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Spectrophotometric determination of esomeprazole in bulk and tablet formulation

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ABSTRACT

Three simple accurate and sensitive UV spectrophotometric methods have been developed for the quantitative estimation of esomeprazole in bulk and its pharmaceutical formulations. In method I, 0.1 N sodium hydroxide was used as solvent. Esomeprazole shows maximum absorbance at 305 nm and obeys Beer-Lambert's law in the concentration range of 5-30µg/mL. In method II, 10% DMF was used as solvent. Esomeprazole shows maximum absorbance at 302 nm and obeys Beer-Lambert's law in the concentration range of 5-30µg/mL. In method III, 20% methanol was used as solvent. Esomeprazole shows maximum absorbance at 300 nm and obeys linearity in the concentration range of 4-24µg/mL. The methods were validated statistically for accuracy, precision and sensitivity.

KEYWORDS

Esomeprazole;
Spectrophotometric method;
Tablet.

INTRODUCTION

Chemically Esomeprazole (ES) is bis(5-methoxy-2-[(S)-[(4-methoxy-3,5-dimethyl-2-pyridinyl)methyl] sulfinyl-1H-benzimidazole-1-yl[1]. It is proton pump inhibitor and is used in the management of acid related disorders[2,3]. The literature survey reveals few HPLC [4-6] and spectrophotometric[7] methods for estimation of from its formulations. ES is available in tablet dosage form. The present paper describes three simple, reproducible and sensitive UV spectrophotometric methods for the determination of ES in tablets.

EXPERIMENTAL

Instrument

A double-beam Shimadzu UV-Vis-spectrophotometer, model UV-1650 with matched quartz cells of 10 mm path length, connected to computer loaded with UV-Probe.software (Version 2.21was used for measurement of absorbance.

Materials and reagents

ES obtained as a gift sample from Aurbindo Pvt. Ltd. Hydrabad, India, was used as working standards. Sodium hydroxide, DMF and Methanol were used as solvents to prepare the standard stock solutions. All the chemicals used were of Analytical reagent grade (S.D.Fine. Chem. Ltd., Mumbai) used without further purification.

Selection of solvents and wavelengths

The solubility of ES was checked in different solvents, and 0.1 N sodium hydroxide, 10% DMF and 20% Methanol were selected as the solvent for dissolving the drug. The ES show absorption maxima at 305, 302 and 300 nm in 0.1 N sodium hydroxide, 10% DMF and 20% methanol, respectively. Typical absorption spectra of ES are shown in figures 1, 2 and 3.

