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Development of First Order Derivative UV-Spectrophotometric Method for Determination of Tamsulosin Hydrochloride in Bulk and in Formulation

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Abstract

A simple, rapid, accurate and economical "First order UV-derivative spectrophotometric" method has been developed for estimation of Tamsulosin (TMS) from bulk and pharmaceutical formulation. The λ max of TMS in methanol and water was found to be 280 nm. The same spectrum was derivatised in to first order derivative; showed maximum amplitude of the trough at 299 nm. The drug follows linearity in the concentration range 10 - 90 µg/ml with correlation coefficient value 0.997. The value of correlation coefficient (r^2) greater than 0.99 indicate good linearity response in the above-mentioned range. The proposed method was applied to pharmaceutical formulation and % amount of drug estimated 99.58 % was found in good agreement with the label claim. The accuracy of the method was checked by recovery experiment performed at three different levels i.e., 80%, 100% and 120 %. The % recovery was found to be in the range 99.00%– 101.63%. The low values of % R.S.D. are indicative of the accuracy and reproducibility of the method. The precision of the method was studied as an intra-day, inter-day variations and repeatability. The % R.S.D. value less than 2 indicate that the method is precise. Ruggedness of the proposed method was studied with the help of two analysts. The results did not show any statistical difference between operators suggesting that method developed was rugged.

Keywords: Tamsulosin, First order UV-derivative method, UV-spectroscopy.

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1. Introduction

Analytical chemistry may be defined as the science and art of determining the composition of material in terms of elements or compounds contained in it [1-2]. Analytical chemistry is divided into two branches quantitative and qualitative [3-4]. A qualitative analysis provides information about the identity of atomic or molecular species or functional groups in sample. A quantitative analysis provides numerical information as to the relative amount of one or more of these components [5-7].

For analyzing the drug samples in bulk, pharmaceutical formulations and biological fluids, different analytical methods are routinely being used. These analytical methods are classified as classical and instrumental [8-10].

The instrumental methods are simple, precise and reproducible as compared to classical methods [11-13]. Therefore, analytical methods developed using sophisticated instruments such as spectrophotometer, HPLC, GC and HPTLC have wide applications in assuring the quality and quantity of raw materials and finished products [14-16].

Spectrophotometric methods [17-19]. The fundamental law that governs the quantitative spectrophotometric analysis is Beer- Lambert's law which is stated as [20],

"When a beam of monochromatic light is passed through a transparent cell containing a solution of an absorbing substance, reduction of intensity of the light may occurs; the rate of reduction in intensity with the thickness of the medium is proportional to the intensity of the light and the concentration of the absorbing substances" [21-22].

Mathematically Beer- Lamberts law is expressed as:

$$A = a\ b\ c;$$

where, A = absorbance or optical density, a = absorptivity or extinction coefficient,
b = path length of radiation through sample (cm), c = concentration of solute in solution

For the quantitative assay of drug samples following spectrophotometric methods are routinely being used [23-25];

1. Simultaneous equation method
2. Orthogonal polynomial method
3. Multi-component mode method
4. Difference spectrophotometric method
5. Method of least squares (use of calibration curve)
6. Derivative spectrophotometric method
7. Absorbance ratio method
8. Chemical derivatization method
9. Geometric correction method

Spectroscopic methods are analytical techniques based on the interaction of electromagnetic radiation with matter to produce an absorption, emission, or scattering spectrum that can be used for qualitative and quantitative analysis of substances [26-28].

These methods are widely used in pharmaceutical analysis for identifying compounds, determining purity, and quantifying drugs in bulk and formulations [29-30].
Spectroscopic methods are essential analytical tools in pharmaceutical sciences, offering simple, rapid, and reliable techniques for the analysis, identification, and quality control of pharmaceutical substances [31-32]. Each method provides unique information about the molecular or atomic characteristics of compounds, enabling comprehensive analysis and regulatory compliance in pharmaceutical analysis [33-34].

