Research Article

Method Development and Validation of Different Brands of Tamsulosin Hydrochloride Capsules by Spectro Flourimetric Method

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ABSTRACT

The objective of this study was to develop and validate the spectroflourimetry method for the quantitative determination of Tamsulosin Hydrochloride in different brands of capsule dosage form (URIMAX 0.4 mg, DYNAPRESS 0.4 mg, URIPRO 0.4 mg). The solvent systems and wavelengths of detection were optimized in order to maximize the sensitivity and minimize the cost of analysis. The excitation and emission wavelengths were found to be 226 nm and 322 nm in 0.1N H_2SO_4. This research paper describes a simple, precise, inexpensive, eco-friendly and a rapid method with good sensitivity. Validation was carried out in compliance with International Conference on Harmonization guidelines.

Keywords: Tamsulosin hydrochloride, Spectroflourimetry, 0.1N Sulphuric acid, Schiffs Reagent.

INTRODUCTION

Tamsulosin hydrochloride (TAM) is a selective antagonist of alpha-1A and alpha-1B adreno receptors at the prostate. It works by blocking α – receptor that are found in the muscle of the prostate gland, which cause the muscle in the prostate to relax. Tamsulosin is given by mouth as hydrochloride salt. Absorption of Tamsulosin from capsules is of > 90%. Tamsulosine is not intended for use as an antihypertensive agent. It is absorbed from the g.i tract and is almost completely bio available[1]. Chemically it is 5-[(2 R)-2-[2-(2-ethoxy phenoxy) ethyl amino] propyl]-2methoxy benzene sulfonamide[2].

MATERIALS AND METHODS

Pure Tamsulosin hydrochloride was received as a gift sample from Pharma Nostra, Brazil and used as such without further purification. URIMAX 0.4 mg, DYNAPRESS 0.4 mg, URIPRO 0.4 mg were purchased from local markets of Manjeri and Calicut. All chemicals were of analytical grade.

Instrument

All spectral measurements were done on Systronics 119 UV/ visible spectrophotometer connected to 230v Ac main.

Reagents

0.1N H_2SO_4, schiffs reagent.

Methodology

The different brands of Tamsulosin hydrochloride were validated by the method given below.

Working standard of drug solution - A standard stock solution was prepared by dissolving 10 mg of pure drug tamsulosin hydrochloride (100 µg/ ml) in 5 ml of 0.1N H_2SO_4 in a 100 ml volumetric flask. Aliquots of standard stock solutions were prepared by suitably diluted with 0.1N H_2SO_4 to obtain the final concentration.

Method Validation

As per ICH guide lines (Q2 (R1) (ICH, 2005) the following method was validated[5,8].

The proposed method obeys Beer Lamberts law in the concentration range of 0.1 to 0.5 µg/ml. In this method the correlation co-efficient (R2) was found to be 0.999.

Accuracy: 20 capsules were taken from each marketed preparation. Recovery studies were carried out by addition of standard drug to the sample at three different concentration. Samples were prepared in triplicate at levels 80%, 100%, 120% of the test concentrations using the working standard of four different brands as per the proposed methods. Absorbance of each solution performed in triplicate.

Linearity: Various aliquots were prepared from the stock solution (1000 µg/ml) ranging from 0.1 to 0.5 µg/ml. The samples were scanned in UV spectrophotometer against sulphuric acid, Schiff's base and water. It was found that the selected capsules of different brand shows linearity between of 0.1 to 0.5 µg/ml.

Range: Linearity of range of the proposed method was calculated by plotting the fluorescence intensity versus concentration[4].

Precision: The experiment was repeated three times in a day to determine intra-day precision and on three different days to determine inter-day precision. six different solutions of same concentration were analyzed three times in a day and the absorbance was noted in intraday and in the interday, solution of the same concentration were analyzed three times for 3 repeated days and the mean, standard deviation and percentage relative standard deviation of absorbance was recorded[4,6,7].

Specificity: 1 mg of tamsulosin was spiked with 5 %, 10 % and 15 % sample and was analyzed for % recovery of tamsulosin.

Robustness: This method helped to determine the analysis at different temperature condition[5]. The result of absorbance at 0.1 µg/ml was noted and the result was indicated as % RSD.

