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Development and validation of Ilaprazole in bulk and pharmaceutical dosage form by UV spectroscopic method

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Abstract

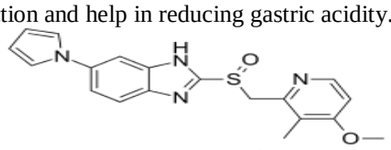
A simple UV spectroscopic method was developed and validated for the estimation of Ilaprazole in bulk and pharmaceutical dosage form using Acetonitrile: ethanol (50:50) as solvent. The quantification was achieved at 307nm. Beers law was obeyed in the concentration range of 2-12 µg/ml. The results of analysis have been validated statistically and recovery studies carried out in the range 80-120% to confirm the accuracy of the proposed method. The relative standard deviation was found to be less than 2.0%. The present result shows that the proposed method can be successfully implemented for estimation of Ilaprazole in bulk and its marketed formulations.

Keywords: UV Spectroscopy, Ilaprazole, Acetonitrile, ethanol

1. Introduction

Ilaprazole is a proton pump inhibitor [1-4] (PPI) used in the treatment of dyspepsia, Peptic ulcer disease (PUD), gastro esophageal reflux disease (GORD/GERD) and duodenal ulcers.

Chemically[2] it is 2-[(RS)-[(4-methoxy-3-methylpyridin-2yl) methyl] sulfinyl]-5-(1H-pyrrol-1-yl)-1H-benzimidazole. It suppresses gastric acid secretion by specific inhibition of the H+/K+-ATPase in the gastric parietal cell by acting specifically on the proton pump; Thus Ilaprazole blocks the acid production and help in reducing gastric acidity.



Literature review reveals that some of the UV [5-8], HPLC [9], UPLC [10] methods have been reported for the estimation of Ilaprazole. Very few assay indicating methods are reported; hence the present work has made an attempt for quantification of Ilaprazole individually in its bulk and tablet formulation by UV spectroscopy as per ICH guidelines.

2. Materials and Methods

2.1 Instrumentation

Absorption spectral measurements were carried out with a UV – Visible spectrophotometer (T60 PG instruments & T190 shimadzu) with spectral bandwidth of 1 nm and wavelength accuracy of 0.3 nm (with automatic wavelength correction with a pair of 5 cm matched quartz cells.

2.2 Chemicals

Acetonitrile & Ethanol used for dilution was of analytical grade. Ilaprazole is a marketed sample manufactured by SIDCO, kartholi Ltd. All other ingredients used were of analytical grade

2.3 Preparation of Standard stock solution

10mg of standard Ilaprazole drug was weighed accurately and taken in a clean and dry volumetric flask. The drug was dissolved in some amount of Solvent (Acetonitrile: Ethanol) and made upto the mark with the same solvent. This gives 1000µg/ml solution. From this solution 1ml was taken and transferred to a 10ml volumetric flask and the volume was made up to the mark by using solvent resulting to a concentration of 100µg/ml. Next again a 1ml of solution was taken from above dilution in 10ml volumetric flask and made up to 10ml this give concentration of 10µg/ml.

