An Elsevier Indexed Journal ISSN-2230-7346

Journal of Global Trends in Pharmaceutical Sciences

VALIDATED AND A SENSITIVE UV SPECTROPHOTOMETRIC METHOD FOR ESTIMATION OF TAMSULOSIN HYDROCHLORIDE IN BULK AND COMMERCIAL FORM

Radhika V, Kanaka Raju A*, Vara Prasada Rao K, Srinivasa Rao Y, Deepthi R

Vignan Institute of Pharmaceutical Technology, Visakhapatnam, Andhra Pradesh, India

*Corresponding author E-mail: **rajuaddipalli81@gmail.com**

ARTICLE INFO

Key Words

Tamsulosin Hydrochloride, UV spectrophotometer, Method validation, Estimation.

Access this article online

Website: https://www.jgtps.com/

Quick Response Code:

ABSTRACT

A simple, sensitive and reliable UV spectrophotometer method has been developed for the estimation of Tamsulosin hydrochloride in the bulk and pharmaceutical dosage form. Estimation was carried out at a wavelength of 235nm using acetonitrile as solvent. The beer lambert's law range was observed in the range of 10-65µg/ml with correlation coefficient (R^2=0.999). The percentage recovery was found to be in the range of 98.9 to 100.7%. The proposed method was found to be simple, accurate, and reproducible. All the parameters of the analysis were chosen according to ICH [Q2 (R1)] guidelines and validated statistically using SD and %RSD.

INTRODUCTION

Tamsulosin hydrochloride chemically is 5-[(2R)-2-[2-(2-ethoxyphenoxy) ethylamino] propyl]-2-methoxybenzenesulfonamide hydrochloride[1].

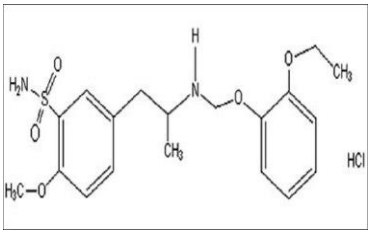

Figure 1: chemical structure of Tamsulosin hydrochloride

Tamsulosin is used by men to treat the symptoms of an enlarged prostate[2] (benign prostatic hyperplasia-BPH). It does not shrink the prostate, But it works by relaxing the muscles in the prostate and the bladder. This helps to relieve symptoms of BPH such as difficulty in beginning the flow of urine, weak stream, and the need to urinate often or urgently (including during the middle of the night). A Literature survey reveals a very few spectrophotometric and HPLC methods were reported for the estimation of Tamsulosin hydrochloride [3-12], hence there is still Need for the development of a sensitive spectrophotometric method for the estimation of Tamsulosin hydrochloride with complete validation as per ICH guidelines.

MATERIALS AND METHOD:

Instrument and reagents

A sample of Tamsulosin hydrochloride with a purity of 99.8% w/w was obtained from spectrum labs, Hyderabad. LAB INDIA double beam UV/visible spectrophotometer and Elite

analytical balance instruments were used. Chemicals and reagents are analytical grades. Tamsulosin hydrochloride of a 0.4 mg label claim was purchased from a local Pharmacy.

Preparation of standard stock solution: A standard drug solution of Tamsulosin hydrochloride was prepared by transferring 100mg of the drug into a 100ml of volumetric flask and made up to the mark with acetonitrile to get a concentration of 1000µg/ml.

Preparation of working stock solution; From the above standard stock solution, 10ml of the sample solution was transferred to a 100ml volumetric flask and made up to the mark with acetonitrile to get a concentration of 100µg/ml.

Absorption maxima: It was scanned by a UV spectrophotometer in the range of 200-400nm using acetonitrile as blank. The maximum absorbance was found at 235nm.

Preparation of sample solution;10 tablets were weighed and powdered, an amount of powder equivalent to 100mg of Tamsulosin was weighed and dissolved in 100ml acetonitrile and then filtered through Whattman filter paper (no.41) to get a clear solution of 1000µg/ml. From the above solution, 10ml was pipetted out and made up to 100ml with diluents to get a final concentration of 100µg/ml.

METHOD VALIDATION[13]

The objective of method validation is to demonstrate that the method is suitable for its intended purpose. The method was validated for linearity, precision, accuracy, robustness, ruggedness, LOD & LOQ.

Linearity: Different aliquots of Tamsulosin in the range of 1.0-6.5ml transferred into a 10ml volumetric flask and made up to the mark with acetonitrile. The solutions were analyzed at 235nm. A Calibration curve was plotted by taking concentration on the x-axis and absorbance on the y-axis.

