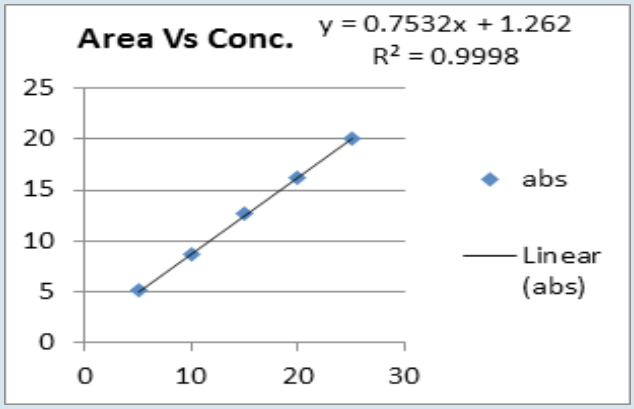


Figure 2: Calibration Curve of ElitriptanHydrobromide (5-25 µg/ml).

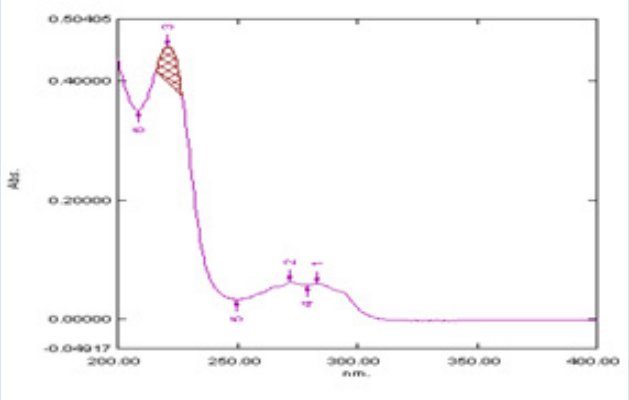


Figure 3: UV spectrum of ElitriptanHydrobromide (10 µg/ml) in water.

Accuracy

The solutions were reanalyzed by proposed method; results of recovery studies are reported in Table 2 which showed that the % amount found was between 97.00% to 101.00% with %R.S.D.>2.The results are given in Table 6.

Application of Proposed Method For Pharmaceutical Formulation

The spectrum was recorded at 221 nm. The concentrations of the drug were calculated from linear regression equation. The % amount found was between 98.00% to 102.00%. The results are given in Table 7.

CONCLUSION

This UV Spectrophotometric method is quite simple, accurate, precise, reproducible and sensitive. The UV method has been developed for quantification of Eletriptanhydrobromide in tablet formulation. The validation procedure confirms that this is an appropriate technique for their quantification in the formulation. It is also use in routine quality control of the formulation containing this entire compound.

ACKNOWLEDGEMENT

The author is very much thankful to Dr. S. B. Kasture, Director, Pinnacle Biomedical Research Institute, Bhopal, and M.P. The author is thankful to Mr. Anna Saheb Gaikwad for his kind support. Also, the author is very much thankful to the management of Amrutvahini College of Pharmacy, Sangamner, M.S, India.

CONFLICT OF INTEREST

The author is not having any conflict of interest associated with this work.

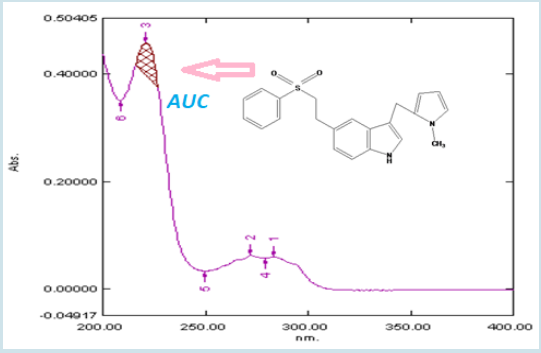
ABBREVIATION USED

HBr: Hydrobromide; **ICH:** International council for Harmonization; **LOD:** Limit of detection; **LOQ:** Limit of Quantitation; **RSD:** Relative Standard deviation.

REFERENCES

1. Dollery C. Therapeutic Drug index, 2nd edition, page no. S21-S25.
2. Willard, Merritt, Dean, Settle. Instrumental methods of analysis, 7th edition: 118-120,131-2.
3. ICH-Guidelines Q2 (R1), Validation of Analytical Procedures: Text and Methodology. (2005).
4. Cooper, Muirhead DC, Taylor JE. Journal of Pharmaceutical Biomedical Analysis. 1997;21,787.
5. Sunitha P, Jhansirani CH. Method development and validation of Eletriptan hydrobromide tablets by UV-visible Spectrophotometric Method. International Journal of Pharmaceutical, Chemical and Biomedical Sciences. 2012;2(4):427-30.
6. Rajasekhar L, Venkatamahesh R, Satyanarayana PS. Development and Validation of derivative Spectrophotometric Method for Quantitative Estimation of Eletriptan Hydrobromide in Bulk and Pharmaceutical dosage form. 2011;2(3):2229-3701.
7. Ponnuru VS, Challa BR, Nandendla R. Quantitative Analysis of Eletriptan in human plasma by HPLC-MS/MS and its application to pharmacokinetic study. Analytical and Bio-analytical Chemistry. 2011;401(8):2539-48.
8. Sunitha D, Rao LA. RP-HPLC Method for the Estimation Eletriptan in Pharmaceutical Dosage From. International Journal Chemical Environment and Pharmaceutical. 2010;1(2):95-9.
9. Zeevi M, Jocic B. Validation of an HPLC Method for the Simultaneous Determination of Eletriptan and UK 120.413. Journal Serbia Chemical Soc. 2006;71(11):1195-205.
10. Jocic B, Zecevic M, Zivanovic L. Study of forced degradation behavior of Eletriptan Hydromide by LC and LC-MS and development of Stability-indicating Method. Journal of Pharmaceutical and Biomedical Analysis. 2009;50(4):622-9.

PICTORIAL ABSTRACT



SUMMARY

- The objective of study is to develop and validate simple, economical method for estimation of Eletriptan HBr.
- The current method is really simple, economical, accurate, and reproducible for estimation of Eletriptan HBr.
- This method follows ICH guidelines Q2 (R1).The analysis is in 200-400nm range and AUC was taken 216-226 nm

ABOUT AUTHORS

Mahesh M. Deshpande: The author is a Research scholar of Jawaharlal Nehru Technological University, Hyderabad, Telangana, India. Also Working as Assistant Prof .in Amrutvahini College of Pharmacy, Sangamner, M.S., India. He is especially interested in Pharmaceutical Analytical method development and validation. His area of interest is Bioanalytical method development and validation.