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DETERMINATION OF OXYFEDRINE DOSAGE FORMS BY D.D.Q METHOD

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ABSTRACT.

This paper describes the validation of a new spectrophotometric method for the assay of oxyfedrine as in pharmaceutical dosage forms, the DDQ method employs for the estimation of drugs containing amino group and phenolic groups. In this method, the reaction of oxyfedrine with 2,3 – dichloro -5-6 – dieyano -1,4 benzoquinone (DDQ) inchloroform mixture forms honey colour – charge – transfer complex. The honey colour charge – transfer complex solution was stable for more than 24 hrs. The absorbance of the honey colour solution was measured at the wave length 470mm against the regent blank. The calibration graph was obtained by plotting absorbance values against the concentrations of oxyfedrine solution. The calibration curve was found to be linear over a concentration range of 50 to 250 μg / ml of oxyfedrine. The linearity of the curve obtained indicate that it obeys Beer's law. Results of analysis were validated statistically and by recovery studies. The procedures described were successfully applied to the determination of oxyfedrine in tablets.

KEYWORDS: D.D.Q method by speetrophotuetrically oxyfedine, Method of validation.

INTRODUCTION

Oxyfedrine chemically 3–((1–hydroxy –1–phenyl–propan–2 yl)amino]–1–(3–methoxy phenyl) propan – 1 – one. Oxyfedrin is soluble in chloroform. It is commercially available in the markets.

1) ILDAMEN 8mg tablets, 2) ILDAMEN 24mg Tablets

It is used to improve myocardial metabolism.

Survey of literature reveals that various methods were reported for the determination of oxyfedrine in pharmaceutical formulations. Which includes in vitro and In vivo synergism between Tetracycline and the cardio vascular agent oxyfedrine HCl against common.

Bacterial strains 54, Antimicrobial potentiality of a new non – antibiotic, the cardiovascular drug oxyfedrine hydrochloride 55, oxyfedrine in myocardial stunning 56 etc.

The present investigation was under taken with the aim of developing new, simple, rapid and accurate method. Hence this spectrophotometric method based on a charge transfer complexation reaction.

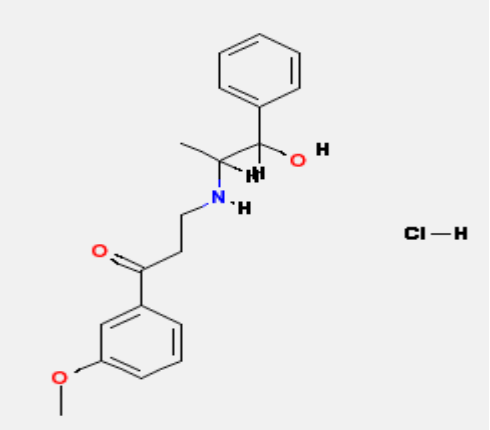

Fig 3.2.4: Structure of oxyfedrine.

EXPERIMENTAL

APPARATUS

1. ELICO UV, Visible double beam spectrophotometer with 10 nm matched quartz cuvettes used for adsorbance values of the drug solution. This instrument provides a unique monochromatic design and a variety of microprocess controlled features to give fast and accurate spectrophotometric measurements.

Reagents

All chemicals used were of analytical reagent grade. Chloroform was used through out the investigation.

D.D.Q: - i.e. 2,3 dichloro -5,6 dicyano – 1,4- Benzoquinone in chloroform.

D.D.Q solution (100 μg/ml), 50 mg was dissolved in chloroform and the resulting solution was made up to the mark in the 50 ml standard flask with chloroform.

2. Oxyfedrin Solution

Pure oxyfedrin solution (50mg) was dissolved in 50 ml chloroform. Futher the stock solution was diluted to 50 ml with chloroform to get working concentration of 50 µg / ml.

Spectrophotometry

The estimation of oxyfedrine by D.D.Q method. In this method was based on the reaction of oxyfedrine with 2,3 – dichloro – 5,6 – dicyano -1,4 – benzoquinone (D.D.Q) to form honey colour charge – transfer complex solution. This honey colour solution was stable for more than 24 hrs. The honey colour solution was used to determine the oxyfedrine spectro photometrically. This sample solution was measured at the wave length range of 400 to 600nm, against the reagent blank. The blank were prepared for this study, the reagent blank containing optimum concentrations of the reagents expect drug.

The absorbance was found to decrease linearly with increasing concentrations of oxyfedrine and this forms the basis for the determination of drug. Finally the estimation of the drug was made through the calibration curve.

PROPOSED ASSAY PROCEDURE

A series of 25ml volumetric flasks, 0.5ml, 1ml. 1.5ml 2.0, 2.5ml of the working standard solution of the drug was pipette into each flask. To each flask, were varying amounts of 0.5 – 2 ml of DDQ reagent solution are added to form honey colour solution. The final volume was brought to 10 ml with chloroform. The resultant solution in each flasks was well mixed and allowed to stand for 5 minutes for complete the reaction The absorbance of the honey colour solution was measured at 470nm by using spectrophotometer against a black solution. The Absorption spectrum of oxyfedrine is presented in figure – 2.