Derivative Spectrophotometry.

It involves the conversion of a normal spectrum to its first, second or higher derivative spectrum [35]. The transformations that occur in the derivative spectra are under stood by reference to a Gaussian band which represents and ideal absorption band [36-37]. In this context of derivative Spectrophotometry, the normal absorption spectrum is referred to as fundamental zero order or D° spectrum. Conventional absorption spectrum is a plot of A vs λ. In this technique plot of A vs λ is transformal in to plot of $dA/d\lambda$ vs λ (first derivative of the absorption spectrum) $d2A/d2\lambda$ vs λ (second derivative) or higher derivative [38].

For quantitative work, the amplitude of a derivative peak can be measured in various ways [39-40]. Although, true derivative amplitude is that measured with respect to the derivative zero, the most common practice is to record the amplitude with respect to a satellite in the spectrum, which affords an extra degree of suppression of interference from extraneous substances [41-43].

Tamsulosin Hydrochloride, (*R*)-5-(2-(2-(2-ethoxyphenoxy) ethyl amino) propyl)-2-methoxybenzenesulfonamide, is a selective α_1A-adrenergic receptor antagonist primarily prescribed for the treatment of benign prostatic hyperplasia (BPH) [44]. Tamsulosin selectively blocks **α_1A-adrenoceptors** in the smooth muscle of the **prostate, bladder neck, and urethra**. This reduces muscle tone, improving urine flow and decreasing symptoms of BPH [45]. Selectivity for **α_1A** over **α_1B** receptors helps minimize systemic blood pressure effects compared to non-selective α-blockers [46-47].

2. Materials and Methods

Tamsulosin Hydrochloride was obtained from Alkem Laboratories Ltd., Mumbai, India as a gift sample. Potassium dihydrogen ortho-phosphate (AR Grade), acetonitrile (HPLC Grade), was purchased from Merck (India) Ltd., Worli, Mumbai, India. Tablets (**Veltam-0.4)** were purchased from Indian market, containing Tamsulosin Hydrochloride 0.4 mg per tablet.

2.1 Instrumentation

Analysis was performed on UV – Spectrophotometer (UV-Shimadzu 2450), UV-visible double beam spectrophotometer having Software UV Probe 2.21. Considering the drug characteristics and solvent properties, the solubility of TMS was checked in different solvents, and methanol: water (2:8) was selected as the solvent for dissolving the drug.

2.2 Preparation of standard stock solution

10 mg of TMS was transferred to 100 ml volumetric flask, dissolved in 20 ml methanol by shaking manually for 10 min. The volume was adjusted to mark to give final strength i.e. 100 µg/ml.

2.3 Selection of wavelength for analysis of TMS

Appropriate volume 1 ml of standard stock solution of TMS was transferred into 10 ml volumetric flask, diluted to mark with double RO water to give concentration of 10 µg/ml. The resulting solution was scanned in UV range (200 nm – 400 nm). In zero order

spectrum TMS showed absorbance maximum at 280 nm. The same spectrum was derivatized into first order using UV- probe software of the UV-spectrophotometer. The amplitude of the trough was found at 299 nm. (Fig. 1)

