



Spectrophotometric determination and estimation of minoxidil in tablet dosage form by UV

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ABSTRACT

A simple, precise, accurate and economical UV spectrophotometric method has been developed and validated for the estimation of Minoxidil in tablet dosage form. Minoxidil shows maximum absorbance at 279.4nm. The method was carried out by using 0.1N HCl as a solvent. The drug shows linearity from the concentration range of 1-6µg/ml and correlation coefficient was found to be 0.9992. The proposed method was statistically validated for precision, accuracy, ruggedness, robustness, limit of detection, limit of quantitation as per the ICH guidelines. Hence this method can be successfully applied for routine analysis of Minoxidil in bulk and tablet dosage form.

Keywords: Minoxidil; 0.1N HCl; UV- spectroscopy; Validation.

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INTRODUCTION

Chemically minoxidil is 2, 4-pyrimidinediamine, 6-(1-piperidinyl)-3-oxide. Its molecular formula is C₉H₁₅N₅O and molecular weight is 209.25. It is freely soluble in methanol, 0.1N HCl, 0.1N H₂SO₄, propylene glycol but slightly soluble in water. Minoxidil is an orally effective direct acting peripheral vasodilator that reduces elevated systolic and diastolic blood pressure by decreasing peripheral vascular resistance. The active metabolite of minoxidil activates the ATP-modulated potassium channel causing K⁺ efflux, hyperpolarization and smooth muscle relaxation. Initially minoxidil described as an antihypertensive agent but it also shows some new applications, especially in the treatment of androgenic alopecia.

According to the literature survey it was found that few analytical methods were reported for the estimation of minoxidil by using UV spectroscopy^[2]. The other methods were also proposed for its determination includes HPLC, RP-HPLC, and electrochemical method^[4,5]. The present investigation is to develop a simple, precise and cost-effective UV method for method development and validation of minoxidil in a pharmaceutical dosage form.

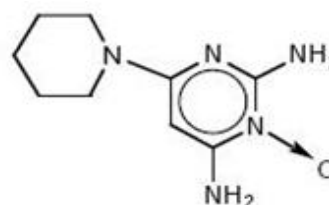


Figure 1: Structure of Minoxidil

MATERIALS AND METHODS

Single pan electronics balance-sartorius GE412, UV visible double beam spectrophotometer (systronics-2203 smart), matches quartz cells corresponding to 1cm path length. Minoxidil was taken as a gifted sample from sun pharmaceuticals pvt. Ltd. and its pharmaceutical dosage forms were purchased from market.

Reagents: 0.1N HCl, Minoxidil, Reference standard

Preparation of standard stock solution: The standard stock solution was prepared by dissolving 25mg of drug in 25ml of 0.1N HCl to produce a 1000µg/ml. From the above solution, 1ml of stock solution is withdrawn and diluted with 100ml of 0.1N HCl to produce 10µg/ml concentration.

Determination of λ_{\max} : 10 $\mu\text{g/ml}$ concentration of minoxidil was prepared and scanned under UV from 250-400nm^[9]. The λ_{\max} of the drug was found at 279.4nm with a sharp peak.

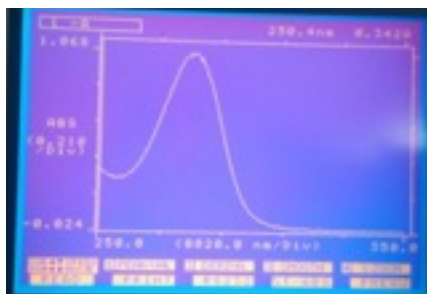


Figure 2: Determination of λ_{\max} minoxidil

Beer's law concentration range: The stock solution was suitably diluted with 0.1N HCl to get a concentration range from 1-6 $\mu\text{g/ml}$ and their absorbance was measured at 279.4nm. Using the absorbance values against concentration, calibration curve was plotted. From the graph it was found that, Minoxidil obeys beer's law between 1-6 $\mu\text{g/ml}$.

Table 1: Linearity data for Minoxidil

S.no	Concentration ($\mu\text{g/ml}$)	Absorbance
1	1	0.196
2	2	0.364
3	3	0.520
4	4	0.685
5	5	0.831
6	6	0.985
Slope		= 0.1571
Correlation coefficient		= 0.9992

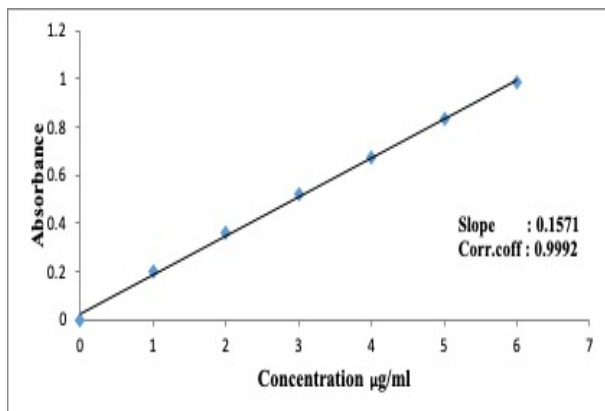


Figure 3: Linearity graph for Minoxidil

Preparation of sample solution: 20 tablets were finely powdered. An accurately weighed quantity of tablet powder equivalent to about 25mg of Minoxidil was transferred to 25ml standard flask. This was diluted with 25ml of 0.1N HCl to give 1000 $\mu\text{g/ml}$. From this 0.2, 0.3, 0.4, 0.5, 0.6ml of sample solution was taken and diluted with 100ml of 0.1N HCl to give 2, 3, 4, 5, 6 $\mu\text{g/ml}$ concentration. The solution was filtered and absorbance value of sample solutions was recorded at 279.4nm.

