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SPECTROPHOTOMETRIC DETERMINATION OF OXYFEDRINE IN TABLETS

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ABSTRACT

The present cerimetric method is a novel spectrophoto metric method for the assay of oxyfedrine in pharmaceutical formulations. The proposed new spectrophotometric method is based on a charge transfer complexation reaction of heterocyclic ring containing drug. In this method, the oxidation of oxyfedrine drug by a known excess amount of cerium IV sulphate in acid medium, and then unreacted cerium IV sulphate was treated with iron II sulphate, After 5 minutes the resultant iron III sulphate, solution was treated with (1M) Ammonium thiocyanate, immediately if forms blood red colour of iron IIIsulphate-thiocyanate drug complex. This blood – red colored complex formed under standardized conditions was, measured at 560 nm against reagent blank, The drug calibration graph was obtained by plotting absorbance values against the concentration of oxyfedrine drug solution. The calibration graph was found to be linear over the concentration range of 50 – 250 g/ml for oxyfedrine. The linearity of the curve obtained indicates 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: Oxyfedrine spectrophotometric cerium IV sulphate, Iron III sulphate (ammonium ferrous sulphate solution) Ammonium thiocyanate solution.

INTERODUCTION

Oxyfedrine chemically, 3[(1 – hydroxyl – 1 – phenyl propan – 2yl)amino] – 1 (3 methoxyphenyl) Propan -1-one. oxyfedrine soluble in distilled water. It is commercially available in the markets. i.e. ILDAMEN, 8mg Tablets.

ILDAMEN – 24mg Tablets

It is used to improve myocardial metabolism survey of literature reveals that various methods were reported for the estimation of oxyfedrine in its pharmaceutical formulations.

This drug was describes analysis, which includes in vitro and In vivo synergism between tetracycline and the cardio vascular agent. Oxyfedrine Hcl against common Bacterial strains 54, Anti- microbial potentiality of a new non- antibiotic. The cardiovascular drug oxyfedrine hydrochloride 55, survey of literature search on internet, I came to conclusion that no spectrophotometric method was reported in the literature for the estimation of oxyfedrine in pharmaceutical formulations.

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.

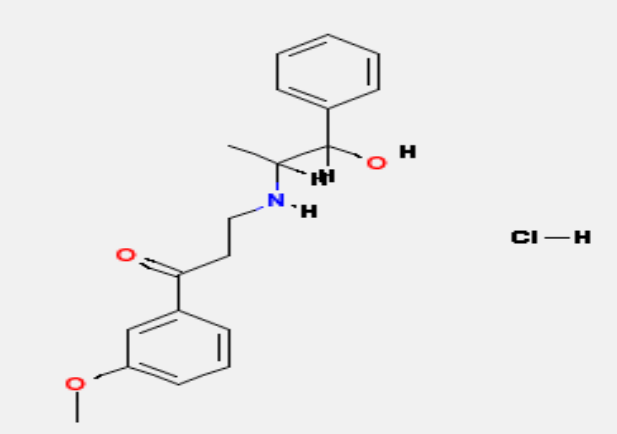

Fig: 3.2.4: Structure of oxyfedrine.

EXPERIMENTAL

Apparatus

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

Reagents

All chemicals were of analytical reagent grade. Double distilled water was used throughout the investigation.

1. Standard Solution of Oxyfedrine Solution

An accurately weighed 50mg of pure oxyfedrine solution is dissolved in double distilled water and the volume was adjusted to 50ml with double distilled water. The stock solution was further diluted to get working concentration of 50 μ g/ml.

2. Cerric Ammoniune Sulphate (0.05M)

2.9826 g of AR cerric Ammonium Sulphate is dissolved in double distilled water and the resulting solution is made up to the mark in the 100 ml standard flask with double distilled water.

3. Ammonium Ferrous Sulphate Solution(0.02M)

0.7842 g of AR Ammonium ferrous sulphate is dissolved in distilled water and the solution is made up to the mark in the 100 ml standard flask with distilled water.

