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Second Derivative Spectrophotometric Method For Determination Of Minoxidil And Finasteride In Bulk And Pharmaceutical Formulation

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Guide

Abstract: ***Simple and reliable second derivative spectrophotometric method was developed and validated for simultaneous estimation of Minoxidil and Finasteride in bulk and Pharmaceutical formulation. The quantitative determination of second derivative were carried out using second derivative values measured at 228 nm 236 nm for Minoxidil and Finasteride respectively. The solution of standard and sample were prepared in DMSO: Methanol (1:9 v/v) and Potassium Phosphate buffer (PH7.2) respectively. The calibration graphs constructed at their wavelengths of determination were linear in concentration range of 15-65 µg/ml and 0.5-2.5 µg/ml for Minoxidil and Finasteride respectively. The developed second derivative spectrophotometric method validated according to ICH guideline.***

Keywords: Minoxidil, Finasteride, Dimethyl Sulfoxide (DMSO), Methanol, Potassium Phosphate buffer (PH 7.2).

I. INTRODUCTION

Minoxidil (MINO) chemically is 2,4-diamino-6-piperidinopyrimiddine-3-oxide (Figure 1) is act by relaxing arteriolar smooth muscle with little effect on venous capacitance. It increased rennin release and proximal tubular Na^+ reabsorbing and water retention. Minoxidil also increase hair growth by acting on alteration of androgenic effect on genetically programmed hair follicles and direct stimulation of resting hair follicles.

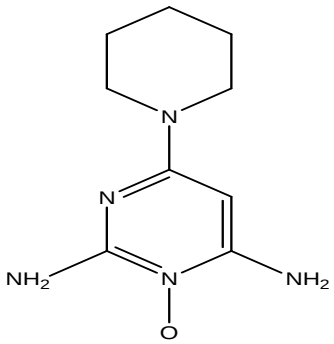

Figure 1: Chemical structure of Minoxidil

Finasteride (FNS) chemically is 17β-(N-tert-butyl carbamoyl)-4-aza-5α-androst-1-en-3-one (Figure.1). It is competitive inhibitor of enzyme 5α-reductase which converts testosterone responsible for androgen action in tissues including prostate gland and hair follicles. Finasteride is antiandrogenic drug and also found effectively in male baldness. It is effective orally, metabolized in liver and excreted in urine faeces.

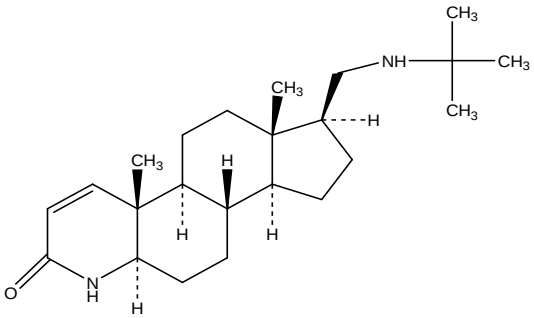

Figure 2: Chemical structure of Finasteride

Litrature survey revealed UV, HPLC and UPLC analytical methods for Minoxidil and Finasteride estimation.

The validation of proposed method is carried out by ICH guideline.

II. MATERIALS AND METHOD

Chemical: Dimethyl Sulfoxide (DMSO), Methanol, 0.1NSodium Hydroxide, Water.

Drugs: Minoxidil, Finasteride.

Instruments-ShimadzuUV-visible spectrophotometer (model UV-1800)

SELECTION OF DERIVATIVE METHOD

The first derivative spectra did not showed zero crossing points in DMSO: Methanol (1:9 v/v) solution and second derivative spectra showed zero crossing in points DMSO: Methanol (1:9 v/v) and showed good resolution characteristic hence second derivative method was selected.

