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AN INDIRECT SPECTROPHOTOMETRIC DETERMINATION OF FUROSEMIDE IN PHARMACEUTICAL PREPARATIONS

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ABSTRACT

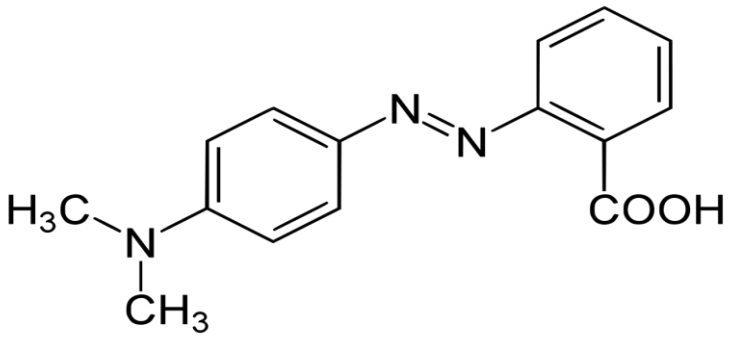
A new, simple, sensitive indirect spectrophotometric method for determination of Furosemide has been developing. The method involved addition of a known excess of sodium hypochlorite to in acidic medium, followed by determination of residual sodium hypochlorite by reacting with a fixed amount of methyl red, measuring the absorbance at 521nm. In this method the amount of sodium hypochlorite reacted correspond to the amount of Furosemide and the measured absorbance was found to increase linearly with the concentration of Furosemide, which is corroborated by the correlation coefficient of 0.9996. Beer's law plot showed a good Correlation in the concentration rang 1-18 µg mL $^{-1}$. The apparent molar absorptivity were calculated to be1.2 $\times 10^{4}$l.mol^{-1}. cm^{-1}. The limit of detection (LOD) and quantification (LOQ) were calculated to be 0.019 and 0.0527µg mL-1. The relative standard deviation (RSD) was less than 1.7% (n=5). The method was applied to the determination of furosemide in pharmaceutical preparations (tablets and injection).

KEYWORDS: Furosemide, Spectrophotometry, Pharmaceutical preparations.

INTRODUCTION

Furosemide (FUR), chemically known as 5-(aminosulfonyl)-4-chloro-2-[(2- furanylmethyl) amino] benzoic acid (Figure 1), is structurally a sulfonamide, an antibacterial agent. However, FUR is a potent diuretic widely used in the treatment of edematous states associated with cardiac chronic renal failure, hypertension, congestive heart, failure, and cirrhosis of the liver.[1] The literature survey reveals that various methods has been reported for determination of FUR. The official methods for the determination of FUR in dosage

forms are based on titrimetry[2], spectrophotometry[3] and HPLC.[4] Besides, there are number of other techniques available in the literature and include, derivative UV spectrophotometry[5], spectrofluoremetry[6], HPLC with UV detection[7], HPLC with LC-L Cdetection[8], ratio-spectra derivative spectroscopy[9] and diffuse reflectance spectroscopy[10], developing a selective and sensitive methods using visible spectrophotometry is of paramount importance.



Chemical structure of furosemide Figure.[1]

M.wt=330.77

Experimental

Apparatus

JASCO V-630 Spectrophotometric with 1.0 cm quartz cells was used for the absorbance measurements.

Reagents

All chemicals used were of analytical purity grade and all solutions were prepared with distilled water.

A standard sodium hypochlorite solution (0.1%) was prepared by dilution of 2mL of 5% sodium hypochlorite to 100 mL with distilled water.

Methyl red 0.01% was prepared by dissolving 0.01 g accurately weighed dye in ethanol and diluting it to 100 mL in volumetric flask.

Standard solution of Furosemide was prepared by dissolving 0.1 g of pure drug in 1L distilled water.

Analytical procedures

Different aliquots of Furosemide standard solution equivalent 1-18µg were transferred into a series of 25mL volumetric flasks, 3mL of 0.1N HCL, and 0.5 ml of sodium hypochlorite solution were added. The content was mixed and let stand for 5 min with occasional shaking. Finally, 3mL of 0.1% methyl red solution was added and the volume was diluted to the mark with distilled water and mixed well. The absorbance of each solution was measured at 521 nm against a reagent blank.