4. Ammonium thio-cyanate (1M): 7 g of AR Ammonium thio-cyanate is dissolved in double distilled water and the resulting solution is made up to the mark in the 100 ml standard flask with double distilled water.

5. Hydrochloric Acid Solution (5N)

Hydrochloric acid solution (5N) is prepared by diluting the requisite volume of concentrated AR hydrochloric acid (Ranbaxy make) with distilled water.

SPECTROPHOTOMETRY

The estimation of oxyfedrine by cerimetric method. In this method was based on the oxidation of the drug by a known excess amount of cerric IV sulphate in acid and oxidation product when unreacted Ce IV sulphate, it oxidized from Iron II Sulphate to Iron III sulphate. After 5 minutes the Iron III sulphate solution was treated with (1M) ammonium thiocyanate, immediately it forms blood – red coloured complex i.e. Iron III sulphate – thiocyanate complex solution. This sample solution was measured at 560nm against a blank solution.

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.

Reaction scheme showing formation of measured Colour

Drug + Ce(IV) ⟹ oxidation product + unreacted Ce(IV) + Ce(III)

Unreacted Ce(IV) + Fe(II) $\xrightarrow{H^+}$ Ce(III) + Fe(III)

Fe(III) + SCN $\xrightarrow{H^+}$ $Fe(SCN)^{2+}$

Blood red coloured complex.

PROPOSED ASSAY PROCEDURE

A series of 25ml of volumetric flasks 0.5ml, 1ml, 1.5ml, 2ml, 2.5ml of the working standard solution of the drug was pipetted into each flask, 1ml of 0.05N cerric ammonium sulphates IV and 1ml of 5N Hydrochloric acid solution and requisite volume of double distilled water are added. The flasks were let stand for 5 minutes with occasional shaking subsequently 1ml of 0.02N ammonium ferrous sulphate was added to each flask and the contents were mixed well, unreacted Ce IV sulphate Iron II to Iron III.

Then after '5' minutes this solution was treated with 3ml of (1M) ammonium thiocyanate solution. It forms blood – red coloured solution of Iron III sulphate thiocyanate complex.

The absorbance of the blood – red coloured solution in each flask was measured at 560nm by using spectrophotometer against a blank solution.

The Absorption spectrum of Oxyfedrine is presented in figure – 2. 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 present in figure – 3 and the results in given in Table -2.

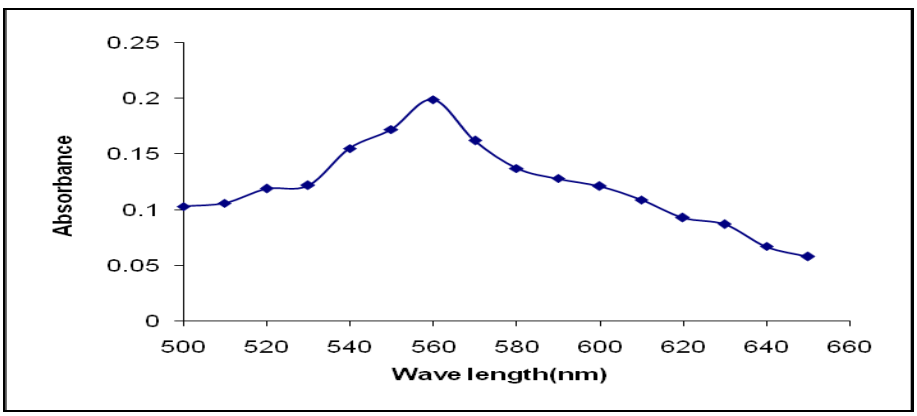

Fig: 4.2.1: Spectrum of oxyfedrine.

Table 1 Spectral data for calibration curve.

