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Analytical Method Development and Validation for Estimation of Tamsulosin Hydrochloride by UV-Spectroscopic method

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Abstract : A Simple, specific, rapid, precise and accurate UV Spectrophotometric method have been developed and Validated for determination of Tamsulosin Hydrochloride. Drug showed the absorption maxima in at 224 nm and was linear for a range of 5 µg/ml -25µg/ml with a correlation coefficient of 0.9997. The validation of the above proposed method was done by carrying out precision and accuracy studies. The percentage recovery at three different levels i.e. 50%, 100% and 150% was found to be 50.1%, 99.2% and 149.2% respectively. The analytical method showed good Intra precision (repeatability) with relative standard deviation 0.306 % and Inter precision with relative standard deviation is 0.411% which is less than 2. The proposed method was validated for the parameter Specificity, Precision, Linearity and range, accuracy and recovery. Hence proposed analytical method for estimation of Tamsulosin Hydrochloride formulation drug by UV spectrophotometer in pharmaceutical can be applied for the routine quality control analysis.
Keywords : Validation, Tamsulosin Hydrochloride, UV Spectrophotometer.

Introduction

Tamsulosin Hydrochloride is a alpha blocker in blood vessels used to treat hypertension usually in conjunction with diuretics when other treatments are ineffective the IUPAC name 5-[(2R)-2-[[2-(2-Ethoxyphenoxy)ethyl]amino] propyl]-2-methoxybenenesuphonamide hydrochloride[1-2].Tamsulosin Hydrochloride having molecular formula $C_{20}H_{29}ClN_2O_5S$ and molecular weight 444.97 g/mol. It is official inEuropean [3], British pharmacopoeia and United States [4]with Potentiometric titration method. Literature survey reveals that few analytical methods are available including Potentiometric titration[3-4], HPTLC[5], HPLC[6-12] and UV Spectrophotometry [13-19].No simple and rapid work has been reported for the estimation of Tamsulosin Hydrochloride. All these reported methods either took a long time for analysis or employ mobile phases with pH adjustment of Buffer solutions which is tedious and anomalous[5-19], especially for routine testing of quality control samples of assay content study. Hence it was felt necessary to build up a simple, rapid, economical and precise Spectrophotometric method for the direct quantitative determination of Tamsulosin Hydrochloride. The current research work deals with the development of UV Spectrophotometric method and its validation as per International Conference on Harmonization (ICH) guideline [20-21]. The developed method was found to be simple, specific, stable, rapid, accurate, precise, reliable, less expensive and timesavingbyUV Spectrophotometric method[13-19] for the estimation of Tamsulosin Hydrochloride in formulation drug.

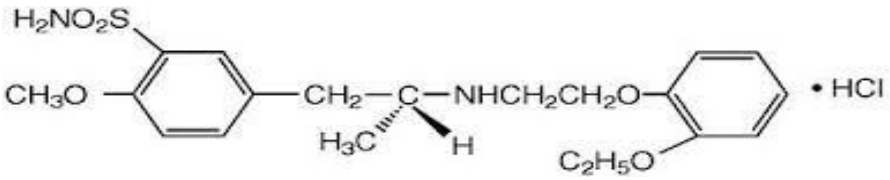

Figure 1: Chemical structure of Tamsulosin Hydrochloride

Materials and Methods

Instrumentation and Materials:

U.V. visible double beam spectrophotometers SL 210 Elico with Spectra treat software having path length 1cm U.V. matched quartz cells were used.Tamsulosin Hydrochloride was a gift sample and Standard from Cure worth Pharmaceuticals, Mumbai and Omicron Pharmaceuticals Surat Gujarat.All chemicals, solvents and reagents i.e. Sodium hydroxide, Perchloric acid 70% and Methanol used, were analytical grade and purchased from S.D. Fine Chem Ltd/Merck Ltd, India.

Method Development:

Preparation of Diluent Solution

Transfer about700 ml of water to the 1000 ml volumetric flask, then slowly add about 1.0 gm of Sodium hydroxide with stirring and add 3.0 ml 70% Perchloric acid, stir and, mix well to dissolve completely then with constant stirring slowly add Methanol up to mark to make volume 1000 ml. use this solution as diluent.

Preparation of Standard Solution

Weighed accurately about 120 mg of Tamsulosin Hydrochloride and transferred to 200ml volumetric flask. Dissolved in Diluent and made up the volume to 200 ml, further transferred 5 ml of solution to 200 ml volumetric flask. Made volume up to mark to get a concentration of 15 µg/ml.