Precision: The closeness of agreement between the obtained values by analyzing the sample multiple times under prescribed conditions. There are 3 levels: repeatability, intermediate precision, and reproducibility. Repeatability is a measure of the exactness under the same working conditions more than a short interior of time, i.e., under ordinary working states of the scientific technique with the same hardware. It is also known as intraday precision. Reproducibility is also known as inter-day precision. Precision is expressed in terms of %Relative Standard Deviation

%RSD = **Standard Deviation**/mean X 100

Standard Deviation (SD)

$$\mathbf{SD} = \sqrt{\sum (x - \bar{x})^2}/n-1$$

Where n= number of entries

Accuracy: Accuracy means the expression of closeness of agreement between the values which is accepted either as a conventional true value or an accepted reference true value and the value found, accuracy is assessed by using 9 determinations covering a minimum of 3 concentrations

Robustness: The Robustness of an analytical procedure is the capacity to remain unchanged by small but deliberate changes in parameters.

Ruggedness: The Ruggedness of an analytical procedure is the degree of reproducibility of results by analyzing the same sample under a variety of conditions like laboratories, instruments, analysts, reagents, etc.

Sensitivity: Limit of detection (LOD) and Limit of quantification (LOQ) of the drug was calculated by using equations according to ICH guidelines

Limit of Detection: It is the lowest amount of the drug in the sample that can be detected, but not necessarily quantified.

$$\mathbf{LOD} = \frac{3.3X\sigma}{s}$$

Where, S=standard deviation

Limit of Quantification: It is an amount of analyte that can be quantified with a specified limit of accuracy and precision.

$$\mathbf{LOQ} = \frac{10X\sigma}{s}$$

Where, S= standard deviation

Linearity: From the standard stock solution, the various dilutions in the concentration of 10-65 µg/ml were prepared. The solutions were scanned at 235nm and the absorbance was recorded and shown in [Table1]. From this, the calibration curve was obtained by plotting absorbance versus concentration of Tamsulosin Hydrochloride and the linearity was represented in figure 2. The correlation coefficient was found to be 0.999.

Precision: The Repeatability of the method was checked by scanning a 40µg/ml solution for 6 times represented in [Table 2]. Intraday precision was determined by checking the absorbance of (40µg/ml) on the same day (morning, afternoon, evening) and the results were represented in [Table 3]. Inter-day precision was determined by checking the absorbance of (40µg/ml) on three different days and the obtained results were represented in [Table 4].

Accuracy: An Accuracy study was conducted by spiking at three concentration levels (80%, 100%, and 120%). At each level, triplicate samples were scanned and the percentage recovery was determined and presented in the [Table 5].

ROBUSTNESS: To determine the robustness of the method one parameter wavelength variation was made slightly different from the selected wavelength. No significant difference was found in the absorbance and hence proposed method was considered as robust and reports were reported in the [Table 6].

Ruggedness: The ruggedness of the developed method was checked by analyzing the samples by different analysts at different days at similar operational conditions. The statistical analysis showed no significant differences between results obtained by employing different analysts and results were shown in the [Table 7].

Limit of detection and limit of quantification

Limit of detection is the lowest amount of an analyte in a sample that can be detected but not necessarily quantified, under the stated experimental conditions. Limit of quantification is the lowest amount of an analyte in a sample that can be quantified, under the stated experimental conditions. The LOD and LOQ for Tamsulosin were found to be 0.03µg/ml and 0.11 µg/ml and results were shown in the [Table 8].

Assay of Tamsulosin tablets

For the analysis, 10 tablets of Tamsulosin were weighed and finely powdered. An accurately weighed quantity of powder equivalent to 100mg quantity of Tamsulosin was taken in a 100ml volumetric flask. Few ml of acetonitrile was added and sonicated for 15 minutes and then filtered through Whattman filter paper (No.41) and volume was adjusted to solvent. From the further dilution was made to get a final concentration of 40µg/ml. The results were presented in the [Table 9].

RESULTS AND DISCUSSION

The method was developed and validated as per ICH guidelines. The method was validated in terms of linearity, precision, accuracy, robustness, ruggedness, LOD, LOQ and specificity. Beer's law is obeyed over the concentration range 10 to 65 µg/ml, using regression analysis. The linear equation $Y=0.014x+0.02$ with a correlation coefficient of $r^2=0.999$. The precision results show %RSD <2 at each level, which indicates that the method is precise.

