

A SIMPLE SPECTROPHOTOMETRIC ASSAY OF EDARAVONE IN BULK SAMPLES

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ABSTRACT

A simple, sensitive, rapid and accurate colorimetric method has been developed for the estimation of edaravone in bulk and pharmaceutical dosage forms. The proposed method was based on the formation of chloroform extractable complex of edaravone with wool fast blue. The absorbance of the extractable ion pair complex is measured at the wavelength of maximum absorbance 585 nm against the reagent blank. Results obtained are statistically validated and found to be reproducible.

KEYWORDS: Spectrophotometry, Wool fast blue, edaravone, Pharmaceutical and Formulation

INTRODUCTION

Edaravone (EDR) is a well-recognized lipophilic free radical scavenger for diseases including neurodegenerative disease, cardiovascular disease, and cancer. However, its oral use is restricted due to poor oral bioavailability. Literature survey reveals that a specific, sensitive and fast method based on reverse phase high performance thin layer liquid chromatography (RP-HPTLC)^[1], HPLC^[2-3] HPTLC^[4] and LC-MS/MS^[5] for the determination of edaravone in human, rat plasma and pharmaceutical formulations. No analytical methods reported so far for the estimation of edaravone in pharmaceutical formulations.

MATERIALS AND METHODS

Instrument

All measurement were done on Milton Roy 1001spectrophotometer by using 10 mm matched quartz cuvettes.

Materials

All chemicals used are of A.R. grade and were purchased from S.D. fine chemicals and LOBA-Chemi, Mumbai. Doubled distilled water were used for preparation of solutions

Buffer solution (p H 1.5): Buffer solution is prepared by mixing 289 ml of glycine solution (37.52 gm of glycine and 29.24 gm of NaCl are dissolved in 500ml of distilled water) with 711ml of 0.1 M Hcl.

Wool Fast Blue solution (0.2% w/v)

Wool Fast Blue solution is prepared by dissolving 200 mg of wool fast blue (Fluka) in 100 ml of distilled water.

Preparation of standard stock solution

The standard stock solution (1mg/ml) of edaravone was prepared by dissolving 100mg of edaravone in 100 ml distilled water. The working standard solutions of edaravone were obtained by appropriately diluting the standard stock solution with the same solvent.

Preparation of Calibration curve

Various aliquots of the standard edaravone solution ranging from 0.5-2.5 ml are transferred into a series of separating funnel. To each flask, 1.0 ml of wool fast blue solution, 1.5 ml of buffer solution and 5 ml of chloroform are added. Reaction mixture in each funnel is shaken gently for 5 min and allowed to stand for 5 min so as to separate aqueous and chloroform layer. The chloroform layer is separated out and absorbance is measured at 590 nm, against the reagent blank prepared in similar manner omitting drug solution. Calibration graph is obtained by plotting absorbance values against the concentration of edaravone solution. The calibration curve is found to be linear over a concentration range of 50 to 250 $\mu\text{g/mL}$ of edaravone. The amount of edaravone present in the sample is estimated from the calibration graph. The results are presented in table 2.

Assay of Bulk drugs

For analysis of bulk drugs, 100 mg of the drug was weighted accurately and transferred into a 100 ml volumetric flask and the volume is made up to 100 ml with methanol. The concentration of the drug solutions is now 1mg/ml. This stock solution is further diluted to obtain the working concentration and treated as per the procedure of the calibration curve. Amount of the drug present in sample was computed from respective calibration curve.

Amount of the drug present in sample was computed from respective calibration curve. The results are represented in table 2.