SELECTION OF WAVELENGTHS (ZERO CROSSING POINTS)

The zero crossing points of Minoxidil were 220,228 and 230 nm and for Finasteride were 236,226 and 232 nm. Out of these wavelengths 228 nm for Minoxidil and 236 for Finasteride were selected as the zero crossing points for method based on their linearity data. At 236 nm Minoxidil showed zero absorbance but Finasteride had considerable absorbance. Similarly at 228 nm Finasteride showed zero absorbance but Minoxidil had considerable amount of absorbance.

PREPARATION OF STANDARD STOCK SOLUTION

Standard Minoxidil and Finasteride stock solution was prepared by dissolving 10 mg of drug in 10 ml volumetric flask separately to get concentration 1000µg/ml in DMSO: Methanol(1:9v/v).

PREPARATION OF SAMPLE SOLUTION

From standard stock solution 1 ml pipette out form Minoxidil and Finasteride separately in 10 ml volumetric flask and volume made with Potassium Phosphate buffer (PH 7.2) to get concentration 100 µg/ml solutions separately. From this Minoxidil, aliquots of 1.5,2.5,3.5,4.5,5.5 and 6.5ml and 0.5,1,1.5,2 and 2.5 for Finasteride were transferred to the 10 ml of volumetric flask separately and volume was made up to mark with Potassium Phosphate buffer (PH7.2) to get concentration for Minoxidil 15,25,35,45,55 and 65µg/ml and 0.5,1,1.5,2 and 2.5 µg/ml for Finasteride.

III. VALIDATION PARAMETERS

LINEARITY

Under experimental conditions described, the graph obtained for second derivative spectra showed in (Figure. 3). The absorbance of solution was measured at 228 nm at 0.007 for MINO and 236 nm at 0.017 for FNS. The calibration curve showed linear relationship was plotted in range of 15-65µg/ml for MINO and 0.5-2.5 µg/ml for FNS are shown in (Figure.3and4)

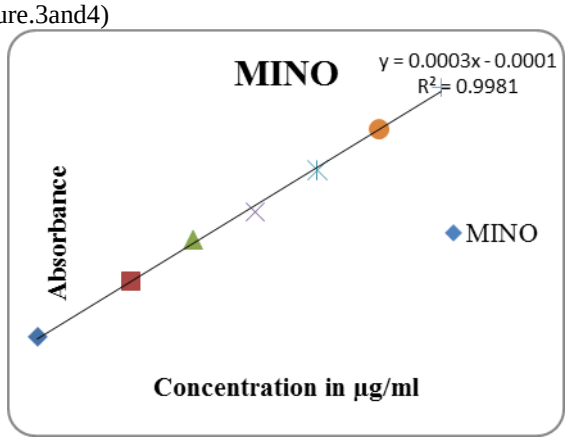

Figure3: Calibration curve of MINO

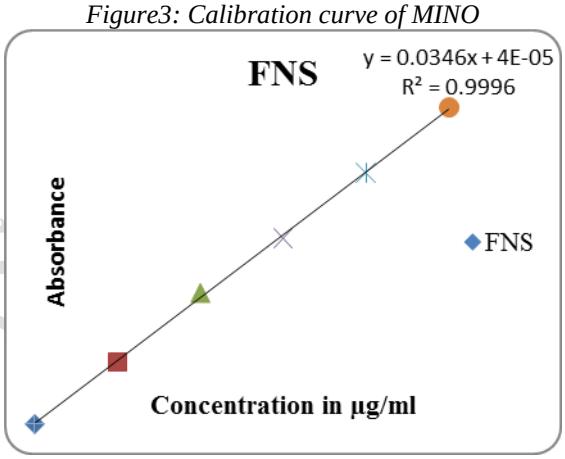

Figure 4: Calibration curve of FNS

Parameters	MINO	FNS
Concentration	15-65µg/ml	0.5-2.5µg/ml
Slope	0.0003	0.003
Intercept	0.0001	0.000
Correlation Coff.(r^2)	0.998	0.998

