SPECTROPHOTOMETRIC ESTIMATION OF ETHAMSYLATE IN BULK DRUG AND ITS PHARMACEUTICAL FORMULATIONS

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ABSTRACT
Ethamsylate is an orally available hemostatic drug. 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 unestablished. Three simple, sensitive, precise and economical UV-spectrophotometric methods have been developed in bulk drugs and its formulations. Ethamsylate is dissolved in distilled water and it has exhibited a absorption maxima at 300.0 nm (Method A) and in the first order derivative spectra a sharp peak is shown at 288.0 nm (Method B). Method C is based on calculation of Area under Curve (AUC) for the analysis of Ethamsylate in the wavelength range of 305.0-295.0 nm. Linearity for the detector response was observed in the concentration range of 10-50μg/ml for Method A and 5-25μg/ml for Method B and C respectively. Results of the analysis were validated for accuracy, precision, Limit of Detection (LOD), Limit of Quantification (LOQ) and were found to be satisfactory. The proposed methods are simple, sensitive, rapid, economical, and suitable for the routine quality control application in pharmaceutical formulations.

Keywords: Ethamsylate, UV spectrophotometry, derivative spectroscopy, Area Under Curve (AUC).

INTRODUCTION
Ethamsylate[1-2] is chemically Diethylammonium 2, 5-dihydroxynbensenesulphonate. Literature survey reveals that very are few analytical methods for the estimation of Ethamsylate by RP HPLC[3] and HPTLC[4] and so far no UV/Visible spectrophotometric methods have been reported for the estimation of Ethamsylate and this led to the development of UV spectrophotometric methods for the estimation Ethamsylate.

MATERIALS AND METHODS
Instrument: A double beam Shimadzu UV-Visible spectrophotometer 1700, 1cm matched Quartz cells were used to measure the absorbance of the resulting solution. Distilled Water was used as a solvent.
Standard Stock solution: Standard stock solution of Ethamsylate was prepared by dissolving 100mg in 100ml of distilled water. Working standard solution of drug was prepared by further dilution with distilled water.

Procedure\(^{[5-6]}\)

Method A – Absorption Maxima Method:

For the selection of analytical wavelength, 30 µg/ml solution of Ethamsylate was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400 nm to 200 nm. From the spectra of drug (Fig. 1), λ\(_{\text{max}}\) of Ethamsylate, 300.0 nm was selected for the analysis. The calibration curve was obtained in the concentration range of 10- 50µg/ml. The amount of drug present was computed from its calibration curve. (Fig-2)

Method B -- First Order Derivative Spectroscopy:

In this method, 15 µg/ml solution of Ethamsylate was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400 nm to 200 nm. A first order derivative spectrum was selected for analysis of drug (Fig 3). First order derivative spectra of the drug (Fig. 3), showed a sharp peak at 288.0 nm, which was selected for its quantitation. The calibration curve of drug was in the concentration range of 5-25 µg/ml. The amount of drug present was computed from its calibration curve. (Fig-4)

Method C -- Area under Curve Method:

For the selection of analytical wavelength, 15 µg/ml solution of Ethamsylate was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400 nm to 200 nm. From the spectra of drug, area under the curve in the range of 305.0-295.0 nm was selected for the analysis. The calibration curve was found to be in concentration range of 5-25 µg/ml at their respective AUC range. The amount of drug present was computed from its calibration curve. (Fig-5).

Assay of Ethamsylate in Pharmaceutical formulations:

Two Brands of commercially available tablets were taken; twenty tablets each weighing 250 mg, were weighed and powered. A tablet powder equivalent to 100 mg was weighed accurately and transferred to 100 ml volumetric flask containing 50 ml of water, the flask was sonicated for 5 min, the volume was made upto the mark with water, and the solution was filtered through whatmann filter paper no 41 from the above stock
solution, working standard solution of 100µg/ml were prepared by further dilution with water, the above procedure is applied for analysis.

RESULTS AND DISCUSSION

The methods described in the present work provide a convenient and accurate way for analysis of Ethamsylate in its pharmaceutical formulations. Absorbance maxima of Ethamsylate at 300.0 nm (Method A) and in the first order derivative spectra at 288.0 nm (Method B) were selected for the analysis. Method C was area under curve (AUC) and the wavelength range for quantitation was 305.0-295.0 nm. Calibration curve was found to be the concentration range of 10-50 and 5-25 µg/ml for Method A, B and C respectively.

TABLE 1: OPTICAL CHARACTERISTIC AND PRECISION

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Method-A</th>
<th>Method-B</th>
<th>Method-C</th>
</tr>
</thead>
<tbody>
<tr>
<td>λ max (nm)</td>
<td>300.0</td>
<td>288.0</td>
<td>295-305</td>
</tr>
<tr>
<td>Beer’s limits (µg/ml)</td>
<td>10-50</td>
<td>5-25</td>
<td>5-25</td>
</tr>
<tr>
<td>Molar absorptivity (L mol⁻¹ cm⁻¹)</td>
<td>4.125×10⁻³</td>
<td>4.915×10⁻³</td>
<td>0.99388×10⁻⁴</td>
</tr>
</tbody>
</table>

Regression equation (Y = a+bc)

| Slope (b)                  | 0.01488 | 0.01848 | 0.347 |
| Intercept (a)              | 0.0168  | 0.00385 | 0.7655|
| Standard deviation         | 0.0003345 | 0.000404 | 0.0001069 |
| % RSD                      | 0.1014  | 0.143   | 0.001387 |
| Correlation coefficient (r)| 0.9995  | 0.9999  | 0.9999  |

% Range of errors *

| Confidence limit with 0.05 level | 0.0005653 | 0.000427 | 0.00013 |
| Confidence limit with 0.01 level | 0.0008364 | 0.000632 | 0.00015 |
| Limit of Detection (LOD)         | 0.1185   | 0.07214 | 0.00101 |
| Limit of Quantitation (LOQ)      | 0.3592   | 0.2186  | 0.00308 |

Y = bC + a where C is the concentration of Ethamsylate in µg/ml and Y is absorbance unit.

* Average of eight determination.
TABLE 2: EVALUATION OF ETHAMSYSYLATE IN PHARMACEUTICAL FORMULATIONS.

<table>
<thead>
<tr>
<th>Labelled Amount (mg)</th>
<th>Amount of drug found by proposed methods (mg)</th>
<th>(%) Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>T₁</td>
<td>250</td>
<td>249.3</td>
</tr>
<tr>
<td>T₂</td>
<td>250</td>
<td>248.1</td>
</tr>
</tbody>
</table>

* Average of six determination.  
  
T₁ – INDI Pharma (Athamstat)  
T₂ – Dr REDDY’S LABS (Dicynene)
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REFERENCES


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