RESULTS AND DISCUSSION

In this method the edaravone treated with wool fast blue dye at 1.5 pH. The resultant solution is extracted with chloroform. The ion pair complex is form in extractable chloroform layer. The absorbance of the extractable ion pair complex is measured at 590 nm against the reagent blank (prepared in a similar manner devoid of drug solution). The calibration curve (concentration vs absorbance) is linear over the range of 50-250 $\mu\text{g/mL}$ of edaravone. The optical characteristics of the proposed method such as absorption maxima, Beer's law limits, molar absorptivity and Sandell's sensitivity are presented in Table 1. The molar absorptivity and Sandell's sensitivity values shows sensitivity of the method. The regression analysis using method of least squares was made for the slope (b), intercept (a) and correlation (r) obtained from different concentrations and results are summarized in the Table 1. The value of correlation coefficient was 0.999, which indicated the good linearity of calibration lines. The percent relative standard deviation calculated from the five measurements of edaravone shown in Table 2. The % RSD is less than 2, which indicates that the method has good reproducibility. The values of standard deviation values are low, indicates high accuracy and reproducibility of the method. The 't' calculated values are compares well with the theoretical value of 2.78 there by indicating that the precision of the method.

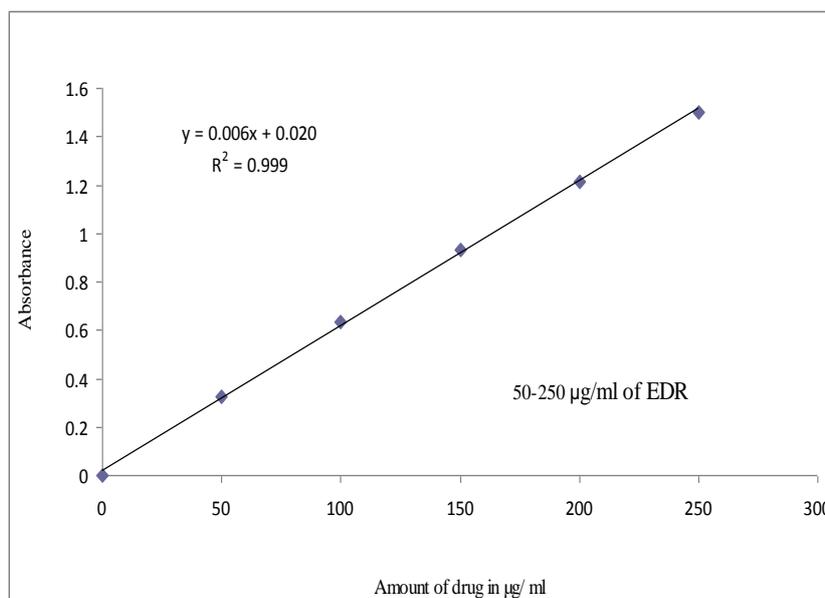
The proposed method is found to be simple, precise, accurate and time saving, reproducible and can be conveniently adopted for routine analysis of estimation of edaravone in bulk drugs samples.

Table 1: Optical Characteristics of the Proposed Method.

| Parameters | Proposed method |
|--|------------------------|
| Wavelength (nm) | 590 |
| Beer's limits, mcg/ml | 50-250 |
| Sandell's, sensitivity, ($\mu\text{g cm}^{-2}$) | 0.0639 |
| Molar absorptivity, ($\text{L mol}^{-1} \text{cm}^{-1}$) | 1.56×10^3 |
| Regression equation, Y^* | $Y = 0.0044x + 0.0011$ |
| Correlation coefficient, (r) | 0.9999 |
| Intercept (a) | 0.0020 |
| Slope (b) | 0.0060 |

Table 2: Assay of Edaravone in Bulk Samples.

| Pure sample(mg) | *Amount found (mg) ±S.D* | % Label claim | * t_{cal} | %RSD |
|-----------------|--------------------------|---------------|-------------|--------|
| 100 | 99.9±0.26 | 99.9 | 0.8431 | 0.2655 |

**Fig.1: Calibration curve of Edaravone.****CONCLUSION**

The developed visible spectrophotometric method was simple, sensitive, accurate, precise, and reproducible and can be successfully applied for the routine estimation of edaravone in bulk samples.

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