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Analytical Method Development and Validation of Minoxidil in Pharmaceutical Dosage Forms by UV Spectrophotometry

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ABSTRACT

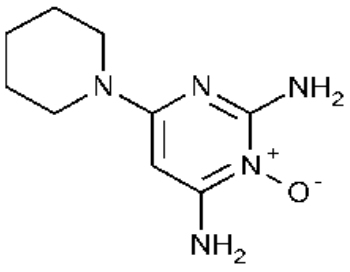
A selective and sensitive spectrophotometric method for determination of Minoxidil has been validated by simple, precise and economical UV spectroscopic method. It has been developed for the estimation of Minoxidil in Pharmaceutical solution. Minoxidil shows the absorbance maxima at 280.4nm. The method is carried out by using n- butanol as a solvent. Then the further dilutions were prepared by using water as the main solvent. It shows the linearity in the concentration range of 0.1-2.5µg/mL. The method allows rapid analysis of pharmaceutical formulation with accuracy and precise. The method was validated by statistically and reproducibility studies which was found satisfactory. The proposed method was successfully applied for the determination of Minoxidil in topical dosage form.

Keywords: Minoxidil, n- butanol, UV-spectroscopy, validation, water.

INTRODUCTION

Chemically Minoxidil is a 2, 4- diamino-6 - piperidinopyrimidine-3-oxide. Minoxidil is a drug that is used for treating male-pattern baldness. Minoxidil is a vasodilator. Hypothetically, by widening blood vessels and opening potassium channels, it allows more oxygen, blood, and nutrients to the follicle. This may cause follicles in the telogen phase to shed, which are then replaced by thicker hairs in a new anagen phase. The mechanism of action leading to growth of hair is exactly unknown (Minoxidil- Wikipedia). The described methods in literature has revealed that several methods such as UV-Spectrophotometric determination of Minoxidil and its application to the assay in pharmaceutical dosage forms (Zahid A et al), Development and validation of stability indicating HPLC method for the estimation of Minoxidil and related substances in topical formulation (Hemant K et al), Bromometric analysis of Lamotrigine, Minoxidil and Cefixime , vibrational spectroscopic analysis of Minoxidil (2,4-Diamino-6-Piperidino pyrimidine-3-Oxide) and related analogs have been reported for the analysis of Minoxidil (Aboul-Kheir et al). In the present investigation an attempt was made to develop a simple and precise ultra-violet (UV) spectrophotometric method with greater precision, accuracy sensitivity for the analysis of Minoxidil in pharmaceutical dosage forms.

Figure.1 Structure of Minoxidil



MATERIALS AND METHODS

Single pan electronic balance- Sartorius GE412, UV visible spectrophotometer, UV visible double beam spectrophotometer, Systronics 2203(smart), Matched quartz cells corresponding to 1 cm path length. Pure samples of Minoxidil were obtained from kaushik therapeutics' pvt Ltd., gurrcumbakkam, Chennai, India. The solvent n-butanol was purchased from Padma pharmaceuticals Pvt Ltd., Kadapa, India.

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Reagents: n-butanol, water, Reference standard Minoxidil

Procedure

Preparation of standard stock solution

The standard stock solution of drug was prepared by dissolving 10mg of the drug in 10 ml standard flask using n-butanol as a solvent to give a concentration of 1000 µg/ml. This stock solution on further dilutions were made to get 2 µg/ml by Using water as solvent

Beer's law concentration range

The stock solution was suitably diluted with water to get concentration range from 1 to 1000 µg/ml .The solutions were scanned in the UV region between 200-400nm and their absorbance were measured at 280.4 Using the absorbance values against concentrations calibration curve was plotted .From the graph it was found that Minoxidil obey's Beer's law range between 0.1-2.5µg/ml.

Preparation of sample

1ml of sample (2% Minoxidil topical solution contains 20mg of minoxidil in 1 ml solution) weighed by syringe and transferred in to a 100-ml volumetric flask, and then make up with n-butanol (butanol)**.** From that 1 ml of solution was transferred by pipette in to a 100 ml volumetric flask then completed to volume with water yields a sample solution having a concentration assumed to be 2µg/ml of Minoxidil

Analysis of Formulation

An accurately measured volume of liquid equivalent to about 100mg of minoxidil was transferred to a 100ml standard flask. The contents of the flask were mixed with n-Butanol solution and dissolve the active ingredients and then make up to the volume with the water as a solvent. The solution was filtered and the filtrate was further diluted with water as the solvent to give a final drug concentration of 0.1-2.5µg/ml. Absorbance values of sample solution were recorded at 280.4nm

The proposed method is validated for the following parameters.

a. Repeatability studies
b. Reproducibility studies

Determination of Repeatability

Repeatability can be defined as the precision of the procedure when repeated by same analyst under the same operating conditions (same reagents equipments, settings and laboratory) over a short interval of time. It is normally expected that at least six replicates be carried out and a table showing each individual result provided from which the mean, standard deviation and co-efficient of variation should be calculated for set of n value. The RSD values are important for showing degree of variation expected when the analytical procedure is repeated several time in a standard situation. (RSD below 2% for built drugs, RSD below 2% for assays in finished product).