Selection of wavelength for analysis of Tamsulosin Hydrochloride

The standard solution having concentration 15 µg/ml was scanned at 200 nm to 400 nm with diluent as the blank to detect maximum wavelength (Figure-2).

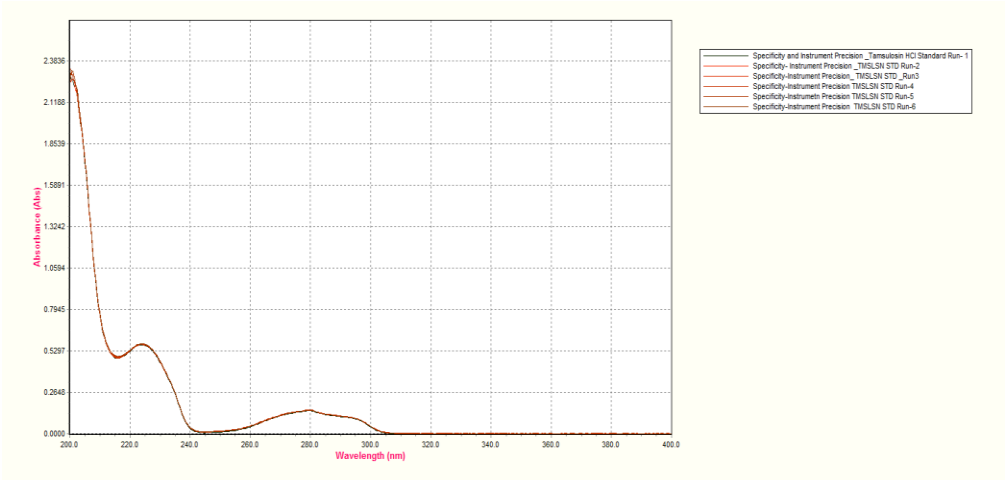

Figure 2: Estimation of Maxima of Tamsulosin Hydrochloride

From the above (Figure-2) spectra of Tamsulosin Hydrochloride wavelength maxima identified for quantification were 224 nm (λmax).

Validation of proposed AnalyticalMethod

The proposed method was validated according to International Conference on Harmonization (ICH) guidelines for validation of analytical procedures[20-21]. Analysis of variance (ANOVA) was used to verify the validity and performance effectiveness of the proposed analytical methods.

Specificity

Specificity is the ability to assess unequivocally the analyte in the presence of components which may be expected to be present. Typically these might include impurities, degradants, matrix, etc. Specificity was done by scanning of Diluent solution and Standard solution of Tamsulosin Hydrochloride having concentrations 15 µg/ml in Spectrophotometric range from 200 nm to 400 nm to check specific absorption maxima at predefined wavelength i.e. 224nm and Standard solution stability study done to evaluate the solution stability at different time interval up to 24 hrs.

Instrument Precision

Instrument precision was performed to check the suitability of the developed analytical method with respect to ability of instrument consistency to provide the precise wavelength maxim when scanned the Standard solution of Tamsulosin Hydrochloride having concentrations 15 µg/ml in the UV range from 200 nm to 400 nm. To check specific absorption maxima at predefined wavelength 224 nm with reproducible absorption detection. Six separated standard preparations were scanned / analyzedaccording to the proposed method of analysis. The % RSD due to Tamsulosin Hydrochloride concentration for the six standards was found 0.599%. The % RSD due to Tamsulosin Hydrochloride concentration for the instrument precision meets the requirements. Results are tabulated in the Table 1.

Table 1. Instrument Precision

Sr. No.	**Standard number**	**Absorbance @224 nm**	**% RSD**
1	Standard Preparation -1	0.5675	0.599% Limit < 2%
2	Standard Preparation -2	0.5731	
3	Standard Preparation -3	0.5738	
4	Standard Preparation -4	0.5736	
5	Standard Preparation -5	0.5690	
6	Standard Preparation -6	0.5768	
Average Absorbance →		0.5723	

Linearity and Range

The linearity of an assay method is its ability to elicit test results, which are directly proportional to the concentrations of drug in samples in a given range. Linearity justifies the use of single-point calibrations. The correlation coefficient of theRegression line for was found that 0.9997.

Five levels of five different concentrations Standard solution of Tamsulosin Hydrochloride having concentrations 5 µg/ml, 10 µg/ml, 15 µg/ml, 20 µg/ml and 25 µg/ml, in the range relative to the working concentrations, were prepared and read according to the method of analysis. A linear regression curve was constructed, the correlation coefficient (R2) and assessment value calculated. The correlation coefficient (R2) for Tamsulosin Hydrochloride obtained is 0.9997. The plot is a straight line and the results are tabulated in the Table 2 and Curve is shown in the Figure 3.

