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Analytical Method Development and Validation for Esomeprazole by Using UV Spectrophotometric Method

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ABSTRACT

A simple, sensitive UV-spectrophotometric technique was developed and validated to measure esomeprazole in bulk and various dose forms. Esomeprazole has the highest absorbance at 299nm by using methanol as a solvent. Many analytical performance criteria, such as linearity, precision, accuracy, and robustness, were also determined using ICH recommendations. LOD and LOQ were determined from the regression equation. The linearity range was found to be 2-10µg/ml, the %RSD for repeatability was found to be less than 2, and the correlation coefficient (r2) was 0.999. The % mean recovery was found to be for the different concentrations for 98-99.23% for esomeprazole. %Assay was found to be between 98.1-98.3.The findings of the analysis were statistically confirmed and supported by recovery studies.

Keywords: Esomeprazole, methanol, UV-Visible spectrophotometry, Validation

INTRODUCTION

Esomeprazole is used to treat the disease known as excessive stomach acid production. Gastro-oesophageal reflux disease (GORD), a condition that causes persistent acid reflux, indigestion, heartburn, and acid reflux, is usually treated with it. Brands include Nexium and Vimovo. Molecular Weight: [345.41g.mol-1] and Molecular Formula: [C17H19N3O3S]. The bioavailability ranges from 50 to 90%, while the elimination half-life is between 1.1 and 1.5 hours.

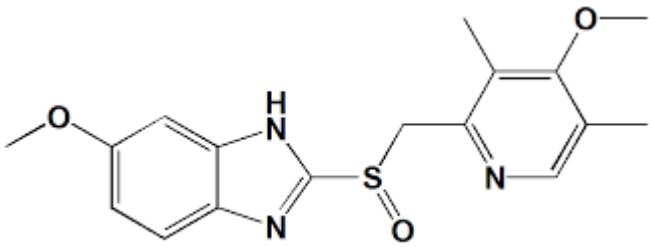

Figure 1: Esomeprazole Structure

Iupac name: (S)-(-)-5-Methoxy-2-[(4-methoxy-3,5-dimethylpyridin-2-yl)methylsulfinyl]-3H-benzoimidazole.

The experimental data underwent statistical validation in order to confirm the suggested method's precision, accuracy, and reproducibility.

Mechanism: Esomeprazole works by specifically inhibiting H+/K+-ATPase in the gastric parietal cell to reduce stomach acid production.

MATERIAL AND METHODS

Equipment's utilized

The SHIMADZU twin beam UV/Visible spectrophotometer model UV1800s was utilized. It has a spectral bandwidth of 1 nm and a wavelength precision of 0.3 nm (with automated wavelength adjustment with a pair of 1 cm matched quartz cells). The extreme Sonicator (Fast clean) model 2k811056 and the SHIMADZU Electronic Balancing model AXE 200 were also used in the analysis.

Materials

Esomeprazole was purchased in pure form from lab in Hyderabad, India. Tablet formulation contain 50mg of esomeprazole, according to the label claim. All chemicals and reagents used were of analytical grade.

Standard stock solution preparation

Esomeprazole was correctly weighed at 10 mg per 10 ml volumetric flask to create the stock solution. Methanol was added to bring it up to the required concentration of 1000 ppm.

Working standard solution preparation

To transfer 1ml to a 10ml volumetric flask, use a pipet. With the addition of methanol, the volume was brought up to standard at a concentration of 100 ppm. There were several dilutions performed in the 2–10 µg/ml range, where the beer's rule was followed. The following dilutions are used: 2, 4, 6, 8, and 10 µg/ml.

Selection of wavelength for analysis of Esomeprazole

Appropriate volume 1ml of standard stock solution of esomeprazole was transferred into a 10 ml volumetric flask, diluted to a mark with distilled water to give the concentration of 10 µg/ml. The resulting solution was scanned in the UV range (200–400 nm). In the spectrum, esomeprazole showed an absorbance maximum at 299nm**.**

Method Validation

The method was validated in terms of linearity, accuracy, precision, robustness, ruggedness, LOD, and LOQ.