The ICH documents recommend that repeatability should be assessed using a minimum of nine determinations covering the specified range for the procedure (i.e. three concentrations and three replicates of ach concentration or using a minimum of six determinations at 100% of the test concentration).

Determination of Reproducibility

Reproducibility means the precision of the procedure when it is carried out under different conditions-usually in different laboratories-on separate, putatively identical samples taken from the same homogenous batch of material. Comparison of results obtained by different analysts, by the use of different equipments, or by carrying out the analysis at different times can also provide valuable information.

RESULTS AND DISCUSSION

The UV spectra of minoxidil were presented. The absorption maxima was observed at 280.4nm. Obeyance to beers law was confirmed by the linearity of the calibration curve of minoxidil. Minoxidil showed linearity in the concentration range of 0.1-2.5 µg/ml. Linearity data was given in table -1 and the curve was shown in fig.2.

The quantitative estimation was carried out in tablet formulations by taking concentrations of 0.1-2.5µg/ml. The brands of formulation shows the percentage purity values range from 99.2 to 101.6 the percentage deviation values were found to be between +0.2 to 0.8 and the values shown in table 3.

Repeatability

The repeatability of the method was confirmed by the assay procedures with 3 different concentrations of 3 replicates each. The results obtained in repeatability test expresses the precision of the given method. And the values were shown in table.4.

Reproducibility

The validation of the proposed method was further confirmed by reproducibility studies. The reproducibility values for day 1 varied from 100.0 to 101.25 %. The reproducibility values for day 2 varied from 99.4 to 101.0 % T data is given in table 5. This serves as a good index of accuracy.

Fig.2 Absorbance Vs Concentration

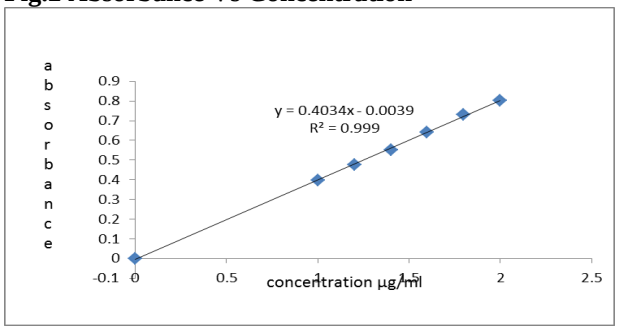

Table.1 Linearity Data

S. No	Concentration(µg/ml)	Absorbance
1	0.1	0.019
2	0.2	0.89
3	0.4	0.156
4	0.6	0.239
5	0.8	0.322
6	1.0	0.402
7	1.2	0.486
8	1.4	0.674
9	1.6	0.727
10	1.82	0.858
11	2.0	0.927
12	2.2	0.974
13	2.4	1.295

Table.2 Quantitative Estimation of Minoxidil Formulation

S. no	Concentration (µg/ml)	Label claim (mg)	Amount present (mg/ml)	Percentage Found (%w/v)	Percentage Deviation (%w/w)
1	1.6	20	19.96	99.5	±0.5
2	1.8	20	19.99	99.6	± 0.4
3	2.0	20	20.0	100.0	± 0.00
4	2.2	20	20.01	100.5	± 0.5
5	2.4	20	20.01	100.8	± 0.8

Table.3 Statistical Data of Formulation

Drug Name	%Assay mean	Standard Deviation(S.D)	Relative Standard Deviation(R.S.D)
Minoxidil	100.08	0.054	0.0053

Table.4 Repeatability Studies of Formulation

Concentration (µg/ml)	Assay (%)	Assay Mean	Standard Deviation	Mean of Standard Deviation(S.D)
2	100.16			
2	100.2	100.2	0.0092	0.0061
2	100.0			

Table.5 Reproducibility studies of Formulation

Concentration	Amount present Day 1	% Found Day 1	S.D Day1	Amount present Day 2	%Found Day 2	S.D Day 2
1.6	1.61	100.6	0.0014	1.6	100.0	0.0012
1.6	1.62	101.25	0.0007	1.61	100.6	
1.6	1.61	100.6	0.0007	1.60	100.0	0.0021
1.8	1.82	101.1	0.0014	1.8	100.0	0.0014
1.8	1.81	100.5	0.0021	1.79	99.4	0.0021
1.8	1.80	100.0	0.0021	1.81	100.5	0.0014
2	2.01	100.5	0.002	2.0	100.0	0.003
2	2.02	101.0	0.0021	2.01	100.5	0.0021
2	2.01	100.5	0.0041	2.02	101.5	0 0032

CONCLUSION

The proposed method of analysis is novel, simple, cost-effective, safe, accurate and reproducible. This method can be routinely employed in the analysis of Minoxidil in topical solution formulations precluding using n-butanol as a solvent.

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