Table 2. Linearity and Range

Sr. No.	StandardConcentration(µg/ml)	Absorbance@224 nm	Correlation coefficient
1	5	0.2000	0.9997 Limit ≥0.999
2	10	0.4068	
3	15	0.5990	
4	20	0.8114	
5	25	0.9987	

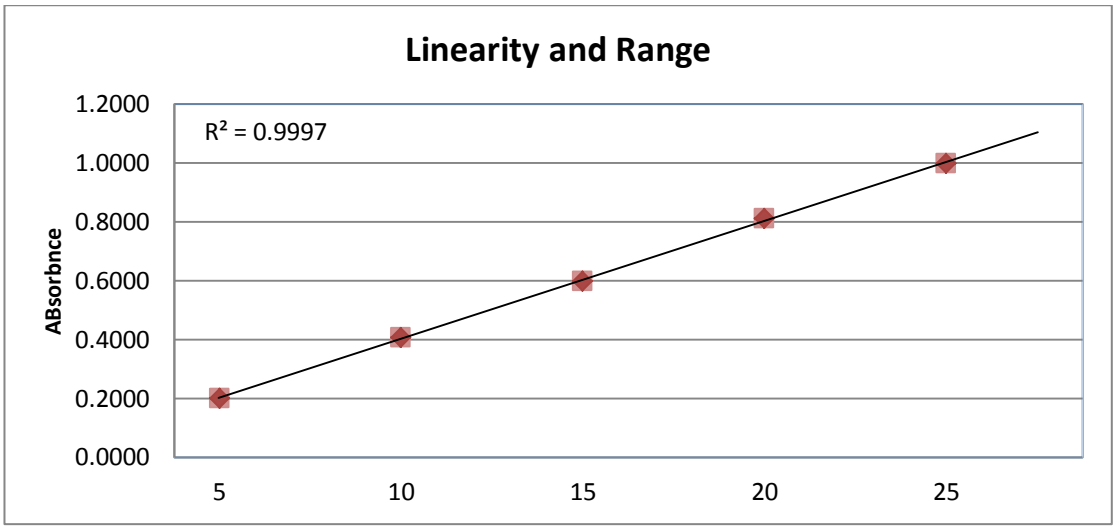

Figure 3: Linearity and Range of Tamsulosin Hydrochloride

Analytical Method Precision

The precision of an analytical procedure expresses the degree of agreement among individual test results when the method is applied to multiple sampling of a homogenous sample.

Intra Precision (Repeatability*)*

This parameter determines the repeatability of assay results under the same operating conditions over a short period of time. The % RSD due to Tamsulosin Hydrochloride concentration for the six samples was found to be 0.306%. Six separated sample preparations were analyzed according to the proposed method of analysis. The % RSD due to Tamsulosin Hydrochloride concentration for the assay meets the requirements. Results are tabulated in the Table 3.

Table 3. Intra Precision (Repeatability) Results

Sr. No.	Sample number Results	Tamsulosin Hydrochloride % Assay content	% RSDof Six Assay content
1	Sample Preparation -1	99.3	0.306% Limit < 2%
2	Sample Preparation -2	99.9	
3	Sample Preparation -3	99.9	
4	Sample Preparation -4	99.3	
5	Sample Preparation -5	99.9	
6	Sample Preparation -6	99.9	
Average % Assay ⟶		99.7	

Inter Precision (Repeatability*)*

This parameter determines the Intermediate repeatability of assay results under the same operating conditions test performed on a different day, using different makes of reagents and solvents. The % RSD due to Tamsulosin Hydrochloride concentration for the six samples was found to be 0.411%. Six separated sample preparations were analyzed according to the proposed method of analysis. The % RSD due to Tamsulosin Hydrochloride concentration for the assay meets the requirements. Results are tabulated in the Table 4.

Table 4. Inter Precision (Repeatability) Results

Sr. No.	**Sample number Results**	**Tamsulosin Hydrochloride% Assay content**	**% RSDof Six Assay content**
1	Sample Preparation -1	99.9	0.411% Limit < 2%
2	Sample Preparation -2	99.8	
3	Sample Preparation -3	99.9	
4	Sample Preparation -4	100.2	
5	Sample Preparation -5	99.1	
6	Sample Preparation -6	100.3	
	Average % Assay ⟶	99.9	

Ruggedness

Ruggedness of the method was determined by carrying out the analysis on different days, different makes of reagents and solvents. The respective test assay results of Tamsulosin Hydrochloride having concentration as 15 µg/ml was illustrious. The result is expressed as shown in table 4. The developed method for estimation of Tamsulosin Hydrochloride was found to be rugged as Shown in table 5.