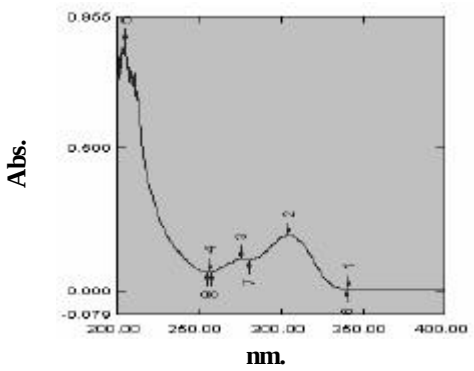

Figure 1: Typical UV spectrum of ES in 0.1N NaOH

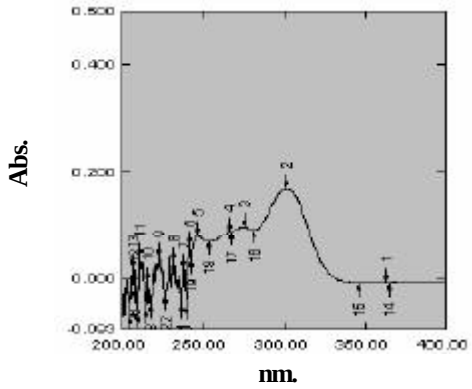

Figure 2: Typical UV spectrum of ES in 10% DMF

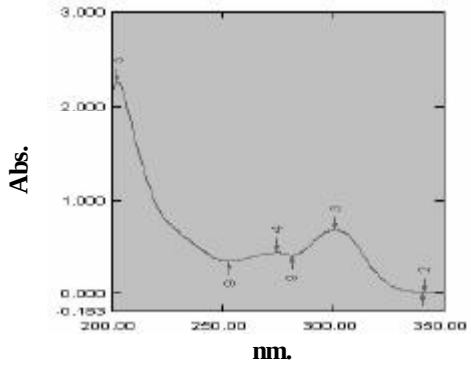

Figure 3: Typical UV spectrum of ES in 20% methanol

Preparation of standard solution

For method I, an accurately weighed 10 mg of ES was transferred to 100mL volumetric flask. It was dissolved in 25mL of 0.1 N sodium hydroxide and the volume was made up to the mark using same solvent to obtain concentration of 100µg/mL. Different aliquots were taken from the stock solution and diluted with the same solvent to prepare a series of concentrations. The solutions were scanned on spectrophotometer in the UV range and their absorbances were measured at 305nm using 0.1 N sodium hydroxide as blank. The calibration curve was found to be linear in the concentration range of 5-30µg/mL. For method II, the similar procedure described in method I was followed using 10% DMF as a solvent. The absorbances were measured at 302nm using 10%DMF as a blank. The calibration curve was found to be linear in the concentration range of 5-30µg/mL. For method III, the similar procedure described in method I was followed using 20% methanol as a solvent. The absorbances were measured at 300nm using 20% methanol as a blank. The calibration curve was found to be linear in the concentration range of 4-24µg/mL. The slope, intercept, correlation coefficient and optical characteristics of three the methods are reported in TABLE 1.

TABLE 1: Optical characteristics and statistical data of the regression equation

Parameters	Method I	Method II	Method III
Solvent	0.1N sodium hydroxide	10 % DMF	20% Methanol
Absorption maximum (nm)	305	302	300
Beer's law limit (µg/ml)	5-30	5-30	4-24
Coefficient of correlation	0.9997	0.9997	0.9997
Y-intercept	0.0027	0.0007	0.0166
Slope	0.0376	0.0387	0.0332

Estimation of ES in bulk drug

Accurately weighed quantity of 20 mg ES was transferred to 100 mL volumetric flask, dissolved in 0.1N NaOH and volume was adjusted to mark to get stock solution of 200µg/mL. From this stock solution aliquots of 1 mL were transferred to six 10 mL volumetric flasks separately and diluted with 0.1N NaOH to the mark to get final concentrations of 20µg/mL. The absorbances of these solutions were measured at 305 nm. Same procedure was followed for estimation of ES using 10% DMF as solvent and absorbances were measured at 302nm. For estimation of drug using 20% methanol, the final solutions of 16µg/mL were prepared and their absorbances were measured at 300nm. The concentration was determined by regression equation and results are shown in TABLE 2.

Assay procedure for tablets

For method I, twenty tablets were weighed and crushed to fine powder. An accurately weighed powdered sample equivalent to 20mg of ES was transferred to 100 mL volumetric flask. The powder was dissolved in 50mL of 0.1N sodium hydroxide by intermittent shak

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TABLE 2: Results of estimation of ES in bulk drug

	Method I	Method II	Method III
Concentration taken (µg/ml)	20	20	16
*Concentration found (µg/ml ± SD)	20.14±0.05	20.08±0.08	16.02±0.17
% RSD	0.27	0.42	1.07

***Mean of six estimations, SD is standard deviation, RSD is relative standard deviation**

TABLE 3: Results of assay of ES in tablets

	Method I	Method II	Method III
Label claim(mg/tab)	20	20	20
*Amount found (mg/tab± SD)	20.22±0.16	20.19±0.20	20.11±0.24
% RSD	0.83	0.99	1.49

***Mean of six estimations**

TABLE 4: Results of recovery studies

Method	Concentration added (µg/ml)	*Concentration recovered (µg/ml) ± S.D.	% Recovery
I	16	16.10 ± 0.08	100.64
	20	20.05 ± 0.24	100.28
	24	24.15 ± 0.23	100.63
II	16	16.06 ± 0.91	100.35
	20	20.11 ± 0.17	100.54
	24	23.80 ± 0.18	99.17
III	12.8	12.74 ± 0.15	99.51
	16	16.08 ± 0.08	100.49
	19.20	19.24 ± 0.17	100.23