Ruggedness: It can be determined by carrying out the analysis by different analyst. The absorbance at 0.1 µg/ml was noted and the result was indicated as % RSD.

Limit of detection: The limit of detection was determined from solutions of different conc. ranging from 0.1-0.5µg/ml. The lowest amount of analyte in a sample which can be detected shows the limit of detection.

Limit of Quantification: The limit of quantification is the concentration that can be quantified with a specified level of accuracy and precision.

For sample Preparation

Different marketed formulations were performed for recovery studies. The capsules were weighed accurately. The capsule content was emptied and weight of empty capsule shells was taken. The difference of whole capsule and empty shells gave the weight of the powder. The powder equivalent to 100 mg was transferred to 100ml volumetric flask. Finally it will be diluted with 0.1N H_2SO_4 for getting suitable concentration.

Assay method

Aliquots of Tamsulosin ranging from 0.1 to 0.5 ml were transferred to a series of 10 ml volumetric flask. To each flask add 5ml of 0.1 N H_2SO_4 and add 1 ml Schiff's reagent and it will be placed in ice bath for 45 minutes. Finally it will be diluted with 0.1N H_2SO_4 for getting suitable concentrations. The absorbance of suitable dilution was made against the blank. The amount of the drug in the sample solution was computed from calibration curve.

Results of the present experiment provided in Graph 1 - Graph 3 and in Table 1 - Table 6.

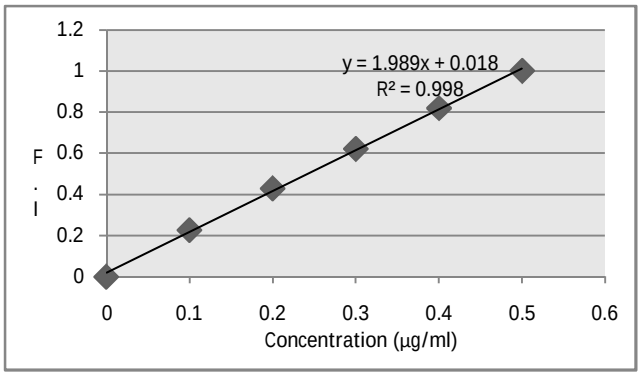
RESULTS AND DISCUSSION

The methods discussed in the present work provide a convenient and accurate way for analysis of different brands of Tamsulosin in different pharmaceutical dosage forms. The developed method was found to be precise as the % RSD values for the intraday and inter day were found to be less than 2%. In order to justify the reliability and suitability of the proposed method known quantities of pure Tamsulosin was added to various pre analyzed formulations, and the mixture were analyzed by the proposed method. Validation parameters, include, LOD, LOQ accuracy, precision etc were calculated.

The developed method was validated as per ICH guidelines and validation parameters are summarized below.

Table 1: Statistical parameters from the calibration plot of URIPRO 0.4 mg

Concentration (µg/ml)	Fluorescence intensity	SD	LOD	LOQ
0.1	0.226	0.0022	0.003 µg/ml	0.011 µg/ml
0.2	0.429			
0.3	0.62			
0.4	0.818			
0.5	0.999			



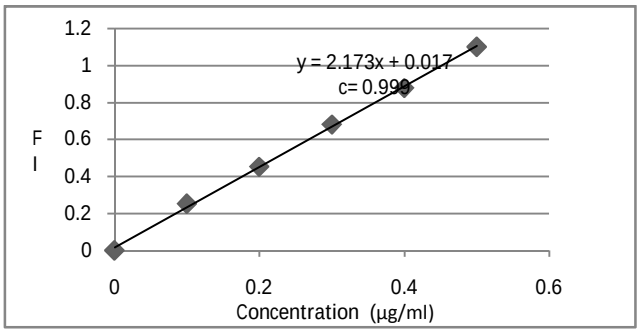
Graph 1: Graph of URIPRO 0.4 mg (Concentration VS Fluorescence intensity

The drug obeys Beer-Lambert's law. The r R^2 value was of 0.998.