Linearity study

Appropriate aliquots of esomeprazole working standard solutions were placed in separate 10 ml volumetric flasks and diluted up to the mark with distilled water to generate final concentrations of 2, 4, 6, 8, and 10 g/ml. Calibration curves were created by graphing absorbance versus concentrations.

Precision

It was demonstrated by intraday studies. In intra day the solution that has the same concentration was analyzed six times in day and % RSD was calculated.

Accuracy

This test was performed at 3 different concentration levels of 50%, 100%, and 150%, with 3 replicate at each percentage. 2ml of 2ppm of standard esomeprazole solution is spiked with 2ml of 4ppm sample solution to get 50%.2ml of 4ppm of standard esomeprazole solution is spiked with 2ml of 4ppm of sample solution to get 100%.2ml of 6ppm of standard esomeprazole solution is spiked with 2ml of 4ppm of sample solution to get 150%. For all these concentrations absorbance was checked at 299nm and percentage accuracy was calculated by using the below formula.

$$Percentage\ Accuracy(\%) = Observed\ Concentration/Nominal\ concentration\ \times 100$$

Robustness

The Robustness of the proposed method is determined for a 10 µg/ml concentration of Esomeprazole by analysis of aliquots from a homogenous slot by ±1 λ_{max} wavelength using the same operational and environmental conditions.

Ruggedness

The Ruggedness of the proposed method is determined for a 10 µg/ml concentration of Esomeprazole by analysis of aliquots from a homogenous slot by two analysts using the same operational and environmental conditions.

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. LOD was calculated by using the regression equation from linearity. LOD was calculated by using following method.

Y= ax + b

Where

a =Slope,

b = Intercept

The number of concentrations used for linearity=N

SD of intercept =SE of intercept × √N

LOD =3.3 × (SD of intercept/slope)

Limit of Quantitation

The Quantitation limit of an analytical procedure is the lowest amount of analyte in a sample, which can be quantitatively determined with suitable precision and accuracy.

LOD was calculated by using the following method.

LOQ =10 × (SD of intercept/slope)

Determination of Esomeprazole in bulk

The 10µg/ml solution was scanned on a spectrophotometer in the UV range 200–400 nm. The concentrations of the drug were calculated from linear regression equations.

Application of the proposed method for pharmaceutical formulation

Ten tablets of esomeprazole were weighed and crushed and mixed in mortar pestle into fine powder. Each tablet having content of esomeprazole equivalent to 40mg.0.22gm of powder was taken and dropped into 10ml volumetric flask. The volume was made up with methanol. Using filter paper, the drug solution filtered. It was scanned on a spectrophotometer in the UV range 200–400 nm. The spectrum was recorded at 299 nm. The concentrations of the drug were calculated from the linear regression equation.

RESULTS AND DISCUSSION

Method validation

The proposed method was validated as per ICH guidelines.

Linearity

The linear regression data for the calibration curves showed good linear relationship over the concentration range 5–30 µg/ml for Esomeprazole which is shown in Figure 2. Linear regression equation was found to be Y = 0.0448X + 0.0285 (r2 = 0.999). The result is expressed in Table 1.

