VISIBLE SPECTROPHOTOMETRIC DETERMINATION OF OXYFEDRINE THROUGH OXIDATIVE COUPLING REACTION IN BULK SAMPLE AND DOSAGE FORMS

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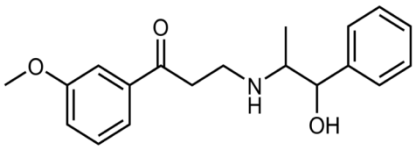
ABSTRACT

A simple, sensitive, highly accurate Visible Spectrophotometric method here been developed for the determination of Oxyfedrine in bulk sample and Dosage forms. This method based on the oxidative coupling reaction of drug with MBTH(3-methyl-2-benzothialinone hydrazone hydrochloride) in presence of sodium periodate ($NaIO_4$) to form colored chromogen exhibiting λ_{max} 632 nm. Beer's law was obeyed in the concentration range of 1-6 µg ml^{-1} .The results of analysis for the methods have been validated statistically and by recovery studies.

Key Words: Oxyfedrine, Visible Spectrophotometry, MBTH.

INTRODUCTION

Oxyfedrine is a vasodilator. It was found to depress the tonicity of coronary vessels. Oxyfedrine improves myocardial metabolism and also exert a positive chronotropic and inotrophic effects which prevent precipitating angina pectoris. Oxyfedrine improves myocardial metabolism to sustain stress due to hypoxia. It has beta-agonist action in low doses but f3- blocking properties in high doses. The IUPAC name of Oxyfedrine is 3-[(2-Hydroxyl-1-methyl-2-phenylethyl)amino]-1-(3-methoxyphenyl)propan-1-one.



Structure of Oxyfedrine

Oxyfedrine hydrochloride is a white crystalline powder. Soluble in chloroform, methanol,ethanol, ethyl acetate and water. A literature survey reveals some Spectrophotometric methods. No visible spectroscopic method for dosage forms of Oxyfedrin reported so far.

The objectives of the work are to develop new visible spectrophotometric method for the estimation of Oxyfedrine in bulk and tablet dosage form with good accuracy, simplicity, precision and economy. Hence the present work deals with the Spectrophotometric estimation of Oxyfedrine using MBTH with sodium periodate ($NaIO_4$). Some other drugs were estimated successfully using MBTH with sodium periodate ($NaIO_4$).

MATERIALS AND METHODS

UV-Visible spectrophotometer: Systronics model(117), wavelength 190-1100nm, high stability, linearity; precision instrument was used for all the spectral measurements.

Reagents: All the chemicals and reagents were of analytical grade and the freshly prepared solutions were used in the present work.

MBTH solution: Prepared by dissolving 200 mg of MBTH in 100 ml distilled water.

($NaIO_4$) solution: Prepared by dissolving 200 mg of sodium meta periodate in 100ml distilled water.

Preparation of standard stock solution of Oxyfedrine: Oxyfedrine (100mg) was accurately weighed and transferred into a standard 100ml volumetric flask and dissolved in distilled water. The final volume was made up to the mark with distilled water. The concentration of the solution is 1mg/ml.

Preparation of working standard solution of Oxyfedrine: 10ml of standard stock solution was transferred into another 100ml volumetric flask and make up it distilled water to get 100µg/ml solution.

Optimization of Reaction Conditions:

Time for Maximum Colour Development and Stability: Time scan was carried out to find the time required for maximum colour development. It was noticed that a minimum of 30 minutes required for maximum colour development(Figure 2). It was also observed that the colour is stable for 5 hours.

Effect of Buffer: Colour development of drug with reagents (MBTH & $NaIO_4$) is carried out in acidic, basic and neutral medium. But it was found that neutral medium has more absorbance compared to the other two (Table 1).

Order of Addition: Different order of additions is carried with Drug, MBTH and $NaIO_4$. But, It was found that $NaIO_4$, Drug and MBTH gave good results (Table 2).

Effect of Concentration of $NaIO_4$: Colour development experiment was carried out by changing the concentration of $NaIO_4$ from 0.5ml to 2.5ml. It was found that 1.0ml of $NaIO_4$ is optimum concentration (Table 3).

Effect of Concentration of MBTH: Colour development experiment was carried out by changing the concentration of MBTH from 0.5ml to 2.5ml. It was found that 1.0ml of MBTH is optimum concentration (Table 4).

Effect of Temperature: It was observed that a slight decrease in the absorbance when temperature increased. So room

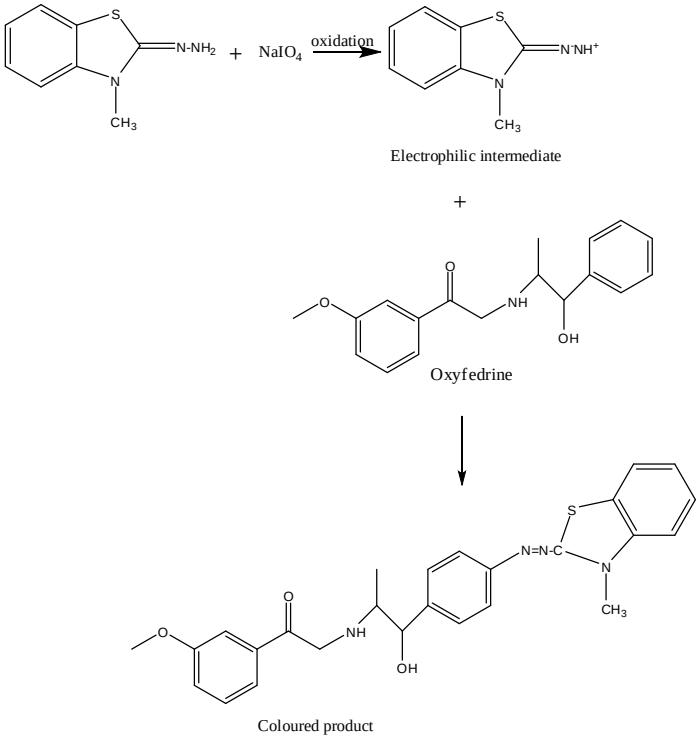
temperature is optimum for the colour development.