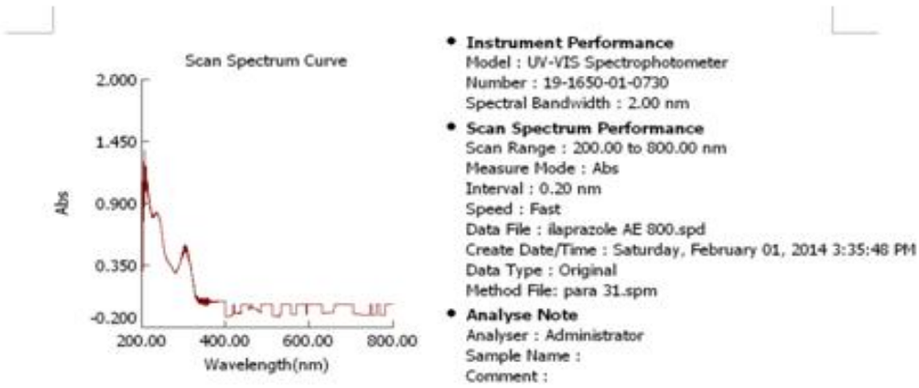
2.4 Preparation of Test solution

Twenty tablets of formulation were weighed and finely powdered. The powder equivalent to 10 mg of Ilaprazole was accurately weighed. It was then transferred to volumetric flask of 10 ml capacity containing 5 ml of solvent and sonicated for 5 min. The flask was shaken and the solution was filtered through Whatmann filter paper (No. 41) into 10 ml volumetric flask. Volume was made up to the mark with solvent to give a solution of 1000 µg/ml (Stock solution A). From this stock solution A, 1 ml was taken and placed in 10 ml volumetric flask. The volume was made up to the mark using solvent to give a solution of 100 µg/ml (Stock solution B). From the stock solution B, 1ml was taken and diluted to 10 ml to give 10 µg/ml.

2.5 Selection of λ max

The solution of 10 µg/ml was prepared from standard stock solution and the prepared diluted solution was scanned by using UV-Visible spectrophotometer from the range between 200nm-400nm on spectrum mode, using proper solvent (Acetonitrile: Ethanol).The UV spectrum had shown highest peak at 307nm. Hence 307nm was selected as λ max for analysis.

Fig no: 1 UV spectrum of Ilaprazole



2.6 Validation parameters

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Linearity

A linear relationship should be evaluated across the range of the analytical procedure. It was demonstrated directly on the drug substance (by dilution of a standard stock solution) and using the proposed procedure.

Accuracy

Accuracy was established across the specified range of the analytical procedure. Accuracy is the closeness of the test results obtained by the method to the true value. Recovery studies were carried out by addition of standard drug to the sample at 3 different concentration levels taking into consideration percentage purity of added bulk drug samples.

Precision

Precision was determined as repeatability and interday and intraday precision, in accordance with ICH guidelines. Variation of results within the day (intraday), variation of results between the days (interday) was analyzed. Intraday precision was determined by analyzing Ilaprazole for six times in the same day at 307nm.

Inter day precision was determined by analyzing the drug for three different days at 307nm.

3. Results and Discussions

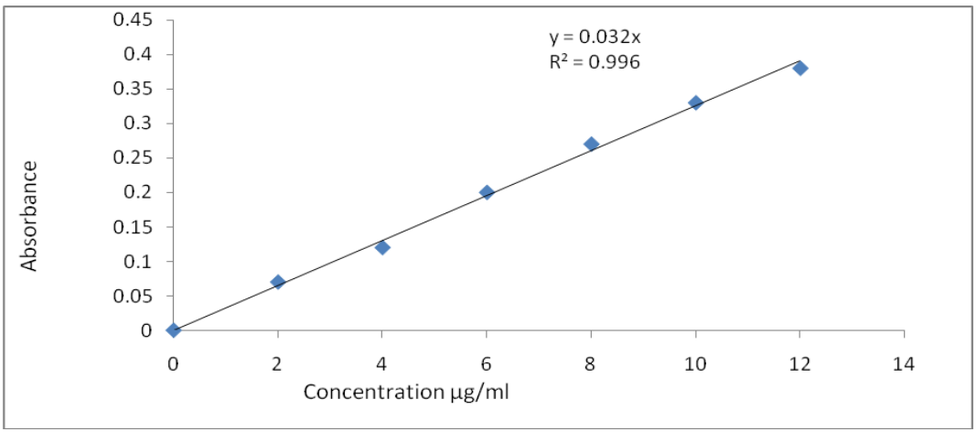
Linearity

In this method Ilaprazole was estimated by using ultraviolet spectroscopy. The method obeys Beer lamberts law in the concentration range of 2-12µg/ml and its wavelength of detection was 307nm. Correlation coefficients of Ilaprazole were found to be 0.999 with a straight line equation Y=0.033X+0.033. The absorbance values are given in Table 1 and figure 2 indicates that the method is linear.

