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Development and Validation of Visible Spectrophotometric methods for the Estimation of Ethamsylate in Pharmaceutical Dosage Forms

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ABSTRACT

Two simple and economical visible spectrophotometric methods (A and B) have been developed for the quantitative determination of Ethamsylate in bulk drug and pharmaceutical dosage forms have been developed. Method A is based on the reaction of Ethamsylate with Folin-Ciocalteu (phenol's) reagent under alkaline conditions forming a blue colored chromogen exhibits absorption maxima at 619 nm. Beer's law was obeyed in the concentration range of 10-50µg/ml. In Method B Ethamsylate undergoes oxidation followed by complex formation reaction with 1,10 phenanthroline in presence of ferric chloride to form red coloured chromogen exhibiting absorption maximum at 510 and obeyed Beer's law in the concentration range of 1-5 µg/ml. These methods were extended to pharmaceutical formulations and there was no interference from any common excipients. The results of analysis have been validated statistically and by recovery methods.

Key words: Spectrophotometry, FC reagent, 1, 10 phenanthroline, Chromogen.

INTRODUCTION

Ethamsylate is chemically Diethylammonium 2, 5-dihydroxybenzenesulphonate[1-3]. It has been used in the prevention and treatment of capillary bleeding in menorrhagia after abortion, epistaxis, malena, hematuria and after tooth extraction but efficacy is unsubstantiated [4]. The literature survey reveals that few analytical methods for this drug are reported, which include chromatographic [5], and spectrophotometric methods[6-7]. The present investigation has been undertaken to develop two simple and accurate spectrophotometric methods using Folin-Ciocalteu reagent and 1,10 phenanthroline. Which are essential for routine quality control analysis of pharmaceutical products containing Ethamsylate as active constituent.

MATERIALS AND METHODS

Instrument
All spectral measurements were made on Shimadzu 1800 UV-Visible spectrophotometer with 1 cm matched quartz cells were used.

Materials
Pure drug of Ethamsylate was obtained from Juggat pharmaceutical pvt Ltd, Bangalore and commercial formulations were procured from local market. All the chemicals used were of analytical grade.

Reagents
Folin-Ciocalteu reagent (1N) was diluted to 2N with distilled water
Alcoholic solution of 1,10 phenanthroline (0.5% w/v)
Aqueous solution of Sodium Hydroxide (1N)
Aqueous solution of ferric chloride(0.5% w/v)

Preparation of Standard solution :
Weigh accurately 100 mg of Ethamsylate and transferred in to 100 ml volumetric flask and dissolve in 100 ml of distilled water to obtain a concentration of 1mg /ml. From this suitable dilutions were made to obtain the working standard concentration of 100 µg /ml .

Preparation of sample solution:
Two brands of commercially available tablets were taken, twenty tablets each weighing 250mg were weighed and powered. A tablet powder equivalent to 100 mg was weighed accurately and transferred in to 100 ml volumetric flask containing 50 ml of distilled water, the flask was sonicated for 5 min, the volume was made up to mark with water, and the solution was filtered through whatmann filter paper 41, from the above stock solution, working standard solution of 100mg/ml were prepared by further dilution with water, the above procedure was applied for analysis.

Assay Procedure:
Method A
Aliquots of standard drug solution ranging from 0.2 to 1.0 ml (1ml=1000µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 1.0 ml of Folin-Ciocalteu reagent and 1.0 ml of 1N sodium hydroxide were added, kept for 10 min to develop the colour and the volume was made up to the mark with distilled water. The absorbance of blue colored chromogen was measured at 619 nm (fig 1) against a reagent blank. The amount of drug present in the sample was computed from its calibration curve(fig 2).

Method B
Aliquots of standard drug solution ranging from 0.1 to 0.5 ml (1ml=1000µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 1 ml of 1,10 phenanthroline, 0.5 ml of ferric chloirde were added and kept for 10 min heating at 40^{o}c and the volume was made up to the mark with water. The absorbance of red colored chromogen was measured at 510 nm (fig 3)

against a reagent blank. The amount of drug present in the sample was computed from its calibration curve(fig 4).

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, Molar absorptivity, and relative standard deviation were calculated and the results are summarized in Table 1. Regression characteristics like slope, intercept and correlation coefficient were calculated and are presented in Table 1.

Commercial tablets of Ethamsylate were successfully analyzed by the proposed methods and the results are presented in Table 2. To evaluate validity and reproducibility of the methods recovery experiments were conducted and the results are shown in Table 2. Comparison of the results obtained with the proposed and UV methods for dosage forms (Table 2) confirms the suitability of these methods for Pharmaceutical dosage forms. To evaluate validity and reproducibility of the methods recovery experiments were conducted and the results are summarized in Table 2. The other active ingradients and excipients usally present in pharmaceutical dosage forms did not interfere.