RESULTS AND DISCUSSION

Validation is defined as the establishing evidence which provide high degree of assurance that a specific process will consistently produce a product meeting its determined specification quality characteristics. The following parameters used for validation studies are

Precision: The closeness of agreements between a series of measurements, multiple sampling of homogeneous samples under prescribed condition, precision is of two types:

- Repeatability
- Reproducibility

Repeatability (System Precision): 4 $\mu\text{g/ml}$ concentration solution of Minoxidil was prepared whose absorbance measured six times for which relative standard deviation was calculated.

Reproducibility (Method Precision): Six individual preparations of Minoxidil were prepared with a concentration of 4 $\mu\text{g/ml}$, whose absorbance was measured at 279.4nm.

Solution stability: 4 $\mu\text{g/ml}$ concentration solution of Minoxidil was prepared and the solution whose absorbance was measured for every half an hour for 90 minutes and the solution were found to be stable up to 90 minutes.

Limit of detection: The detection limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value^[13].

$$\text{LOD} = \frac{3.3 \times S.D}{\text{Slope}}$$

Table 2: Results for LOD

S.no.	Parameter	Minoxidil
1	Slope	0.1601
2	Standard deviation	0.0209
3	LOD	0.34202

Limit of quantitation: The limit of quantitation of an individual analytical procedure is the lowest amount of analyte in a sample which can be quantitatively determined with precision and accuracy.

$$\text{LOQ} = \frac{10 \times S.D}{\text{Slope}}$$

Table 3: Results for LOQ

S.no.	Parameter	Minoxidil
1	Slope	0.1601
2	Standard deviation	0.0209
3	LOQ	1.14040

Accuracy: Accuracy of method is the closeness of the measured value to the true value for the sample. Accuracy is usually determined by recovery studies.

Table 4: Results for formulation Lonitab 5mg

S.no.	Conc (µg/ml)	Label claim (mg)	Amount present	% of label claim	% Dev
1	2	5	0.00503	98.6	0.014
2	3	5	0.00505	99.0	0.010
3	4	5	0.00503	98.6	0.013
4	5	5	0.00500	98.0	0.019
5	6	5	0.005064	99.2	0.007

Table 5: Results for formulation dilminox 5mg

S.no.	Conc (µg/ml)	Label claim (mg)	Amount present	% of label claim	% Dev
1	2	5	0.00505	98.9	0.011
2	3	5	0.00509	99.8	0.002
3	4	5	0.00508	99.6	0.005
4	5	5	0.00509	99.5	0.005
5	6	5	0.0050	99.4	0.006

Table 6: Results for System Precision

S.no.	Conc (µg/ml)	System Precision	Method Precision
		Absorbance	Absorbance
1	4	0.658	0.566
2	4	0.605	0.565
3	4	0.634	0.585
4	4	0.512	0.570
5	4	0.576	0.607
6	4	0.666	0.599
Average		0.6085	0.5824
Standard deviation		0.0579	0.0179
% RSD		0.0952	0.0308

Table 7: Results of %Recovery Studies

S.no.	Concentration (µg/ml)	Mg found	Mg added	% recovery
1	80	0.004952	0.005	99.03
2	100	0.004993	0.005	99.85
3	120	0.004903	0.005	98.06

Table 8: Ruggedness Parameters

Analyte	Concentration (µg/ml)	Absorbance	% RSD
Analyte - 1	4	0.5869	0.0305
Analyte - 2	4	0.5824	0.0308

Table 9: Ruggedness Parameters

Analyte	Concentration (µg/ml)	Absorbance	% RSD
Analyte - 1	4	0.5869	0.0305
Analyte - 2	4	0.5824	0.0308

Recovery studies are performed by spiking pure powdered drug into the sample solution. The spiked samples are prepared at a concentration range of 80%, 100%, 120%.

Procedure: The sample solution was prepared to get a concentration range of 3µg/ml, 4µg/ml, 5µg/ml, into which 5mg of pure powdered drug was added to get 80%, 100%, 120% concentration range. The percentage recovery was calculated for these concentrations from absorbance obtained.

The percentage recovery was calculated by using the following formula;

$$\text{Percentage recovery} = \frac{\text{amount obtained}}{\text{amount added}} \times 100$$

The percentage recovery for the spiked preparation should be within 98-102 %.

Ruggedness: The extent to which is turned precision should be established depends on circumstances which the procedure is intended to be used. Intermediate precision expresses with in laboratory variation i.e., different days, different analyst and different equipments.

Procedure: The procedure followed for this is the same followed in the method precision was repeated on two different days by two different analysts. The result for the intermediate precision recorded in the table.

Acceptance Criteria: The relative standard deviation for the preparation should not be more than 2%.

Robustness: Robustness of the method is its ability to remain unaffected by small ranges in parameters such as changes in wavelength, changes in pH, changes in the temperature etc.

Robustness examines the effect of operational parameters on the analytical method.

Procedure: 4µg/ml concentration of Minoxidil was prepared. Absorbance was measured at two different wavelengths closer to the λ_{max} of the drug.

Table 10: Robustness Results

S.no.	Concentration (µg/ml)	Wavelength (nm)	Absorbance
1	4	277.4	0.583
2	4	279.4	0.588
3	4	281.4	0.599

No change in absorbance value

CONCLUSION

On the basis of our experimental results, we conclude that the UV spectrophotometric method developed for the determination of minoxidil was found to be precise, accurate and cost effective. Hence this method can be used for routine analysis of Minoxidil in bulk and pharmaceutical dosage forms.

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