Recommended procedure: Into a series of 25ml volumetric flasks 1.0ml of 0.2% $NaIO_4$ was added. To the flasks aliquots of working standard Oxyfedrine solution (0.3, 0.6, 0.9, 1.2 and 1.5ml) was added. After that 1.0ml of 0.2% MBTH solution was added to each flask and kept aside for 30 minutes. The volume was made up to the mark with distilled water. The absorbance was measured at 632nm against similar reagent blank. The amount of Oxyfedrine was determined from the calibration curve (Figure 3).

Procedure for the assay of Oxyfedrine in pharmaceutical dosage forms: Twenty five tablets were weighed accurately and reduced to fine powder, drug equivalent to 100 mg of Oxyfedrine taken in a 100 ml volumetric flask, dissolved in distilled water, and the volume was made upto the mark with distilled water, filtered by using Whattmann-42 filter paper. The filtrate was quantitatively diluted with distilled water to yield concentrations in the linear range of the assay of Oxyfedrine.

RESULTS AND DISCUSSION

Oxyfedrine possesses different functional groups such as secondary amine, keto and hydroxyl of varied reactivity. In recent years there has been growing interest in the role of sodium metaperiodate, a specific oxidant can be used as an analytical reagent in the assay of drugs. The method is based on the oxidative coupling reaction with MBTH in the presence of $NaIO_4$. Under the reaction conditions, MBTH loses two electrons and one proton on oxidation, forming the electrophilic intermediate which can be substituted on Oxyfedrine to form a blue coloured products. Probable mechanism of the reaction was given in the scheme below.



Scheme.1.Reaction of Oxyfedrine with MBTH in the presence of $NaIO_4$

Table.1.Effect of Buffer on Colour Development

Medium	Absorbance
Acidic	0.190
Basic	Turbidity
Neutral	0.740

Table.2.Order of Addition

1	2	3	Absorbance
Drug	$NaIO_4$	MBTH	1.705
Drug	MBTH	$NaIO_4$	1.752
$NaIO_4$	Drug	MBTH	1.861
$NaIO_4$	MBTH	Drug	0.277
MBTH	Drug	$NaIO_4$	1.469
MBTH	$NaIO_4$	Drug	0.493

Table.3.Variation of Absorbance w.r.t $NaIO_4$

Volume of $NaIO_4$ (0.2%)	Absorbance
0.5 ml	0.187
1.0 ml	0.653
1.5 ml	0.518
2.0 ml	0.335
2.5 ml	0.251

Table.4.Variation of Absorbance w.r.t MBTH

Volume of MBTH (0.2%)	Absorbance
0.5 ml	0.306
1.0 ml	0.721
1.5 ml	0.569
2.0 ml	0.322
2.5 ml	0.101

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Figure.1.Absorption spectrum of oxyfedrine with MBTH-$NaIO_4$

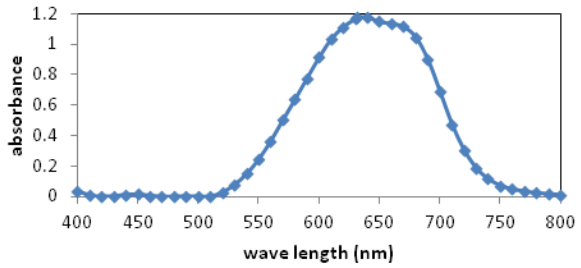


Figure.2.Time scan: Progress of reaction of Oxyfedrine with MBTH-NaIO4

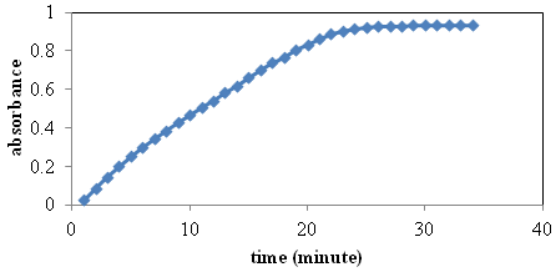
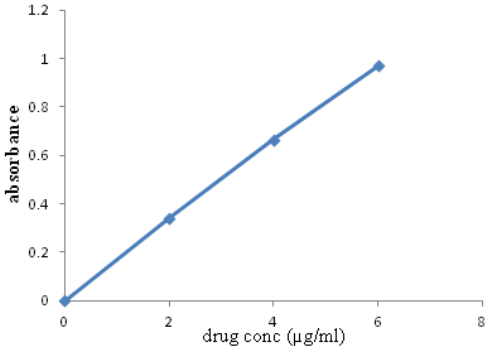


Figure.3.Beer's Law plot of Oxyfedrine with MBTH-$NaIO_4$



CONCLUSION

The proposed two methods were found to be simple, selective and sensitive. The statistical parameters and recovery study data clearly indicate the reproducibility and accuracy of the methods. Analysis of the authentic samples containing Oxyfedrine showed no interference from the common excipients. Hence, these methods could be considered for the determination of Oxyfedrine in the quality control laboratories.

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