Table 1: Linearity

Drug	Amount added µg/ml	Amount recovered µg/ml	% Recovery
Minoxidil	24.9	24.5	99.75
	25	24.7	99.85
	26	25.23	99.39
Finasteride	0.4	0.399	99.35
	0.5	0.48	99.60
	0.6	0.52	99.26

Table 2: Recovery studies

Parameter	Inter-day Precision		Intra-day Precision	
	SD	%RSD	SD	%RSD
Minoxidil	0.5501	0.5548	0.5501	0.5548
Finasteride	0.1501	0.1506	0.1501	0.1506

Table 3: Precision studies

Drug Name	Amount labeled (%)	Amount estimated(µg/ml)	% label claim
Minoxidil	5%	4.97	99.39
Finasteride	0.1%	0.99	99.25

Table 4: Analysis of formulation

Parameters	MINO	FNS
Wave length	228	236
Concentration	15-65µg/ml	0.5-2.5µg/ml
Regression equation	Y=0.003x+0.0001	Y=0.033x+0.000
Slope(m)	0.003	0.033
Intercept(c)	0.001	0.000
Correlation coefficient (r2)	0.998	0.988
LOD (g/ml)	1.2	0.7
LOQ(µg/ml)	0.8	1.5
Accuracy (Recovery %RSD)	<2	<2
Precision %RSD		
Interday (n=6)	0.5501±0.5548	0.1501±0.1506
Intraday(n=6)	0.5501±0.5548	0.1501±0.1506

Table 5: Summary of validation

RECOVERY STUDIES

The recovery studies were carried out at three different levels i.e. 80%. 100% and 120%. The percentage recovery values were shown in Table No. 2

PRECISION

To determine precision of method MINO and FNS solution at concentration 25 and 0.5 µg/ml respectively was analyse six times for second derivative spectrophotometric method. The solution for standard deviation were prepared fresh every day. The precision values were shown in Table No. 3

LOD AND LOQ

The limit of detection (LOD) and limit of quantification (LOQ) of developed method were determined by injecting progressively low concentration of the standard solution. The LOQ of Minoxidil and Finasteride was found to be 0.8µg/ml and 1.5 µg/ml respectively. LOD was found to be 1.2 µg/ml and 0.7 µg/ml respectively.

ASSAY PROCEDURE

It was employed for analysis of Minoxidil and Finasteride in topical formulation i.e. Morr F 5%containing 5% Minoxidil and 0.1% Finasteride. In this method the higher percentage of recovery and non interference of the formulation excipients in analysis method for both drugs in there combined dosage form. The %RSD value Indicated suitability of this method for routine analysis of Minoxidil and Finasteride in there combined dosage form. Table No.4

IV. CONCLUSION

A convenient and rapid UV method has been developed for simultaneous estimation of Minoxidil and Finasteride in topical dosage form. The developed method can be easily applied to pharmaceutical dosage form.