Table 1: Linearity of esomeprazole

Concentration(µg/ml)	Absorbance(nm)
2	0.063
4	0.153
6	0.234
8	0.326
10	0.424

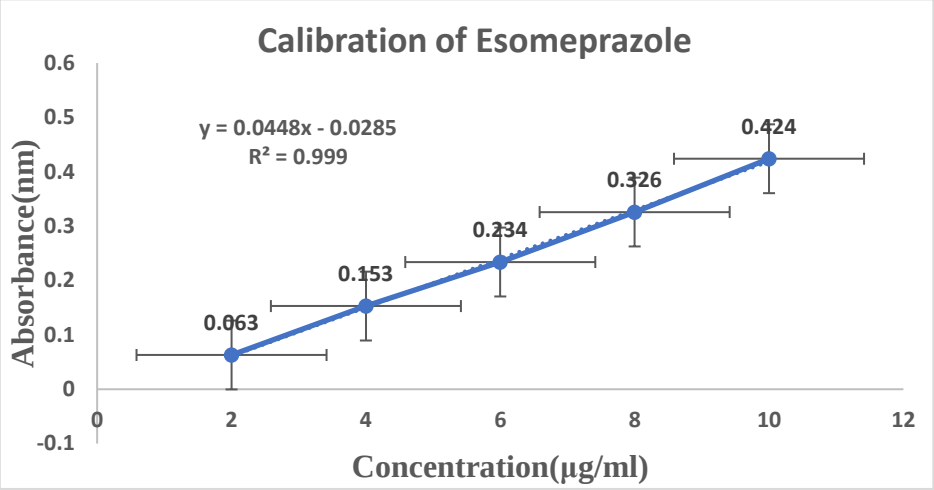

Figure 1: Calibration of Esomeprazole

Precision

The precision of the developed method was expressed in terms of % relative standard deviation (% RSD). These results show reproducibility of the assay. The % RSD values found to be less than 2 . Results of precision were shown in Table 2.

Table 2: Intra-day precision of Esomeprazole

Concentration(µg/ml)	Absorbance(nm)
10	0.422
10	0.418
10	0.422
10	0.422
10	0.423
10	0.424
Average	0.421833333
SD	0.00186339
%RSD	0.004417361

Accuracy

The results of recovery studies are reported in Table 3 which showed that the % amount found was between 98% and 99.23% with % RSD > 2.

Table 3: Results of Accuracy

%Level	**Absorbance(nm)**	**%Recovery**	**Mean %Recovery**
50%	0.236 0.235 0.236	99.1 99.5 99.1	99.23
100%	0.331 0.332 0.333	98.4 98.1 97.8	98.1
150%	0.433 0.430 0.433	97.7 98.6 97.7	98

Robustness

The peak area was measured for same concentration solutions, six times at ± λ_{max}. The results are in the acceptable range. The results are given in Table 4. The result showed that the % RSD was less than 2%.

Table 4: Results of Robustness

Concentration(µg/ml)	Absorbance(298nm)	Absorbance(299nm)	Absorbance(300nm)
10	0.321	0.422	0.522
10	0.333	0.418	0.518
10	0.321	0.422	0.522
10	0.322	0.422	0.522
10	0.322	0.423	0.523
10	0.321	0.424	0.524
Average	0.323333333	0.421833333	0.521833333
SD	0.004346135	0.00186339	0.00186339
%RSD	1.344165444	0.441736068	0.357085273

Ruggedness

The peak area was measured for same concentration solutions, six times by 2 different analyst. The results are in the acceptable range for both the drugs. The results are given in Table 5. The result showed that the % RSD was less than 2%.

Table 5: Results of Ruggedness

Concentration(µg/ml)	Analyst-1 (299nm)	Analyst-2 (299nm)
10	0.422	0.421
10	0.418	0.433
10	0.422	0.421
10	0.422	0.422
10	0.423	0.422
10	0.424	0.421
Average	0.421833333	0.423333333
SD	0.00186339	0.004346135
%RSD	0.441736068	1.026646048

LOD and LOQ

LOD and LOQ were calculated from regression equation. Result of LOD and LOQ were shown in Table 6.

Table 6: Results of LOD and LOQ

SE of intercept	0.005533233
SD of intercept	0.012372685
LOD	0.911380812
LOQ	2.761760038

Determination of Esomeprazole in bulk

The concentrations of the drug were calculated from linear regression equations. The % amount found was between 99.12% and 100.43% [Table 7].

Application of the proposed method for pharmaceutical formulation

The spectrum was recorded at 299 nm. The concentrations of the drug were calculated from the linear regression equation. The % amount found was between 98.1% and 98.3% [Table 7].

Table 7: Analysis of bulk and tablet formulation

Concentration(µg/ml)	Analysis of Esomeprazole in bulk	Analysis of Esomeprazole in Formulation	%Amount found
10	0.429 0.429 0.429	0.422 0.421 0.422	98.3 98.1 98.3

CONCLUSION

This UV-Visible spectrophotometric technique is quite simple, accurate, precise, reproducible, and sensitive. The UV method has been developed for quantification of Esomeprazole in tablet formulation. Validation was done according to ICH Q2 R1 guidelines. The validation procedure confirms that this is an appropriate method for their quantification in the formulation. It is also used in routine quality control of the formulations containing this entire compound.

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