***Mean of three estimations**

TABLE 5: Results of intra-day and inter-day precision

Method	Conc. [µg/mL]	*Intra-day conc. found [µg/mL]		*Inter-day conc. found [µg/mL]	
		Mean ± S.D.	% RSD	Mean ± S.D.	% RSD
I	10	10.67 ± 0.16	1.54	10.29 ± 0.17	1.66
	20	20.67 ± 0.30	1.47	20.20 ± 0.19	0.98
	30	30.10 ± 0.25	0.83	30.08 ± 0.24	0.81
II	10	10.04± 0.12	1.22	10.08 ± 0.08	0.87
	20	19.98 ± 0.14	0.70	20.18 ± 0.26	1.31
	30	30.10 ± 0.26	0.87	30.08 ± 0.23	0.77
III	8	8.19 ± 0.09	1.12	8.30 ± 0.08	1.01
	16	16.45 ± 0.13	0.84	16.46 ± 0.09	0.55
	24	24.79 ± 0.17	0.68	24.61± 0.27	1.13

***Mean of three estimations**

ing and volume was made up to the mark with same solvent to get stock solution of 200µg/mL. The solution was filtered through Whatmann filter paper no. 41. From stock solution 1mL was taken and diluted to 10mL with 0.1 N sodium hydroxide. The absorbance of this solution was recorded at 305 nm and concentration of sample was determined. For method II, the similar procedure described in method I was followed using 10% DMF as solvent and the absorbance was recorded at 302 nm. Similarly for method III, the stock solution of 200µg/mL was prepared of using 20% methanol. From this the solution of 16µg/mL was prepared and absorbance was recorded at 300nm. The whole procedure was repeated for six times. The results are reported in TABLE 3.

RESULTS AND DISCUSSION

The methods developed were validated as per ICH guidelines[8,9]. Recovery studies were carried out for three developed methods by addition of known quantity of pure drug solution to preanalysed tablet sample solution at three different concentration levels. The results of recovery studies are reported in TABLE 4. The intra-day and inter day precision were determined by analyzing samples of ES at concentrations of 10, 20 and 30µg/mL in 0.1N NaOH, 10% DMF respectively and a in 20% methanol at concentrations 8, 16 and 24µg/mL. Determinations were performed with three replicates on same day as well as on three separated days over a period of week. The results are shown in TABLE 5. The results of the validation tests were found to be satisfactory and therefore this method can be applied to analyze drug formulations.

REFERENCES

[1] S. Budawari; 'The Merck Index', 13th Ed., Merck and Co. Inc., USA, (2001).

[2] S.C.Sweetman; 'Martindale-The Complete Drug Reference', 33rd Ed. Pharmaceutical Press, London, **(2002).**

[3] T.N.Riley, J.Deruiter, J.H.Block, J.M.Beale; 'Wilson and Gisvold's Textbook of Organic Medicinal and Pharmaceutical Chemistry', Lippincott Williams and Wilkins, 724 **(2004)**.

[4] Ia Hultman, Helene Stenhoff, Mathias Liljeblad; J.Chrom.B, 848 **(2007)**.

[5] R.Setty, G.Subramanian, A.Ranjith Kumar, S.Pandey, N.Udupa; Indian Drugs, **42(3)**, **(2005)**.

[6] Arm Ad An Onal, Aysel OZtunc; J.Food and Drug Anal., **14**, **(2006)**.

[7] P.M.Raj, R.V.Rao, P.B.Mukherjee, V.S.Sarvanan, N.Gopal, T.M.Kalyankar, T.Shivakumar; Asian J.Chem., **19**, **(2007)**.

[8] ICH; Harmonized Tripartite Guideline, Validation of Analytical Procedure Methodology, Q2B, **(1996)**.

[9] 'The United States Pharmacopoeia 27', The National Formulary 22, Asian Ed., United States Pharmacopoeial Convention, Inc., **(2004)**.