Preparation of Furosemide drugs

Tablets

To minimize a possible variation in the composition of the tablets, the mixed content of 20 tablets, were weighed and grounded, then the powder equivalent to 100 mg of Furosemide was stirred well with water for 15min and the volume was made to 1L with distilled water, filtered through whatman No. 42 filter paper.

Injections

5ml vial containing 100 mg of Furosemide was transferred into 1L volumetric flask and diluted up to the mark with distilled water,

RESULT AND DISCUSSION

The versatility of sodium hypochlorite as an analytical reagent can be gauged by its applications in the spectrophotometric determination of many organic compounds of therapeutic importance. The use depends mainly on its ability to affect the oxidation of diverse functional groups.[11-13] Taking advantage of the rapid oxidation reaction of sodium hypochlorite with Furosemide. Furosemide is a reducing agent owing to the presence of thiol group(-SH) in its structure. The proposed spectrophotometric methods are indirect and based on the determination of residual sodium hypochlorite after bringing the reaction between Furosemide and sodium hypochlorite to completion. The residual sodium hypochlorite was determined by methyl red indicators. When added in increasing concentration to a fixed concentration of sodium hypochlorite, Furosemide consumes the latter proportionally and there is a concomitant drop in the remaining concentration of sodium hypochlorite. When a indicator concentration is added to decreasing concentration of sodium hypochlorite, a concomitant increase in the indicator concentration result a proportional increase in absorbance at the respective ג max is observed with increasing concentration of Furosemide.

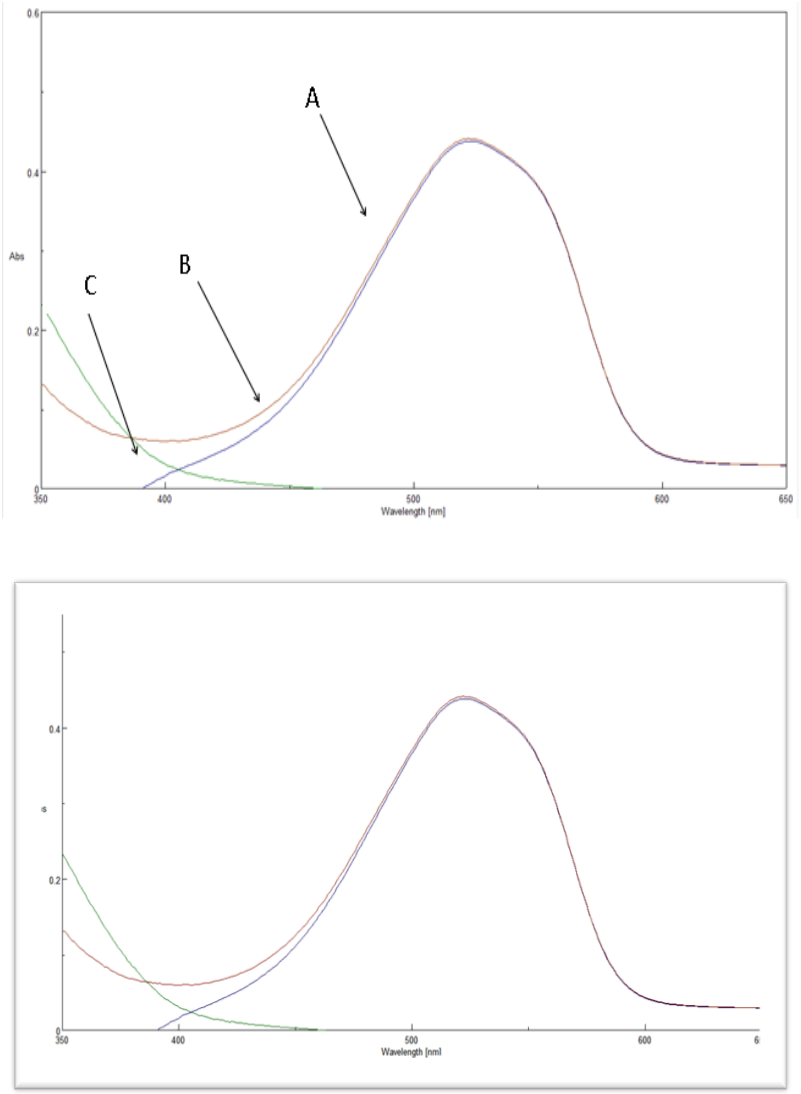


Figure 2: Absorption spectra of A- µg /ml of Furosemide against reagent blank, B- µg/ml of Furosemide against distilled water, C- blank against distilled water.