Volume of drug solution	Absorbance at 560nm
0.5ml	0.199
1ml	0.412
1.5ml	0.603
2ml	0.795
2.5ml	1.022

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

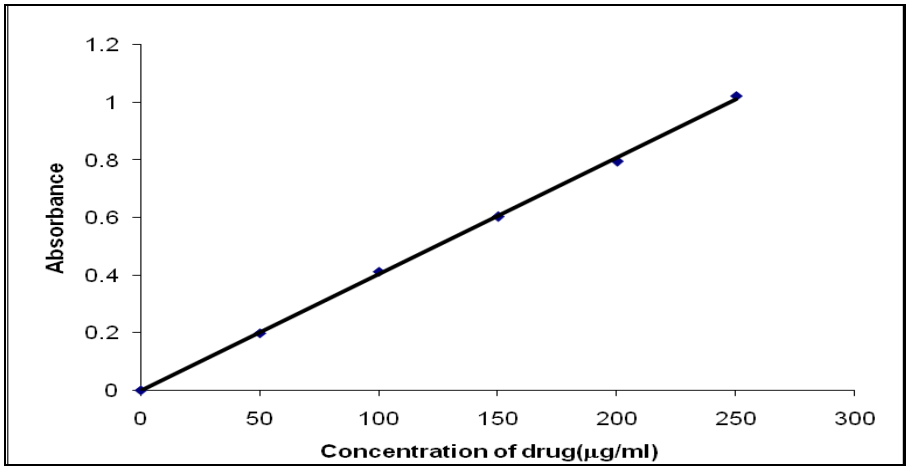

Fig: -0 3 Calibration curve of oxyfedrine.

Val 4.2.2: 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 serially diluting the stock solutions of oxyfedrine and measured the absorbance value at 560 nm by spectrophotometer. Calibration curves were constructed by plotting the absorbance difference values against the amount of drug in μg/ml.

Statistical analysis

A statistical analysis was performed on the statistically significant variables using the statistical software. The following parameters were determined Standard deviation (SD) Relative standard deviation (RSD) and student t-test, F – test.

The standard deviation (S.D) and Relative Standard deviation (RSD, t-test and F-test of the oxyfedrine was calculated from five measurements of replicate samples.

The Assay values oxyfedrim in tablets were shown in Table – 2.

Table. 4.2.7. Assay of Oxyfedrin 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*
Tablet1	24	23.939 ± 0.0248	99.74	0.1040	1.5814	1.1372	23.962 ± 0.0265
Tablet2	24	23.940 ± 0.0252	99.75	0.1055	1.4704	1.1046	23.962 ± 0.0265
Tablet3	24	23.945 ± 0.0255	99.77	0.1067	1.1853	1.0792	23.962 ± 0.0265

***Average of six determinations based on label claim.**

- S.D : Standard deviation
- R.S.D ; Relative standard deviation
- A : Calculated ‘t’ value by proposed method
- ‘F’ Calculated ‘F’ value by proposed method.

The values of standard deviation (SD) and Relative standard deviation (RSD) are low, indicated high accuracy and reproducibility of this method. The data of assay values of commercial for mulations was subjected to statistical evaluation for students ‘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.

RESULTS AND DISCUSSION

The method is based on the oxidation of oxyfedrine by a measured excess of cerium (IV) sulphate in HCl medium, reduction of the residual oxidant by a fixed amount of iron (II) and subsequent formation of iron (III)-thiocyanate complex, which is measured at 560 nm. When a fixed concentration of cerium (IV) sulphate is reacted with increasing concentrations of oxyfedrine, there will be a proportional increase in the concentration of the oxidant. The unreacted oxidant, when treated with a fixed concentration of iron (II) accounts for a proportional increase in the iron (III) concentration. This is observed as a proportional increase in the absorbance of iron (III)-thiocyanate complex with the drug concentration, which formed the basis for the assay of drug. The Standard deviation, RSD%, t_{cal} and F test of the oxyfedrine is calculated from six measurements of replicate samples. The values of

Standard deviation, RSD%, t_{cal} and F test, were shown in Table.4.2.7. 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. Commonly encountered excipients such as starch, talc, glucose, alginate and stearate did not interfere in the proposed methods.

The described method is rapid and reliable, and hence can be used for routine analysis.

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.

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