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Colorimetric Determination of Ethamsylate in Bulk and its Pharmaceutical Formulations.

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Abstract
Two simple and economical spectrophotometric methods (A and B) have been developed for the quantitative determination of Ethamsylate in bulk drug and pharmaceutical form has been developed. Method A is based on oxidative coupling reaction of Ethamsylate with 3-methyl benzothiazolinone hydrazone (MBTH) reagent under alkaline conditions forming a green colored chromogen and exhibits absorption maxima at 580 nm. Beer's law was obeyed in the concentration range of 4-20 µg/ml. In Method B Ethamsylate undergoes oxidation followed by complex formation reaction with 2,2[1] Bipyridine in presence of ferric chloride to form red coloured chromogen exhibiting absorption maximum at 510 nm and obeys 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, MBTH, 2, 2[1] Bipyridine, 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 MBTH reagent and.2,2[1] bipyridine which are essential for routine quality control analysis of pharmaceutical products containing Ethamsylate as active constituent.

Materials and Methods
Apparatus
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.

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Reagents
Alcoholic solution of 2 2[1] bipyridine (0.1 %w/v), Aqueous solution of MBTH (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 0.4 to 2.0 ml (1ml=100µg/ml) were transferred in to a series of 10 ml volumetric flasks. To each flask 0.5 ml of $FeCl_3$(1%w/v), 1 ml of MBTH were added,

kept aside for 10 min to develop the color and the volume was made up to the mark with distilled water. The absorbance of blue colored chromogen was measured at 580 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 2,2 bipyridine, 0.5 ml of ferric chloride 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 520 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 summarized 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 ingredients and excipients usually present in pharmaceutical dosage forms did not interfere.

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 its pharmaceutical formulations.

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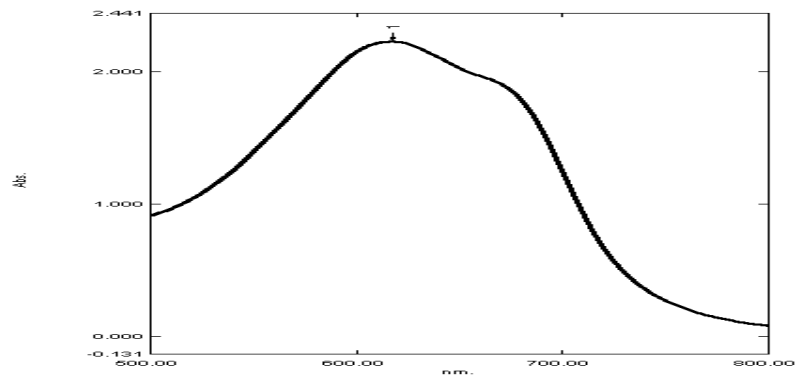

Fig 1: Absorption Spectrum of ETH by MBTH.

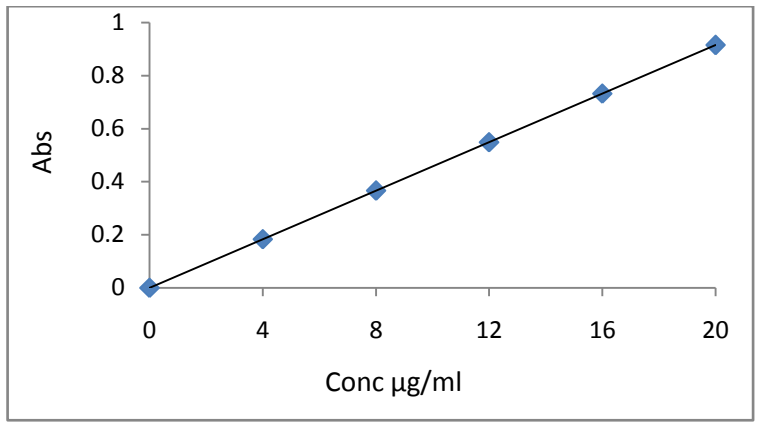

Fig 2: Calibration Curve of ETH by MBTH.

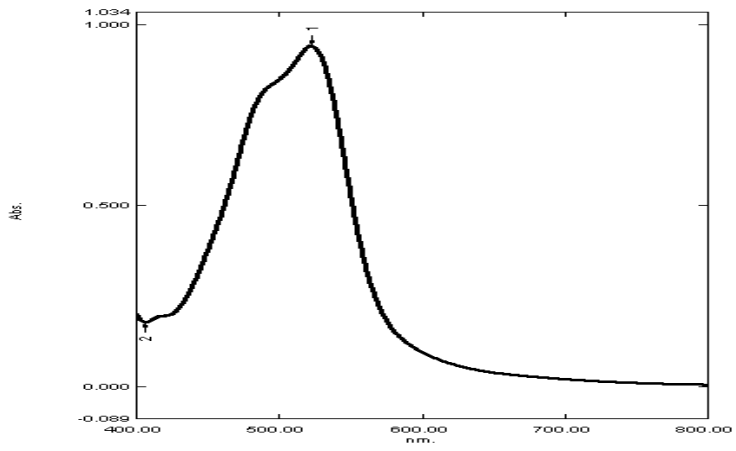

Fig 3: Absorption Spectrum of ETH by 2,2[1]BPD.

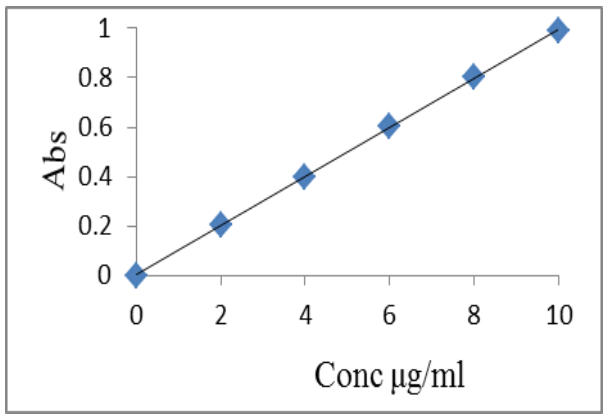

Fig 3: Calibration Curve of ETH by 2,2[1]BPD.

Table 1: Optical characteristics and Precision.

Parameters	**Method A**	**Method B**
λ max (nm)	580	520
Beer's law limits (µg/ml)	0.4-2.0	1-5
Molar absorptivity (L mol^{-1} cm^{-1})	1.3768 $X10^4$	1.704$X10^4$
Regression equation (Y = a+bc)		
Slope (b)	0.0186	0.0645
Intercept (a)	0.0860	0.0003
% R S D	0.0001	0.2590
Correlation co-efficient (r)	1.0030	1.0007
Limit of Quantitation (LOQ)	0.0600	0.01562
Limit of Detection (LOD)	0.0198	0.0510
Range of errors**		
Confidence limit with 0.05 level	4.8760 X 10^{-4}	1.5648 X 10^{-4}
Confidence limit with 0.01 level	7.2363 X 10^{-4}	1.5601 X 10^{-4}

Y=bc+a were C is the concentration of Ethamsylate in µg/ml and Y is absorbance unit,

** for eight measurements.

Table 2: Evaluation of Ethamsylate in Tablet Dosage formulations.

	Label Claim (mg)	**Amount of drug obtained by proposed methods (mg)**		**Reference method**	**% Recovery***		**% Recovery**
		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).

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