Table 5. Ruggedness

Sr. No.	**Precision**	**% RSD of assay of Six Preparation**	**Limit For Ruggedness**
1	Intr**a** Precision	0.306	NMT 2%
2	Inter Precision	0.411	
	% RSDof Overall 12 Assay content	0.365	

Accuracy

This parameter determines the accuracy of the assay results under the same operating conditions test.

A sample was constituted analyzed for the accuracy with known quantity of standard samples of Tamsulosin Hydrochloride at 50%,100%, 150% concentration levels and assayedas per the method stated under analyticalMethods respectively. Three determinations were performed under each concentration levelsrespectively. Results are shown in Tables 6,7,8. The % RSD due to recovery of Tamsulosin Hydrochloride at 50%,100%, 150% concentration levels was found to be 0.271%, 0.407% and 0.422% respectively. Nine sample preparations were analyzed according to the proposed method of analysis. The % RSD due to Tamsulosin Hydrochloride concentration for the assay meets the requirements and within 98.0% to 102%. Results are tabulated in the Table 6, 7, 8.

Table 6. Accuracy and Recovery Results @ 50 % Concentration level

Sr. No.	**Accuracy** @ 50% **level**	**Recovery of Tamsulosin Hydrochloride % Assay content**	**% Recovery** 98.0% to 102%	**% RSD**
1	Sample Preparation -1	50.2	100.2	0.271% Limit < 2%
2	Sample Preparation -2	50.1		
3	Sample Preparation -3	50.1		
Average % Assay →		50.1		

Table 7. Accuracy and Recovery Results @ 100 % Concentration level

Sr. No.	**Accuracy @ 100% level**	**Recovery of Tamsulosin Hydrochloride %Assay content**	**% Recovery** 98.0% to 102%	**% RSD**
1	Sample Preparation -1	99.5	99.2	0.407% Limit < 2%
2	Sample Preparation -2	99.4		
3	Sample Preparation -3	98.8		
Average % Assay →		99.2		

Table 8. Accuracy and Recovery Results @ 150 % Concentration level

Sr. No.	**Accuracy @ 150% level**	**Recovery of Tamsulosin Hydrochloride%Assay content**	**% Recovery** 98.0% to 102%	**% RSD**
1	Sample Preparation -1	148.6	99.5	0.422% Limit < 2%
2	Sample Preparation -2	149.2		
3	Sample Preparation -3	149.8		
Average % Assay →		149.2		

Solution Stability of Standard Solution

Solution stability of the standard solution performed up to 24 hrs with different time interval and found the solution is stable showing cumulative % RSD of different time interval is 0.698 which is less than the 2. Hence the Tamsulosin Hydrochloride solution is stable up to 24 hrs at room temperature.

Results and Discussion

The method discussed in the present work provides a simple, stable, rapid, accurate, precise, reliable, less expensive (Economical), time *saving* and convenient method for the analysis of Tamsulosin Hydrochloride using U.V. Spectrophotometry. λmax selected for quantitation was 224 nm. In the developed analytical method, the linearity was observed 0.9997 in the concentration range of 5µg/ml -25 µg/ml.

Method precision for the Tamsulosin Hydrochloride at concentrations level 15 µg/ml was found in the range of 99.1%-100.3%. Accuracy of the proposed method was ascertained by recovery studies and the results were expressed as percent recovery and were found in the Range of 98.8%-100.4%.Values of standard deviation and coefficient of variance was satisfactorily indicating the accuracy of both the methods. Intra-day and Inter-day precision studies were carried out by analyzing the sample of Tamsulosin Hydrochloride at different time interval on the same day and on different days respectively. Standard deviation and coefficient of variance for Intra-day and Inter-day precision studies was found to be less than 2 indicating precision of the proposed method.

Based on the outcome of analytical method development and analytical validation study test results, it was found that, the proposed analyticalmethod for estimation of Tamsulosin Hydrochloride by UV Spectrophotometry is Accurate, Precise, Reproducible, Stable, Simple, Rapid Time saving andless expensive (Economical).The analytical method can be employed for routine quality control of Tamsulosin Hydrochloride in pharmaceutical analysis.

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