RP – HPLC method for the estimation of Tamsulosin Hydrochloride in Tablet Dosage Form

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A rapid and sensitive reverse phase RP-HPLC method is proposed for the estimation of tamsulosin hydrochloride in tablets. Tamsulosin hydrochloride was chromatographed on a reverse phase C18 column with a mobile phase consisting of acetonitrile and water in the ratio of 50:50 v/v. The mobile phase was pumped at a flow rate of 1.5 ml/min. The eluents were monitored at 214 nm. The retention time of the drug was 1.7 min. With this method, linearity was observed between area under curve and concentration of tamsulosin hydrochloride in the injected solution, in the range of 5 to 100 µg/ml. The method was found to be applicable for analysis of the drug in tablets. The results were validated statistically.

Key words: RP-HPLC, stability indicating, tamsulosin HCl, validation

Tamsulosin Hydrochloride is chemically 5-{(2R)-2[{2-(2-ethoxyphenoxy)ethyl} amino]propyl}-2-methoxybenzene sulfonamide[1-5]. It is an α1 adrenoceptor blocking agent, exhibit selectivity for α1 receptors in the human prostate[6-8]. It brings about relaxation of prostatic and urethral smooth muscle. Literature survey reveals that the few techniques like HPLC, chiral LC method and non-aqueous potentiometric methods have been reported for the estimation of tamsulosin hydrochloride[9-11]. The focus of present study was to develop and validate a rapid, stable and economic RP-HPLC method for the estimation of tamsulosin hydrochloride in pharmaceutical dosage forms.

A binary gradient RP-HPLC with Shimadzu LC-10AT and LC-10AT VP series HPLC pumps, a 20 µl sample injection loop (manual) and SPD 10A VP UV/Vis absorbance detector was used for the study. The output signal was monitored and integrated using Shimadzu CLASS-VP Version 6.12 SP1 software.

A pure sample of tamsulosin hydrochloride was procured from Cipla. Tamsulosin hydrochloride tablets were purchased from a local pharmacy. The mobile phase consists of acetonitrile and water (50:50 v/v) was selected as it was found to ideally resolve the peaks of tamsulosin hydrochloride. The components of the mobile phase were pumped to the column at a flow rate of 1.5 ml/min that produced column back pressure of 195 kgf/cm^2. The eluents were monitored at 214 nm having ambient column temperature. Acetonitrile and water used were of HPLC grade (Merck) and all other reagents were of AR grade. Mobile phase was prepared by mixing 500 ml of acetonitrile with 500 ml of water. The mobile phase was sonicated for 15 min and then it was filtered through a 0.45 µ membrane filter paper. About 10 mg of standard tamsulosin hydrochloride was accurately weighed and taken in 100 ml volumetric flasks separately and dissolved in the mobile phase. The volume was adjusted up to the mark with mobile phase to give stock solutions of 100 µg/ml. The standard stock solution was filtered through a 0.22 µ membrane filter paper. To determine the content of tamsulosin hydrochloride in conventional tablets, twenty tablets were weighed; their average weight was determined and finely powdered. The tablet powder equivalent to 10 mg of tamsulosin hydrochloride was weighed and transferred to a 100 ml volumetric flask and dissolved in 50 ml of mobile phase. The solution was ultrasonicated for 20 min and filtered through 0.22 µ membrane filter paper. The sample solution was further diluted with mobile phase to obtain sample solutions within the Beer Lambert's range for the drug solution.

Linearity was determined by taking appropriate aliquots of the drug from the standard stock solution into a series of 10 ml volumetric flasks. It was diluted up to the mark with mobile phase in such a way that final concentrations of tamsulosin hydrochloride was in the range of 5-100 µg/ml. Triplicate dilutions of each

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concentration of the drug were prepared separately. From these triplicate solutions, 20 µl injections of each concentration of the drug were injected into the HPLC system two times separately and chromatographed under the optimized conditions. Evaluation of the drug was performed with the UV detector set at 214 nm and peak areas were recorded. The plots of peak area Vs respective concentration of tamsulosin HCl was found to be linear in the range of 5-100 µg/ml. The specificity of the RP-HPLC method was determined by complete separation of tamsulosin HCl as shown in (fig. 1).

Also parameters like retention time (Rt), resolution (R_s) and tailing factor (T) of tamsulosin HCl were evaluated. The precision of the method was determined using the standard solutions of tamsulosin HCl. Standard solutions of concentrations, 20 and 40 µg/ml prepared in triplicate were used for studying the degree of precision of the developed method[12-15]. Repeatability of the assay was determined by duplicate injections of the prepared solutions in the same day for the intraday precision (Table 1). To check the accuracy of the proposed method, recovery studies were carried out by applying the standard addition method. A known amount of standard tamsulosin HCl corresponding to 80, 100 and 120% of the label claim was added to preanalysed sample of tablet dosage form. The recovery studies were carried out three times, at each level of recovery (Table 2).

The LOD and LOQ were separately determined (Table 3) based on the standard deviation of response of the calibration curve. The standard deviation of y-intercepts of regression lines and slope of the calibration curve may be used to calculate LOD and LOQ. LOD= 3.3×D/S and LOQ= 10×D/S, where, D is the standard deviation of the y-intercepts of regression line and S is the slope of the calibration curve.

Optimization of mobile phase was performed based on resolution, asymmetric factor and peak area obtained. Overlain UV spectra of tamsulosin showed that drug absorbs at 214 nm. This wavelength was selected as the detection wavelength. Calibration curve was obtained by plotting peak area versus concentration and it was found to be linear.