The accuracy of the method was checked by recovery studies was found to be accurate within the range of 98.9 to 100.7%. The robustness and ruggedness results reveal that the method is more sensitive. There was no interference observed from the excipients present in the formulation, indicated that the method is specific. Determination of Tamsulosin Hydrochloride in tablet formulation showed the content of Tamsulosin Hydrochloride was very close to the label amount. The %RSD values in all the parameters were within the acceptable limit (<2%). All the characteristics of the method are represented in [Table 9].

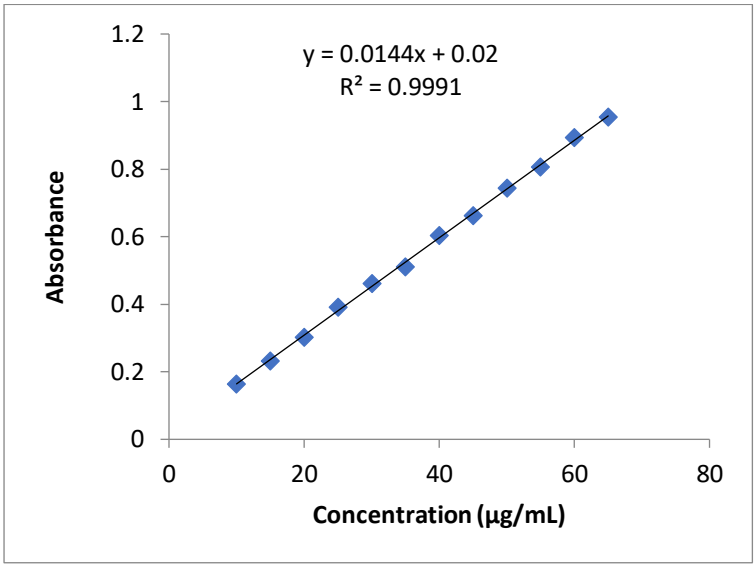

Figure 2: calibration graph of Tamsulosin Hydrochloride

Table 1: Linearity data

Concentration (µg/ml)	Absorbance
10	0.1644
15	0.2330
20	0.3025
25	0.3912
30	0.4614
35	0.5111
40	0.6046
45	0.6631
50	0.7437
55	0.8064
60	0.8940
65	0.9540

Table 2: Repeatability data

Concentration (µg/ml)	Absorbance	Statistical analysis
40	0.6634	Mean=0.6656
40	0.6649	%RSD=0.31%
40	0.6638	
40	0.6622	
40	0.6643	
40	0.6757	

Table3: Intraday precision

Concentration (µg/ml)	%RSD			Average %RSD
	1	2	3	
40	0.81	0.36	0.21	0.46

Table 4: Inter-day precision

	%RSD			
Concentration (µg/ml)	1	2	3	**Average %RSD**
40	0.31	0.11	0.28	0.23

Table 5: Accuracy data

Level of addition (%)	Tablet Amount (µg/ml)	Amount added (µg/ml)	Amount found (µg/ml)	%Recovery	%Mean Recovery ±SD
80%	40	32	31.7	99.26	99.91%
100%	40	40	39.9	99.93	±0.306
120%	40	48	48.28	100.56	

Table 6: Robustness data

Concentration (µg/ml)	λ1 (234nm)	λ2 (235nm)	λ3 (236nm)
	Absorbance	Absorbance	Absorbance
40	0.6615	0.6634	0.6728
40	0.6618	0.6649	0.6639
40	0.6642	0.6638	0.6648
40	0.6671	0.6622	0.6651
40	0.6654	0.6643	0.6662
40	0.6635	0.6751	0.6675
	%RSD=0.13%	%RSD=0.31%	%RSD=0.22%

Table 7: Ruggedness data

Concentration (µg/ml)	Analyst 1		Analyst 2	
40	0.6725	Mean=0.6662	0.6622	Mean=0.6655
40	0.6632	%RSD=0.21%	0.6634	%RSD=0.34%
40	0.6645		0.6616	
40	0.6667		0.6643	
40	0.6654		0.6757	
40	0.6651		0.6663	

Table 8: LOD & LOQ Results

	Limit of detection	Limit of quantification
	0.03µg/ml	0.11µg/ml

Table 9: Validation parameters for Tamsulosin

Parameters	Results
Absorption maxima (nm)	235nm
Linearity range (µg/ml)	10 - 65
Regression equation	Y=0.014x+0.02
Correlation coefficient (r^2)	0.999
Molar extinction coefficient	618600
LOD (µg/ml)	0.03
LOQ (µg/ml)	0.11
Accuracy (%Recovery± SD)	99.91±0.303
% Assay	98.2%
Precision	
Intraday precision (%RSD)	0.46
Inter-day precision (%RSD)	0.23
Sandell's sensitivity ($\mu g/cm^2$/0.001 absorbance units)	0.066