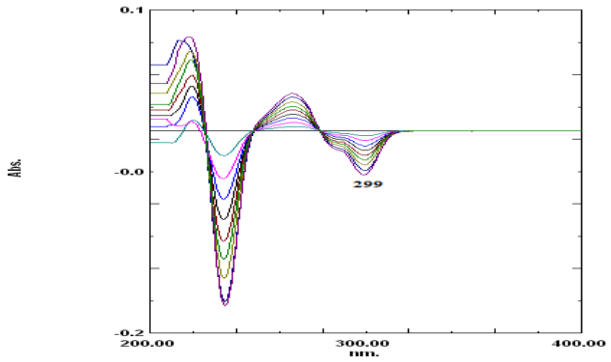

Fig. 1: First Order Derivative Spectrum of TMS at 299 nm

2.3 Study of calibration curve

Different aliquots of TMS in range 1-9 ml were transferred into series of 10 ml volumetric flasks and the volume was made up to the mark with double RO water to get concentrations 10, 20, 30, 40, 50, 60, 70, 80 and 90 µg/ml, respectively. The solutions were scanned on spectrophotometer in the UV range 200 - 400 nm. The spectrum was derivatized into first order using UV-probe software of the spectrophotometer; amplitude of the trough was recorded at 299 nm **Table 1**. The calibration plot was constructed as concentration vs amplitude; **Figure 2**.

Table 1: Linearity study of TMS

Sr. No.	Concentration µg/ml	Amplitude Mean ± S.D. (n=6)	% R.S.D.
1	10	0.06064 ± 0.00075	**1.23**
2	20	0.08628 ± 0.0010	**1.20**
3	30	0.13680 ± 0.0027	**1.98**
4	40	0.18058 ± 0.0012	**0.68**
5	50	0.23212 ± 0.0016	**0.72**
6	60	0.2730 ± 0.0016	**0.60**
7	70	0.3200 ± 0.0018	**0.36**
8	80	0.36718 ± 0.0012	**0.33**
9	90	0.4189 ± 0.0033	**0.78**

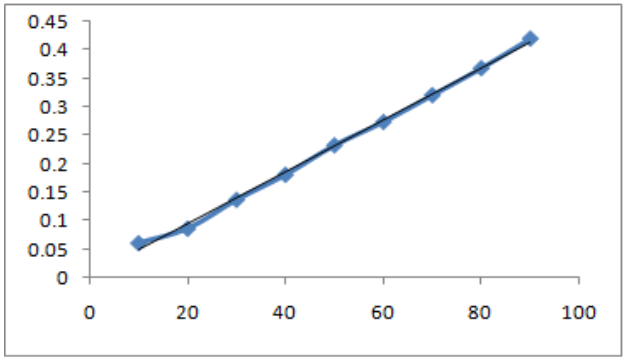

Figure 2: Calibration curve of TMS at 299 nm

Y = 0.004 X + 0.002; Slope = 0.004, Intercept = 0.002,
Coefficient of correlation = 0.997

2.4 Determination of TMS in bulk

Accurately weighed 10 mg of TMS was transferred into 100 ml volumetric flask containing 20 ml methanol and volume was made up to the mark using double RO water. Appropriate volume 2 ml of this solution was transferred to 10 ml volumetric flask and volume was adjusted to mark using double RO water. The resulting solution was scanned on spectrophotometer in the UV range 200 - 400 nm and amplitude of corresponding trough was measured at 299 nm. The concentrations of the drug were calculated from linear regression equations; results are shown in **Table 2**.

Table 2: Analysis of TMS in bulk

Concentration (µg/ml)	Amount found (mg)	Amount found (%)
20	20.08	100.38
	19.80	99.00
	19.73	98.63
	20.30	101.50
	20.18	100.88
	20.23	101.13
Mean ± SD	20.05 ± 0.24	100.25 ± 1.18
% R.S.D.	1.17	1.17

2.4 Application of proposed method for tablet formulation

For analysis of commercial formulation; twenty tablets were weighed, mean weight determined and crushed into fine powder. An accurately weighed quantity of powder equivalent to 5 mg of TMS was transferred into 25 ml volumetric flask containing 20 ml methanol, shaken manually for 10 min, volume was adjusted to mark with double RO water and filtered through Whatman filter paper no. 41. Appropriate volume 2 ml of solution was transferred to 10 ml volumetric flask and volume was adjusted to mark with double RO water. It was scanned on spectrophotometer in the UV range 200 - 400 nm. The spectrum was derivatized into first-order derivative and amplitude of the trough was recorded at 299 nm. The concentrations of the drug were calculated from linear regression equation; results are shown in **Table 3**.