The amount of drug found was between 99-101%. The sample recoveries in formulations were in good agreement with their respective label claim which suggested non-interference of formulation excipients in the estimation. The lower values of % RSD in

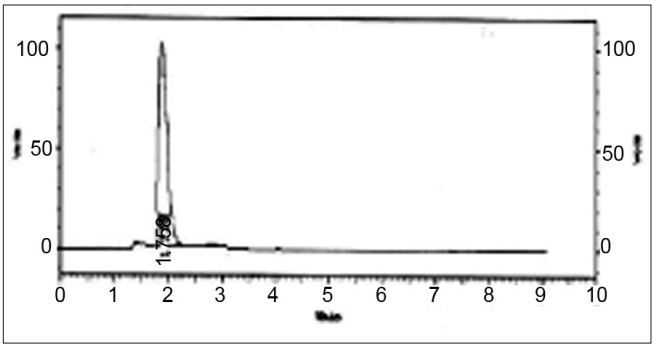

Fig. 1: Typical chromatogram of tamsulosin HCl

TABLE 1: STATISTICAL EVALUATION OF TABLET ANALYSIS

Tablet formulation	% Mean*	±SD	% RSD
T1	99.68	0.64	0.6420
T2	99.87	0.21	0.2102

*Mean of six determinations (n=6), T1 and T2 are two different brands of tablet formulations

TABLE 2: STATISTICAL DATA FOR RECOVERY STUDIES

Tablet formulation	Type of recovery in %	% Mean*	SD	% RSD
T1	80	99.73	0.3680	0.3690
	100	99.82	0.4262	0.4269
	120	99.80	0.1883	0.1886
T2	80	100.27	0.161	0.1605
	100	99.87	0.103	0.1031
	120	100.60	0.195	0.1938

*Mean of six determinations (n=6), T1 and T2 are two different brands of tablet formulations

TABLE 3: VALIDATION AND SYSTEM SUITABILITY PARAMETERS

Parameter	Result
Linearity range (µg/ml)	5-100
Regression coefficient (r^2) ±SD	0.9997
Retention time (min)	1.7
Tailing factor	1.33
Theoretical plates	22011
Limit of detection (µg/ml)	0.0190
Limit of quantification (µg/ml)	0.0575

(Table 2) indicate that the method is precise and accurate. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.0190 µg/ml and 0.0575 µg/ml, respectively. Hence it can be concluded that the proposed method is a stability indicating simple, specific, precise and accurate which can be employed for the estimation of tamsulosin HCl in bulk and formulations.

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REFERENCES

1. Budavari S. The Merck Index, 13th ed. Whitehouse Station, NJ: Merck and Co Inc; 2003.
2. Martindale PK. The Complete Drug Reference, 32nd ed. London; The Pharmaceutical Press; 1999.
3. Singh H, Kapoor VK. Medicinal and Pharmaceutical Chemistry. 2nd ed. Delhi; Vallabh Prakashan Publishers, 1996. p. 330.
4. Block J, Beale JM. Wilson and Gisvold's text book of organic medicinal and pharmaceutical chemistry. 8th ed. Philadelphia: Lippincott Williams and Wilkins; 1982. p. 541.
5. Reynolds JEF, Prasad BA. In: Martindale, The Extra Pharmacopoeia, 31st ed. London: The Pharmaceutical Press; 1996.
6. Tripathi KD. Essentials of medical pharmacology. 5th ed. New Delhi: Jaypee Brothers Medical Publishers (P) Ltd; 2003. p. 119-24.
7. Reynolds JEF, Prasad BA. In: Martinadale, editor. The complete drug reference. 33rd ed. London: The Pharmaceutical Press; 1996. p. 978-3.
8. Satoskar RS, Bhandarkar SD, Ainapure SS. Pharmacology and Pharmacotherapeutics. 18th ed. Mumbai: Popular Prakashan; 2003. p. 270.
9. Srinivasu MK, Rao BM, Vittal TV, Rajendra Kumar P, Chandrasekhar KB. A validated chiral LC method for the enantioselective analysis of tamsulosin hydrochloride and its enantiomer (S)-5-[2-[[2-(0-ethoxyphenoxy) ethyl]amino]propyl]-2-methoxy benzenesulfonamide, monohydrochloride on amylose based stationary phase. Indian Drugs 2005;42:230-7.
10. Rao BM, Srinivasu MK, Thilakumar T, More S, Rajendra Kumar P. A Validated non-aqueous potentiometric titration method for the quantitative determination of tamsulosin in tamsulosin hydrochloride bulk drug. Indian Drugs 2005;42:175-7.
11. Zhang Z, Yang G, Liang G, Liu H, Chen Y. Chiral separation of tamsulosin isomers using HPLC using cellulose tris (3,5-dimethylphenylcarbamate) as a chiral stationary phase. J Pharm Biomed Anal 2004;34:689-93.
12. Shinde VM, Khanolkar DH. Simultaneous determination of chlorpropamide and Phenformin hydrochloride from tablets from RP HPLC. Indian Drugs 1998;35:384-6.
13. Palled MS, Rajesh PM, Chitter M, Bhat AR. Reverse phase high performance liquid chromatographic determination of Zidovudine and lamivudine in tablet dosage forms. Indian J Pharm Sci 2005;67:110-2.
14. ICH, Q2B, Validation of Analytical Procedures: Methodology, International Conference on Harmonization: Geneva; 1996.
15. Sethi PD. Quantitative analysis of drugs in pharmaceutical formulations. 3rd ed. New Delhi: CBS Publishers; 2003. p. 548-9.

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