Table 1: Linearity Results

S. No	Concentration	Absorbance	
		Standard	Sample
1	2	0.098	0.07
2	4	0.154	0.12
3	6	0.219	0.2
4	8	0.271	0.27
5	10	0.332	0.33
6.	12	0.379	0.38

Fig No: 2 Linearity curve of Ilaprazole



Accuracy

The accuracy of the proposed methods was assessed by recovery studies at three different levels i.e. 80%, 100%, 120%. The values of recovery (%), RSD (%) listed in Table 2 indicate the method is accurate.

Table 2: Results of Accuracy Studies by UV spectroscopy

S. No	Concentration	Absorbance		% Assay
		Standard	Sample	
1	80%	0.318	0.317	99.6
2	80%	0.319	0.320	100
3	80%	0.320	0.319	99.6
1	100%	0.352	0.350	99.4
2	100%	0.351	0.354	100.8
3	100%	0.354	0.352	99.4
1	120%	0.411	0.412	100.2
2	120%	0.410	0.409	99.5
3	120%	0.411	0.410	99.7
Arithmetic Mean				99.7
Standard deviation				0.1
% Relative Standard Deviation				0.01

Precision

The intra & inter day variation of the method was carried out and the standard deviation and % RSD (% RSD < 2%) within a day and day to day variations for Ilaprazole listed in Table 3 and Table 4 ,indicates the proposed method is precise.

Table 3: Results for Intraday Precision

S. No	Absorbance		% Assay
	Standard	Sample	
0th hour	0.352	0.335	95.1
1st hour	0.341	0.340	100.2
2nd hour	0.342	0.343	99.4
Arithmetic Mean			98.2
Standard Deviation			38.4
% Relative Standard Deviation			0.039

Table 4: Results for Interday Precision

S. No	Absorbance		% Assay
	Standard	Sample	
Day 1	0.352	0.350	99.4
Day 2	0.369	0.365	98.9
Arithmetic Mean			99.1
Standard Deviation			0.94
% Relative Standard Deviation			0.009

Robustness

The spectrophotometry method shows that developed method was robust. the robustness was performed by different analysts (analyst-I and analyst-II), different wavelengths (305nm & 309nm) and different instruments (T60 pg instruments & T190 shimadzu) in the same laboratory by estimating the multiple samples of single concentration.

Differential Wavelength:

Table 5: Differential Wavelength values for standard and sample

S. No	Absorbance		%Assay
	Standard	Sample	
305nm	0.276	0.275	100.3
	0.274	0.273	99.6
	0.278	0.278	100.7
309nm	0.413	0.413	100.4
	0.412	0.412	99.7
	0.415	0.415	100.4
Arithmetic Mean			100.1
Standard Deviation			0.1
%Relative Standard Deviation			0.01

Differential Analyst:

Table 6: Differential Analyst values for standard and sample

S. No	Absorbance		% Assay
Analyst 1	Standard	Sample	
	0.351	0.353	100.5
	0.351	0.354	100.8
	0.352	0.357	101.4
Analyst 2	0.355	0.354	99.7
	0.354	0.357	100.8
	0.358	0.356	99.4
Arithmetic Mean			100.4
Standard Deviation			0.8
%Relative Standard Deviation			0.007

Differential Instrument:

Table 7: Differential Instrument values for standard and sample

S. No	Absorbance		% Assay
Analyst 1	Standard	Sample	
	0.351	0.350	99.7
	0.352	0.353	100.2
	0.351	0.351	100
Analyst 2	0.352	0.350	99.4
	0.352	0.352	100
	0.351	0.353	100.5
Arithmetic Mean			99.7
Standard Deviation			0.14
%Relative Standard Deviation			0.001

4. Conclusion

The proposed method was found to be simple, selective and sensitive. The validation parameters were also found to be within the limits. The method showed acceptable linearity and accuracy and is highly sensitive therefore it could be used easily for the routine analysis of pure drugs and their formulations for Ilaprazole.

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