Available online on 15.07.2019 at http://jddtonline.info

Journal of Drug Delivery and Therapeutics

Open Access to Pharmaceutical and Medical Research

Open Access Research Article

UV Spectrophotometric Stability Indicating Method Development and Validation for the Determination of Finasteride Bulk and Dosage Form.

Shraddha T. Nemane, Sachin B. Gholve*, Omprakash G. Bhusnure, Trupti M. Rajmanya, Shivani V. Kaulkhere, Sagar S. Waghmare

Department of Quality Assurance, Channabasweshwar Pharmacy College, Latur - 413512, Maharashtra, India

ABSTRACT

A simple, specific and economic UV spectrophotometric method has been developed using as diluents Methanol to determine the finasteride content in bulk and pharmaceutical dosage formulations. The quantitative determination of the drug has been carried out at a predetermined λmax of 255 nm, it was proved linier in the range 2-12 µg/mL and exhibited good correlation coefficient (R2=0.999) and excellent mean recovery (98-99%). LOQ and LOD were found to be1.178µg/ml and 5.40µg/ml respectively. The method was validated statically and by recovery studies for linearity, precision, repeatability and reproducibility as per ICH guideline. The obtained results proved that the method can be employed for the routine analysis of finasteride in bulk as well as in the commercial formulations.

Keywords: Finasteride, UV Spectroscopy, Method Validation.

Article Info: Received 02 May 2019; Review Completed 22 June 2019; Accepted 27 June 2019; Available online 15 July 2019

Cite this article as:

Nemane ST, Gholve SB, Bhusnure OG, Rajmanya TM, Kaulkhere SV, Waghmare SS, UV Spectrophotometric Stability Indicating Method Development and Validation for the Determination of Finasteride Bulk and Dosage Form., Journal of Drug Delivery and Therapeutics. 2019; 9(4):170-172 http://dx.doi.org/10.22270/jddt.v9i4.3012

***Address for Correspondence:**

Sachin B. Gholve, Department of Quality Assurance, Channabasweshwar Pharmacy College, Latur - 413512, Maharashtra, India

INTRODUCTION

Analytical method development

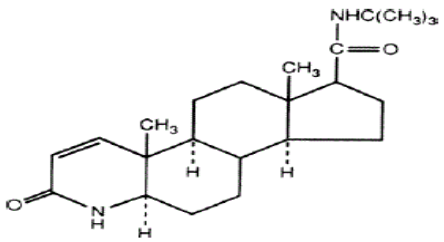
Analytical methods are planned to establish the identity, purity, physical characteristics and potency of the drugs and to support drug testing against specifications during manufacturing and quality release operations as well as during long term stability studies.

Method validation

Validation of an analytical method is the process by which it is established, by laboratory Studies, that the performance characteristics of the method meet the requirements for the Intended analytical applications.

Finasteride chemically (5alpha, 17beta)-(1,1-Dimethylethyl)-3-oxo-4-azaandrost-1-ene-17-carboxamide. Finasteride is an antiandrogen which acts by inhibiting 5-alpha reductase, the enzyme that converts testosterone to dihydrotestosterone. It is used as antiandrogen, Finasteride is used to shrink an enlarged prostate in adults men.Scalp hair loss in men.It can also be used to treat excessive hair growth in women and as a part of hormone therapy for transgender women.

Literature survey reveals HPLC method development for the determination of Finasteride. . In this study, efforts were made to develop a simple, easy and economic UV spectrophotometric method using a diluents methanol for the determination of finasteride in raw material as well as in the marketed dosage formulations. The developed method was optimized and validated as per the guidelines of International Council on Hormonisation (ICH) and demonstrated excellent specificity, linearity, precision and accuracy for finasteride.



Chemical structure of Finasteride

MATERIALS AND METHODS

Instruments

A Shimadzu UV–visible spectrophotometer (UV1800, Shimadzu Corporation, Kyoto, Japan) was used for all absorbance measurements with matched quartz cells.

Materials

All chemicals and reagents were of analytical grade. Finasteride in the form of powder with certificate of analysis was provided Arti pharmaceutical Pvt. Ltd, Mumbai.

Determination of wavelength of maximum absorption

A standard stock solution of Finasteride (100 µg/mL) was prepared using diluents to further obtain 10 µg/mL. An UV spectroscopic scanning (200-400 nm) was carried out with final diluted solution to determine λmax for the detection of finasteride using diluents as a blank.

Linearity and Range

For linearity study, six solutions at different concentrations (2, 4, 6, 8, 10 and 12 µg/mL) were prepared using six different aliquots of stock solution, and the obtained data were used for the linearity calibration plot. Limit of detection (LOD) and limit of quantification (LOQ) for the assay were also calculated.