Table-1 Optical characteristics and Precision

Parameters	Method A	Method B
λmax (nm)	619	510
Beer's law limits (µg/ml)	20-100	10-50
Molar absorptivity (L mol^{-1} cm^{-1})	9.058 X 10^3	1.882X10^3
Regression equation (Y = a+bc)		
Slope (b)	0.0344	0.0715
Intercept (a)	0.0010	0.0020
% R S D	0.1941	0.2570
Correlation coefficient (r)	1.0000	1.0007
Limit of Quintitation (LOQ)	0.2900	0.0084
Limit of Detection (LOD)	0.0959	0.0929
Range of errors**		
Confidence limit with 0.05 level	1.0576 X 10^{-3}	2.1148 X 10^{-3}
Confidence limit with 0.01 level	1.5648 X 10^{-3}	1.5601 X 10^{-2}

Y=bC+a were C is the concentration of Ethamsylate in µg/ml and Y is absorbance unit
*** for eight measurements*

Table-2 Evalution of Ethamsylate in Tablet Dosage formulations

	Label Claim (mg)	Amount of drug obtained by proposed methods (mg)		Reference method	% Recovery*		% Recovery reference method
		A	B	UV	A	B	UV
M_1	250	249.97	249.50	249.58	99.49	99.37	99.37
M_2	250	248.98	249.00	249.64	99.46	99.54	99.28

**mean of six determinations, M_1 = Athamstat (Indi pharma Pvt Ltd),*
M_2 = Dicynene (Dr Reddy's Labs).

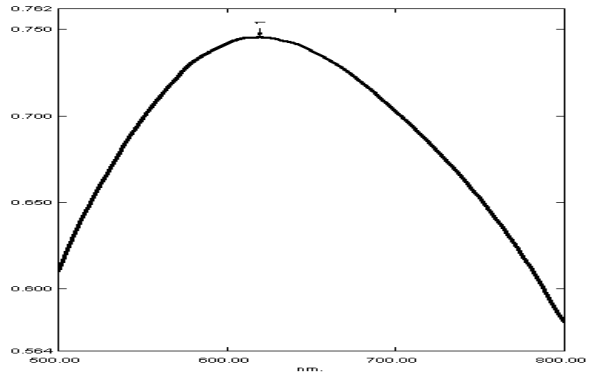

Fig 1 Absorption Spectrum of ETH by FC

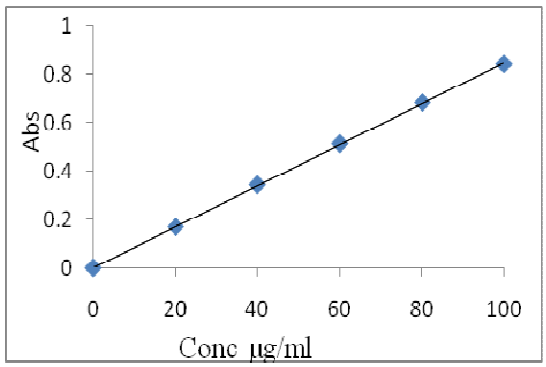

Fig 2 Calibration Curve of ETH by FC

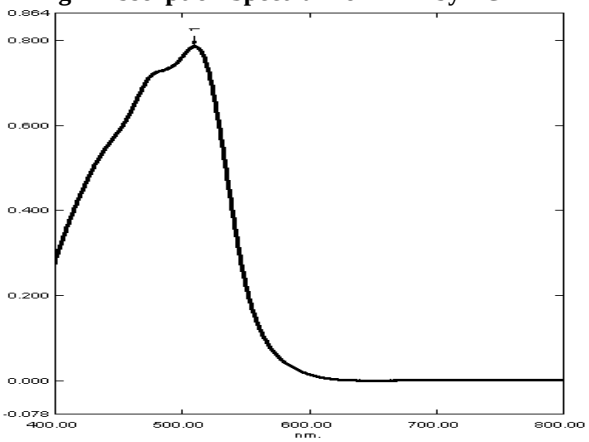

Fig 3 Absorption Spectrum of ETH by 1,10,Phe

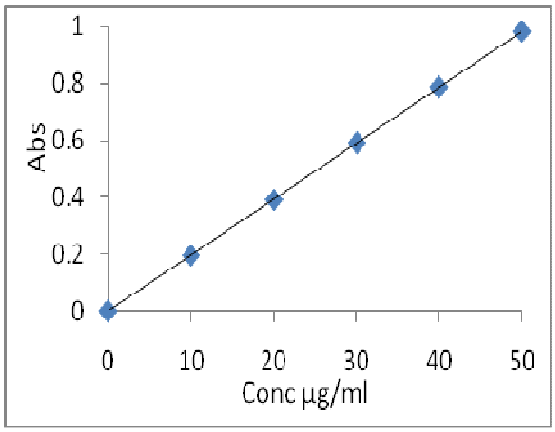

Fig 3 Calibration Curve of ETH by 1,10,Phe

CONCLUSION

The proposed visible spectrophotometric methods for the estimation Ethamsylate are simple, sensitive, economical and can be used for the routine quality control of the drug in bulk as well as in pharmaceutical formulation

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