Table 3: Analysis of tablet formulation

Label claim	Amount found (mg)	Amount found (%)
0.4 mg	0.3960	99.00
	0.4015	100.38
	0.3990	99.75
	0.4055	101.38
	0.4065	101.63
	0.3980	99.50
Mean ± S.D.	0.401 ± 0.0042	100.27 ± 1.05
% R.S.D.	1.050	1.05

Brand name: Veltam-0.4
Batch No: DJ1505
Average weight: 0.1143 gm

2.5 Validation of the method

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

2.6 Recovery studies

To the pre-analysed sample solutions, a known amount of standard stock solution was added at different levels i.e. 80%, 100% and 120 %. The solutions were reanalyzed by proposed method; results of recovery studies are reported in **Table 4**.

Table 4: Results of recovery studies

Pre-analysed sample solution (µg/ml)	Amount of drug added (µg/ml) (n=3)	Amount recovered (µg/ml) (n=3)	% Recovery	% R.S.D.
20	0	20.11	100.54	1.06
	16	16.03	100.21	1.61
	20	19.79	99.79	1.29
	24	24.02	100.06	1.84

2.7 Precision

Precision of the method was studied as intra-day and inter-day variations. Intra-day precision was determined by analyzing the 20, 30 and 40 µg/ml of TMS solutions for three times in the same day. Inter-day precision was determined by analyzing the 20, 30 and 40 µg/ml of TMS solutions daily for three days over the period of week; results are reported in **Table 5**.

Table 5: Results of precision studies

Component	Conc. (µg/ml)	Intra-day precision (n=3)		Inter-day Precision (n=3)	
		Conc. found	% R.S.D.	Conc. found	% R.S.D.
TMS	20	20.33	1.45	20.18	1.39
	30	30.12	1.00	30.28	1.56
	40	39.91	0.96	40.73	1.22

2.8 Repeatability

Repeatability was determined by analyzing 20 µg/ml concentration of TMS solution for six times and the results are reported in **Table 6**.

Table 6: Results of repeatability studies

Component	Amount taken (µg/ml) (n=6)	Amount found (%)	%RSD
Tamsulosin	20	99.92 ± 1.44	1.45

2.9 Ruggedness

Ruggedness of the proposed method is determined by analysis of aliquots from homogenous slot by two analyst using same operational and environmental conditions and the results are reported in **Table 7**.

Table 7: Results of ruggedness studies

Component	Amount taken (µg/ml) (n=3)	Amount Found (%)	
		Analyst I ±S.D.	Analyst II ±S.D.
Tamsulosin	20	99.97 ± 0.81	99.85 ± 0.78

3. Summary And Discussion

A simple, rapid, accurate and economical "First order UV-derivative spectrophotometric" method has been developed for estimation of TMS from bulk and pharmaceutical formulation. The λ max of TMS in methanol and water was found to be **280 nm** [49]. The same spectrum was derivatised in to first order derivative; showed maximum amplitude of the trough at **299 nm**. The drug follows linearity in the concentration range **10 - 90 µg/ml** with correlation coefficient value 0.997. The value of correlation coefficient (r^2) greater than 0.99 indicate good linearity response in the above-mentioned range. The proposed method was applied to pharmaceutical formulation and % amount of drug estimated **99.58 %** was found in good agreement with the label claim. The accuracy of the method was checked by recovery experiment performed at three different levels i.e., 80%, 100% and 120 %. The % recovery was found to be in the range **99.00%–101.63%**. The low values of % R.S.D. are indicative of the accuracy and reproducibility of the method. The precision of the method was studied as an intra-day, inter-day variations and repeatability. The % R.S.D. value less than 2 indicate that the method is precise. Ruggedness of the proposed method was studied with the help of two analysts. The results did not show any statistical difference between operators suggesting that method developed was rugged.

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