Intra-day precision (repeatability) and inter-day precision study (intermediate precision)

Finasteride 100 µg/mL was prepared following the same dilution pattern of stock solution. Three different aliquots of stock solution were then diluted to 10 mL to obtain the concentrations of 4, 8 and 12µg/mL. This procedure was repeated in the different days.

Stability study

Samples prepared for repeatability study were preserved for 24 h at room temperature and analyzed on the following day to test for short-term stability.

Accuracy/recovery study

This study was carried out using tablet of Finasteride. Calculation was done from the label claim and the average weight of the final product. Previously used dilution pattern was followed for the tablet to obtain three concentrations—80%, 100% and 120% of reference solution.

Specificity in the presence of excipients

The test for the specificity was carried out using only excipients. Spectra for placebo tablet, blank, and sample were compared. Secondly the specificity was determined by subjecting the sample solution to accelerated degradation by heat (60 °C) for 48 h in order to verify that none of the degradation products interfered with the quantification of the drug.

Assay of content of Finasteride in selected marketed brands

Market brands of Finasteride tablet from different manufacturers were randomly selected and analyzed using the newly developed and validated method. Sample solutions of brand (10 mg/mL) were also prepared and assayed for content of finasteride against the standard. The content of finasteride in the marketed brands was determined using standard calculations.

Stress Degradation Studies

Acid degradation

The preparation of 0.01N hydrochloric acid (HCl) was done by diluting 0.085 ml of conc. HCl to 100 ml of distilled water was accurately weighted and was transferred to a labeled round bottomed flask. Take 10mg of drug and add 5ml of methanol makeup upto 10ml with 0.01N HCL. Reflux the sample for 2 hrs. And pipette out 1ml to10 ml volumetric flask. Make the final dilution so as to get final concentration 10ppm. Take the reading on UV. Check the degradation behavior of drug.

Base degradation:

The 0.01N Sodium Hydroxide (NaOH) was prepared by dissolving 0.04 gm of sodium hydroxide pellets in 100 ml of distilled water. The solution was standardized with 0.01 N HCl as per Indian Pharmacopoeia (I.P).

Finasteride was accurately weighted and was transferred to a labeled round bottomed flask. . Take 10mg of drug and add 5ml of methanol makeup up to 10ml with 0.01N NAOH. Reflux the sample for 2 hrs. And pipette out 1ml to 10 ml volumetric flask and adjust with mobile phase. Make the final dilution so as to get final concentration 10 ppm. Take the reading on UV. Check the degradation behavior of drug.

Neutral condition:

Weight accurately 10 mg drug and transferred in to100 ml water in round bottom flask. Take 10mg of drug and add 5ml of methanol makeup up to 10ml with distil water. Reflux it for 2 hours. Pipette out 1ml in to 10 ml volumetric flask and adjust with mobile phase. As well as also prepare the 10µg/ml sample solution. Take the reading on UV. Check the degradation behavior of drug.

Photo stability study:

Photo stability was performed by placing 10 mg of Finasteride in daylight for 8 -12 hours. The samples were diluted with methanol up to 10ml in a volumetric flask. Pipette out 1ml sample diluted up to 10 ml by mobile phase As well as also prepare the 10µg/ml sample solution. Take the reading on UV. Check the degradation behavior of drug.

Dry heat:

Standard Finasteride was placed in an oven at 600ºC for 2 hours to study dry heat degradation. 10 mg drug samples were diluted with methanol up to 10ml in a volumetric flask. Pipette out 1 ml and were diluted up to 10 ml by mobile phase. As well as also prepare the10 µg/ml sample solution. Take the reading on UV. Check the degradation behavior of drug.

Oxidation Study

Weight accurately 10 mg drug and transferred in 100 ml round bottom flask and added 3% w/v hydrogen peroxide. Keep it at dark place for 2 hours. Pipette out 1ml in to 10 ml volumetric flask and adjust with mobile phase. As well as also prepare the 10µg/ml sample solution. Take the reading on UV. Check the degradation behavior of drug.