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REFERENCES

[1] Indian Pharmacopoeia VOL.II, Published by the Controller of Publication Govt. Of India commission Ghazibad; 2010:1697.
[2] KD Tripathi, Essentials of Medical Pharmacology, 6th edn., Jaypee;2010:548.
[3] Indian Pharmacopoeia VOL.II, Published by the Controller of Publication Govt. Of India commission Ghazibad; 2010:1349.
[4] KD Tripathi, Essentials of Medical Pharmacology, 6th edn.,Jaypee;2010:294.
[5] Vijayamma G., Salomi P., Venkatesh P., Hepcy K. R., Gowramma A., Mounika P., Analytical Method Development and Validation of Minoxidil in Pharmaceutical Dosage Forms by UV Spectrophotometry, International Journal of Innovative Pharmaceutical Research,2015,6(1):464-467.
[6] Zahid A Z., Mirza S., Ismail M. Imran W.S.,UV-Spectrophotometric determination of minoxidil and its application to the assay in pharmaceutical dosage forms, DerPharma Chemica,2012;4(1):568-573.
[7] Rudrapal M., Surya K. S., Sridhar N. and Raghavendra M., development and Validation of RP-HPLC Method for Simultaneous Estimation of Minoxidil and Aminexil in Topical Formulation, Asian Journal of Chemistry; Vol. 28, No. 1 (2016), 157-160.
[8] Bansal M., Rathore P., Goel B., Quantitative Determination of Finasteride from Tablet Formulation by UV-Spectrophotometry, J. Pharm. BioSci.,2013;1:48-50.
[9] Nasare M., Satish J., Manikanta A. K., Prasad V.N., Huidrom M. S., Diwan P. V., Second derivative spectrophotometric method for simultaneous determination of tamsulosin and finasteride in pharmaceutical formulations, Asian J. Pharm. Ana. 2012; 2(3):73-76.
[10] Reddy Y. K. , Reddy G. V. S., Jaya Veera K. N., Hotha K. K., A Stability Indicating UPLC Method for Finasteride and Its Related Impurities, 2012;3: 737-745.
[11] Manne S., Kakarla R., Raavi P., Buchi N. N., Rapid analysis of finasteride in bulk and formulations by rp-hplc-pda method, 2012;57 (4):1469-1471.

[12] Kamepalli S., Sankar D. G., Konda A., Souri O.B., Simultaneous estimation of finasteride and tamsulosin hydrochloride by reverse phase HPLC in bulk and pharmaceutical dosage form, 2012; 3(8):1905-1908.
[13] Sindhura M., Raghavi K., Prashanthi R. , Nalluri B.N., Simultaneous Estimation of Finasteride and Tamsulosin Hydrochloride in Combined Dosage Forms by RP-HPLC-PDA Method, 2012;02(6): 203-209.
[14] Demir H. D., CUCU A. K., Serap S., Determination of finasteride by hplc and its analytical method validation, 2004;4:664-667.
[15] Kategaonkar A.H., Patel D. M., Choudhari V. P., Simultaneous determination of Finasteride and Tamsulosin in pharmaceutical preparations by ratio derivative spectroscopy, Journal of Pharmacy Research, 2009;2(6):1065-1067.
[16] Srinivas G., Kishore K. K., Reddy Y. R., K.,Gangaram V., A Validated stability indicating LC method of assay and related substances for Finasteride, Journal of Chemical and Pharmaceutical Research, 2011, 3(6):987-996.
[17] Patel P.D., Surti N.I., Upadhyay U.M., Development and Validation of Stability Indicating Method for Minoxidil and Finasteride in its Pharmaceutical Dosage Form, 2015;4(2):221-238.
[18] Sudhakar M., Sridhar S. Pravallika C., Analytical Method Development and Validation for Simultaneous Estimation of Finasteride and Minoxidil in Pharmaceutical Dosage Form by RP HPLC Method, (2015), 5(2): 954-959.
[19] Patel D. B. and Patel N. J., Validated RP-HPLC and TLC methods for simultaneous estimation of tamsulosin hydrochloride and finasteride in combined dosage forms, Acta Pharm., 2010(6): 197–205.
[20] Sujatha K., A new validated rp-hplc method for the estimation of finasteride in its tablet dosage forms, International Journal of Advances in Pharmaceutical, 4(11):2529 – 2533.
[21] Vijaya L. N., Koteswara G.S., Roja B. R., Manasal K., Bhavani V. P., Development and Validation of UV Spectrophotometric Method for the Estimation of Finasteride in Tablets, International Journal of Pharma Sciences, 2013;3(1): 123-125.
[22] Thimmaraju M. K., Venkat R., Srikanth G., Reddy G. J.,UV spectrophotometric method for simultaneous determination of finasteride and tamsulosin in combined dosage form,International Journal of Pharmacy and Biological Sciences,2011;1(3):303-310.
[23] ICH-Guidelines Q2 (R1), Validation of Analytical Procedures: Text and Methodology. 2005.