Table 2: Validation parameters of URIPRO 0.4 mg

Validation parameter	Values
Linearity	0.1-0.5µg/ml
L.O.D	0.003µg/ml
L.O.Q	0.011µg/ml
Range	0.1-0.5µg/ml
R.S.D	0.990
Accuracy	99.65±4.5%
Precision (R.S.D)	
Interday (n=6)	0.13-0.35
Intraday (n=6)	0.262-0.375
Ruggedness (R.S.D)	
Analyst(1)	0.546
Analyst(2)	0.522
Robustness(R.S.D)	
Ac temp	0.965
Room temp	0.963

The drug shows linearity in the range of 0.1-0.5µg/ml. The interday precision ranges from 0.13-0.35 and the intraday precision ranges from 0.262-0.375.



Graph 2: Graph of URIMAX 0.4 mg (Concentration VS Fluorescence intensity

Table 3: Statistical parameters from the calibration plot of URIMAX 0.4 mg

Concentration (µg/ml)	Fluorescence intensity	SD	LOD	LOQ
0.1	0.252	0.01	0.015 µg/ml	0.046 µg/ml
0.2	0.452			
0.3	0.680			
0.4	0.878			
0.5	1.100			

The drug obeys Beer-Lambert's law. The R^2 value was of 0.999.

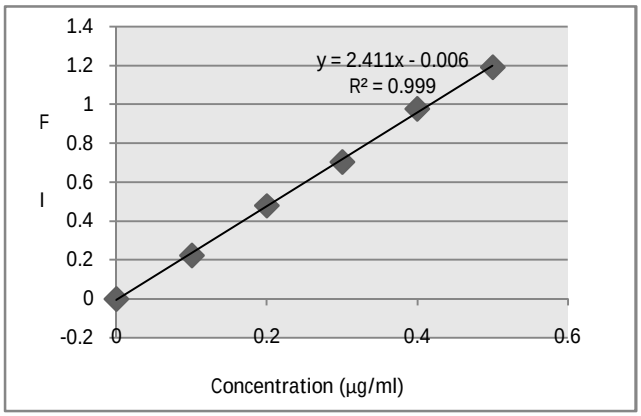
Table 4: Validation parameters of URIMAX 0.4 mg

Validation parameter	Values
Linearity	0.1-0.5µg/ml
L.O.D	0.015µg/ml
L.O.Q	0.046µg/ml
Range	0.1-0.5µg/ml
Accuracy	99.89±0.5%
Precision (R.S.D)	
Interday (n=6)	0.18-0.39
Intraday(n=6)	0.272-0.388
Ruggedness (R.S.D)	
Analyst(1)	0.659
Analyst(2)	0.621
Robustness (R.S.D)	
Ac temp	0.889
Room temp	0.886

Table 5: Statistical parameters from the calibration plot of DYNAPRESS 0.4 mg

Concentration (µg/ml)	Fluorescence intensity	SD	LOD	LOQ
0.1	0.224	0.0 34	0.011 µg/ml	0.038 µg/ml
0.2	0.479			
0.3	0.705			
0.4	0.977			
0.5	1.191			

The drug shows linearity in the range of 0.1-0.5µg/ml. The interday precision ranges from 0.18-0.39 and the intraday precision ranges from 0.272-0.388. Accuracy is of 99.89±0.5%



Graph 3: Graph of DYNAPRESS 0.4 mg (Concentration VS Fluorescence intensity)

The drug obeys Beer-Lambert's law. The R^2 was of 0.999.

Table 6: Validation parameters of DYNAPRESS 0.4 mg

Validation parameter	Values
Linearity	0.1-0.5µg/ml
LOD	0.011µg/ml
LOQ	0.038µg/ml
%R.S.D	0.95
Range	0.1-0.5µg/ml
Accuracy	99.78±1.1%
Precision (R.S.D)	
Interday (n=6)	0.19-0.42
Intraday (n=6)	0.282-0.389
Ruggedness (R.S.D)	
Analyst(1)	0.831
Analyst(2)	0.866
Robustness (R.S.D)	
A.c temp	0.963
Room temp	0.959

The drug shows linearity in the range of 0.1-0.5µg/ml. The interday precision ranges from 0.18-0.39 and the intraday precision ranges from 0.272-0.388. Accuracy is of 99.78±1.1 %

CONCLUSION

The proposed methods were simple, sensitive and reliable with good precision. The reagents utilized in the proposed methods are cheap, readily available and the procedure involved having no critical criteria. This validation assures that the proposed developed procedure have a remarkable role in routine quality control tests.

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