Method Validation

Under the optimized conditions, A liner correlation is showed in Figure 3 was found between absorbance max and furosemide concentration, rang 1-18 µg mL^{-1}. The correlation coefficient was 0.999 the slop of curve was 0.036, y= 0.0367 + 0.0068, R = 0.9993, n = 10.

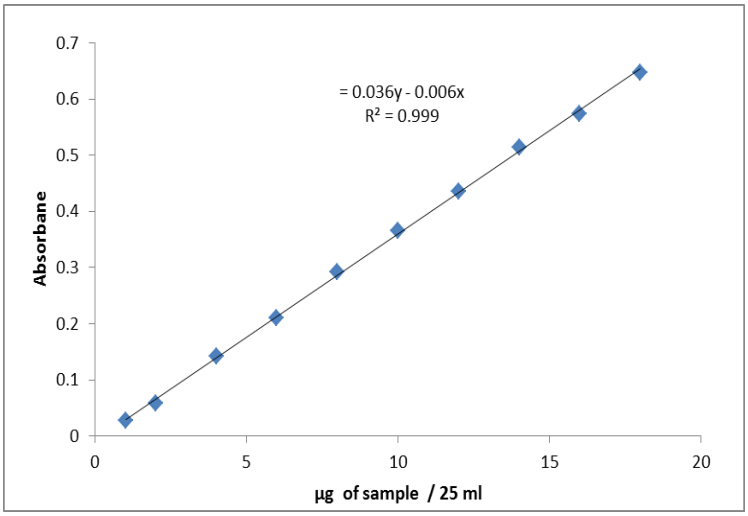


Fig 3; Calibration curve of Furosemide.

The limit of detection (LOD) and quantification (LOQ) was calculated using the formula LOD= 3.3σ/S and LOQ 10 σ/S where is the standard deviation of ten reagent blank determination and s is the slope of the calibration curve (14). The results also presented in table[1] and reveal the high sensitivity of the present method Table [1]: Optimum reaction conditions.

Table 1: Analytical and regression parameters of the proposed methods.

Linearity range (µg /ml)	1-18
Limit of detection(LOD)*(µg /ml)	0.01901
Limit of quantitation(LOQ)*(µg /ml)	0.06337
Molar absorptivity(l.mol-1.cm^{-1})	1.20069×10^{4}
Sandell's sensitivity (µg/Cm^{2})	0.00275
Slope	0.0367
Intercept	0.0068 -
Correlation coefficient	0.9993

For ten determination*

Accuracy and precision

To evaluate the accuracy and precision of the method a pure drug solution was analyzed four different concentration, each determination being repeated five times. the relation error(%) and relative standard deviation (RSD) values were summarized in table (2). From table (2) it was clear that relative error ± 1.3% was as accurate Moreover, the method was found to be precise with RSD values < 1.7%.

Table 2: Accuracy and precision of the proposed method.

Furosemide taken (µg)	Recovery	Average 0f recovery	Er(%)	RSD%
1	98.64		-1.36	0.79
4	99.44	99..88	-0.559	1.67
6	100.9		0.935	0.213
10	100.54		0.54	1.47

Applications

Table(3) give the procedures was applied for the assay of pharmaceutical preparation of the two drug was studied and the recovery results was higher than 98% indicating that successfully applicability of the proposed method.

Table 3: Determination of Furosemide. in pharmaceutical formulations.

Pharmaceutical preparation	Certified value (mg)	found	Recovery (%)
Furosemide/tablets	40mg/tab	39.62	**99.07**
Lazine/injecting	20mg/amp	20.2	**101.38**

Mean value of five determination.

CONCLUSION

The proposed method developed was simple, selective and a wide range of determination without the need for heating or solvent extraction. The proposed method don't take more than 10 mints and successfully applied to the determination of furosemide in Pharmaceutical preparation.

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