RESULTS AND DISCUSSION

Method development and optimization

An accurately weighed quantity of finasteride (10 mg) transferred into a 100 ml volumetric flasks, dissolved well and diluted to the mark with Methanol to obtain standard solution having concentration of (100 µg/ml). A 1 ml of solution transferred into a 10 ml volumetric flasks and diluted to the mark with finasteride to obtain the solutions having the concentrations of 10µg/ml for finasteride. The pre-determined wavelength of maximum absorption (λmax) was 255 nm. (Fig. 2)

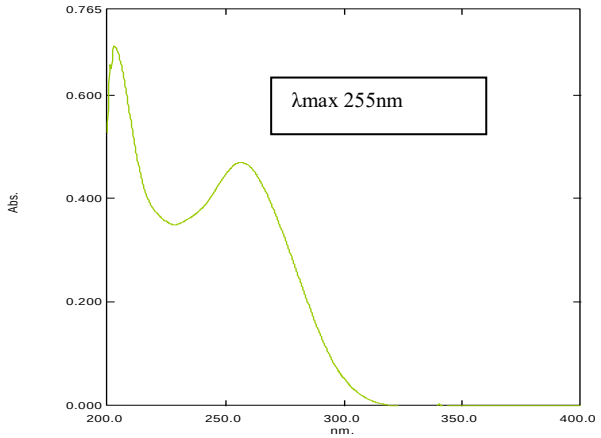


Figure 2: UV Spectrum of Finasteride

Method validation

Linearity and range

The calibration curve obtained was evaluated by its correlation coefficient. The absorbance of the samples in the range of 2.0–12.0 mg/mL was linear with a correlation coefficient (R2) was obtained 0.999 (Table 1). The LOD and LOQ were calculated as 1.178µg/ml and 5.40µg/ml respectively.

Table 1: Linearity data

Concentration µg/ml	Absorbance
2	0.074
4	0.177
6	0.282
8	0.425
10	0.483
12	0.585

Intra-day and inter-day precision

The intra-day and inter-day precision study (Table 2) of the developed method confirmed adequate sample stability and method reliability where all the RSDs were less than 2%.

Stress Degradation Studies

Stability studies results were studied as stress degradation studies (Table 3) of the developed method to confirmed and checked adequate sample stability in different conditions.

Table 2: Intra-day and inter-day precision determined for three different concentrations of Finasteride (n=6).

Conc.µg/mL	Intra-day precision		Inter-day precision	
	Absorbance measured	RSD (%)	Absorbance measured	RSD (%)
4	0.03533	1.4605154	0.032167	1.1595
8	0.04716	0.8651	0.04483	0.8310
12	0.08083	0.504	0.070667	1.33200

Table 3: Short term stability determined by the proposed method

Sr. No	Stress condition	Degradation %	Remark
1	Photolytic	47.34	Unstable
2	Thermal	50.60	Unstable
3	0.01N HCl	40.90	Unstable
4	0.01N NaOH	49.90	Unstable
5	H_2O_2	52.95	Unstable
6.	Neutral	37.39	Unstable

CONCLUSION

The results and the statistical parameters demonstrate that the proposed UV spectrophotometric method is simple, rapid, specific, accurate and precise. Therefore, this method can be used for the determination of finasteride either in bulk or in the dosage formulations without interference with commonly used excipients and related substances.

REFERENCES

1. Sharma BK, Instrumental Methods of Chemical Analysis, Introduction to Analytical Chemistry, Goel publishing house, Meerat, 19th edition, 2000; (1)4, 200-203.
2. Nash RA, Wachter AH, Pharmaceutical Process Validation, An International third edition, Volume 129.
3. Sharma Y. R., [2002], Elementary organic spectroscopy principal and chemical analysis, S. Chand publication Page No 9.
4. Chatawal G.R., Anand S. K., Instrumental method of chemical analysis, Himalaya publication, fifth edition, Page No 2.149, 2.566, 2.624.
5. Patel D, Patel P, Desai S, Meshram D; Development and Validation of an RP-HPLC-UV Method for the Analysis of Drugs Used for Benign Prostatic Hyperplasia in Pharmaceutical Preparations; Journal of Pharmaceutical and Applied Chemistry An International Journal;2017; 3(2); 99-104.
6. Ahmed SM, Elbashir **AA**; Development and Validation of Spectrophotometric Method for Determination of Finasteride in Pharmaceutical Formulation Using 1,2-Naphthoquine-4-sulfonate (NQS); Journal of Analytical & Bioanalytical Techniques; 2015;1(1);1-6
7. Gaikwad SS, Khan H. N., Zamiruddin M, Bharkad VB; AUC spectroscopic method for of finasteride in pharmaceutical formulation;JPSBR;2017;7(5);310-3013.
8. Thimmaraju M, Rao V, Gurrla S; RP-HPLC Method for the determination of finasteride in bulk and pharmaceutical formulations; IJPIJ; 2011; 1(6);31-38.
9. Sharma BK, Instrumental Methods of Chemical Analysis, Introduction to Analytical Chemistry, Goel publishing house, Meerat, 19th edition, 2000; (1)4, 200-203.
10. Anonymous. Shimadzu Instruction manual pharamaspec UV-1700 series operation guide, Shimadzu Corporation, Kyoto, Japan, 2001, 6.2.