CONCLUSION

A validated UV spectrophotometric method has been developed for the estimation of Tamsulosin Hydrochloride in bulk as well as the Pharmaceutical dosage form. The developed method was found to be simple, precise, specific, reproducible and linear over the concentration range studied. The proposed method can be used for routine analysis of Tamsulosin in bulk as well as pharmaceutical formulations.

Acknowledgment: The authors are thankful to spectrum labs, Hyderabad, India for providing the gift sample of Tamsulosin Hydrochloride for research work. I am thankful to the principal and the management, Vignan Institute of Pharmaceutical Technology, Visakhapatnam for providing necessary facilities to carry out this research work.

REFERENCES:

1. P. Narayan and H.S. Tunguntla, Rev. Urol., 2005, 7, 542.
2. K. Kawabe, A. Veno, Y. Takimoto, Y. Aso and H. Kato, J. Urol., 1990, 144: 908.
3. Bari S.B., Bakhshi A. R., Jain P. S, and Surana S. J., Development and Validation of Stability-Indicating HPTLC Determination of Tamsulosin in Bulk and Pharmaceutical Dosage Form, *Chromatography Research International*, 2011: 6.
4. Siddhartha B, Gupta Ravichandra, Analytical Method Development and Validation for simultaneous estimation of Tamsulosin and Tolterodine in Bulk and Pharmaceutical dosage form by RP-HPLC method, *Asian Journal of Pharmaceutical and clinical research*, 2014, 7 (2): 156-160.
5. Rao M.V.B., Sreedhar B, Analytical Method Development and Validation of RP-HPLC Method for determination of Tamsulosin Hydrochloride, *International Journal of Chemical Science*, 2008, 6 (3): 1695-1701.
6. Sindhura M., Raghavi K., Prashanthi R. and Nalluri B. N., Simultaneous Estimation of Finasteride and Tamsulosin Hydrochloride in Combined Dosage Forms by RP-HPLC-PDA Method, *Journal of Applied Pharmaceutical Science*, 2012, 2 (06): 203-209.
7. Bandla Jahnavi, Gorja Ashok, Method Development and Validation For The Simultaneous Estimation of Dutasteride and Tamsulosin Hydrochloride In Pharmaceutical Dosage Forms By RP-HPLC, *World Journal of Pharmaceutical Research*, 2014, 3(3): 4113-4121.
8. DendukuriMrudula, G. Sai Prasad, Rao P. V., Simultaneous Estimation And

Validation of Tamsulosin and Deutasteride in Bulk and Pharmaceutical Dosage Form, *International Journal of Research in Pharmaceutical and Nano Sciences*, 2014, 3(4): 242 – 248.

9. Reddy Sravan kumar, Debnath Manidipa, Viriyala Raj Kumar, Analytical Method Development & Validation For Simultaneous Determination of Dutasteride And Tamsulosin in Bulk as well as In Pharmaceutical Dosage Form By Using RP-HPLC, *International Journal of Pharmacy and Pharmaceutical Sciences*, 2014, 6(3): 77-84.

10. Kamepalli Sujana, D. Gowri Sankar, Konda Abbulu and O. Bala Souri, Simultaneous estimation of Finasteride and Tamsulosin Hydrochloride by reverse phase HPLC in bulk and pharmaceutical Dosage Form, *International Journal of Pharmacy and Life Sciences*, 2012, 3(8): 1905-1908.

11. Khaled Bin Sayeed, Sh.Rizwan, Hanifa Begum, Development and Validation of Stability Indicating Method for the Simultaneous Determination of Tamsulosin and Dutasteride in Bulk Drugs and Pharmaceutical Dosage Forms Using UV Spectrophotometric Method, *International Journal of Research and Development in Pharmacy and Life Sciences*, 2015, 4(2): 1434-1446.

12. Gadhave Nilam, Ghante Minal, Simultaneous estimation of Tamsulosin hydrochloride and Finasteride in combined dosage form by UV spectroscopy method. *Journal of Pharmacy Research*, 2011, 4(8): 2672- 2674.

13. ICH Hormonized-tripartite guidelines. Validation of analytical procedure: text and methodology Q2 (R1), 2005.