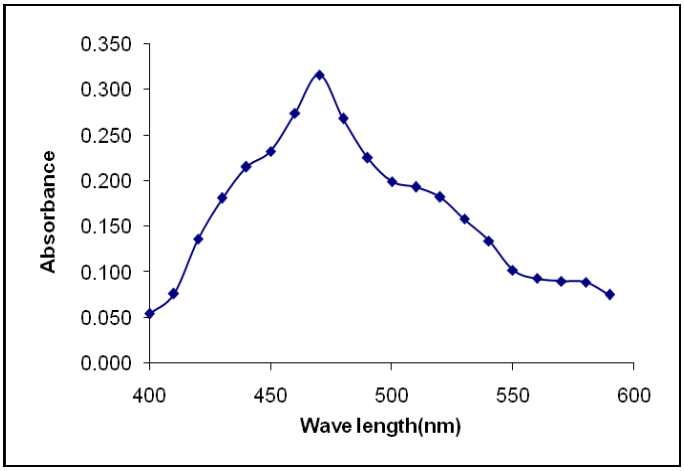

Fig 2: Spectrum of oxyfedrine.

The oxyfedrine curve was obtained by plotting absorbance values against the amount of standard drug. The amount of oxyfedrine present in the sample was computed from the calibration curve are presented in figure – 3 and the result is given in Table – 1.

Table 1: spectral data for calibration curve.

Solvents	Durg solution)
0.5 ml	0.151
1 ml	0.312
1.5 ml	0.452
2 ml	0.613
2.5 ml	0.792

The drug calibration graph was obtained by plotting absorbance values against the concentration of oxyfedrine solution. The calibration curve was found to be linear over a concentration range of 50 to 250 μg/ml of oxyfedrine. The linearity of the curve obtained indicates that it obeys Beer's law. The amount of oxyfedrine present in the sample was read from the calibration graph. The results are present in Figure - 3 and the result is given in table – 1.

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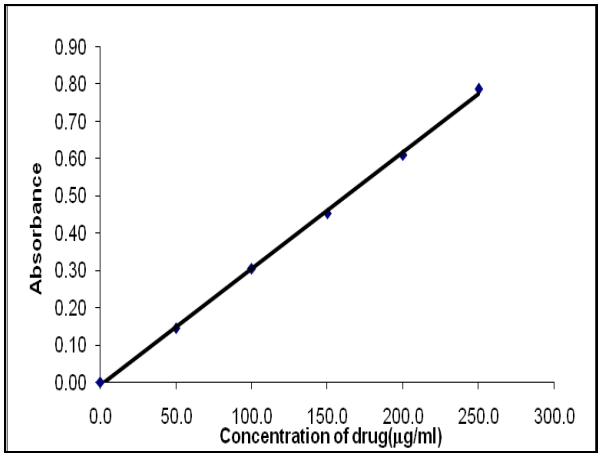

Fig.5.1.2: Calibration curve of oxyfedrine.

Validation of the Method

This method was validated in terms of linearity, accuracy, precision, specificity and reproducibility, of the sample applications. The linearity of this method was investigated by serially diluting the stock solutions of oxyfedrine and measured the absorbance value at 470mm by spectrophotometer. Calibration curves were constructed by plotting the absorbance difference values against the amount of drug in μ g/ml.

Statistical analysis

A statistical analysis was performed on the statistically significant variables using the statistical soft ware. The following pare meters were determined, standard deviation (S.D), Relative standard deviation (RSD) Student t – test, F-test.

The standard deviation (SD) Relative standard deviation (RSD%) and t – test and F– test of variation of the oxyfedrine was calculated from five measurements of replicate samples and the results are summarized in table – 2.

Table 2:- Statistical Analysis of Estimation of oxyfedrine.

Table 1: Assay of oxyfedrine in tablets.

Sample	Labelled amount (mg)	*Amount found by proposed method ± S.D	% of Label claim	RSD%*	t^*_{cal}	F*	*Amount found by Reference method ± S.D
Tablet 1	24	23.953 ± 0.0229	99.8	0.0956	0.6750	1.3418	23.962 ± 0.0265
Tablet 2	24	23.941 ± 0.0290	99.75	0.1213	1.3069	0.8351	23.962 ± 0.0265
Tablet 3	24	23.949 ± 0.0462	99.79	0.1931	0.5971	0.3295	23.962 ± 0.0265

*Average of six determination based on label claim

S.D: Standard deviation.

RSD : Relative standard deviation ‘a’ Calculated ‘t’ value by proposed method.
‘F’ Calculated ‘F’ value by proposed method.

The values of standard deviation and Relative standard deviation are low, indicated high accuracy and reproducibility of this method. The data of assay values of commercial formulations was subjected to statistical evaluation for student ‘t’ test to study the proposed method. The calculated ‘t’ values are less than ‘t’ theoretical values with 4(n-1 = 5-1) degree of freedom at 5% level of significance indicate that there is no significant difference between proposed method and standard method.

RESULTS AND DISCUSSION

The present study was carried out to develop a simple, rapid, sensitive, precise, reproducible and accurate spectrophotometric method for the estimation of oxyfedrine in pharmaceutical dosage forms.

In this method the drug react with DDQ solution to form honey colour charge complex. The honey coloured charge complex solution formed is measured at 470 nm against reagent blank. The amount of drug read from calibration curve. The calibration curve is linear over the range of 50-250 µg/ml of oxyfedrine. The values of Standard deviation, RSD%, t_{cal}, F test, are shown in Table.5.1.4. The values of standard deviation, RSD% are low, indicates high accuracy and reproducibility of the method. The data of assay values of commercial formulations is subjected to statistical evaluation for student ‘t’ test to study the proposed method. The calculated ‘t’ values are less than ‘t’ theoretical values with 4 (n-1= 5-1) degrees of freedom at 5% level of significance indicate that there is no significant difference between proposed method and standard 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 oxyfedrine in bulk drugs samples and pharmaceutical formulations.

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