



Estimation of Furosemide Spectrophotometrically in Pharmaceutical preparations by Oxidative Coupling Reaction

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ABSTRACT

A simple, rapid and sensitive spectrophotometric method was described for the determination of Furosemide (FUR). The method is based on the oxidative coupling reaction between Furosemide and 1-Naphthylamine-4-sulfonic acid in the presence of potassium permanganate to form a yellow colored product with maximum absorptions at 465nm, which is soluble in water. Beer's law is obeyed in the concentration range of 3 to 23 $\mu\text{g} \cdot \text{ml}^{-1}$ with a molar absorptivity of $1.065 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$, and Sandell's sensitivity of $0.0310 \mu\text{g} \cdot \text{cm}^{-2}$, respectively. The correlation coefficient of 0.9994, with recovery average % of 99.948. Limit of detection (LOD), limit of quantification (LOQ) of $0.971 \mu\text{g} \cdot \text{ml}^{-1}$ and $3.237 \mu\text{g} \cdot \text{ml}^{-1}$ and a relative standard deviation (RSD) % of 0.641 to 0.812. The proposed method has been used to successfully determine furosemide in pharmaceutical formulations (Tablet).

1. Introduction

Furosemide is a loop diuretic that is used to treat fluid retention in the body caused by heart failure, liver scarring, or renal illness. It can be used to treat high blood pressure as well [1]. (FUR) is an abbreviation for furosemide, which is also known as frusemide [2]. In 1959, furosemide was patented, and in 1964, it was licensed for medicinal usage [3]. There are several chemical names for furosemide [5- (aminosulfonyl)-4-chloro-2-[(2-furanylmethyl) amino] benzoic acid, 4-chloro-N-furfuryl-5-sulfamoylanthranilic acid, 4-chloro-N-(2-Furylemethyl-5-sulfamoylanthranilic acid, 4-chloro-2-furfurylamino-5-sulphamoyl benzoic acid] (See Fig. 1). Chemical formula is $\text{C}_{12}\text{H}_{11}\text{ClN}_2\text{O}_5\text{S}$ and molecular weight is 330.745 g/mole it's a low soluble in water, chloroform, and ether but it is soluble in acetone, methanol, and dimethyl formamide [4]. FUR is a crystalline powder that is white to slightly yellow in color and has a melting point of 206°C [5]. Furosemide has potential interactions with these medications: Aspirin and other salicylates, other diuretics (e.g. ethacrynic acid, hydrochlorothiazide), Synergistic effects with other antihypertensives (e.g. doxazosin), and Sucralfate [6]. For the determination of furosemide, various analytical

approaches have been used, spectrophotometry [7-10], HPLC [11-14], and other methods [15-20].

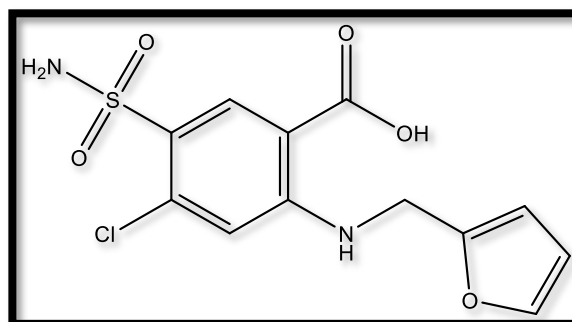


Fig.1: Chemical Structure of Furosemide

The purpose of this study is to develop and validate a simple, sensitive, and specific spectrophotometric method for estimating furosemide in pharmaceutical formulations.

2. Experimental Part

2.1. Instrumentation Used:

Spectrophotometric measurements were made using UV-visible double beam a type (T92+ Spectrophotometer, China), with using 1cm of matching quartz cells.

2.2. Materials and Solution of the Used:

The substances employed in this study were all extremely pure equipped by my company (Fluka, bdh, SDI), and throughout the tests, methanol and distilled water were employed as solvents to preparing solutions.

Furosemide standard solution ($1000 \mu\text{g}.\text{ml}^{-1}$)

It was prepared by dissolving 0.1000 g of furosemide powder in an amount of methanol and then completed the volume to the mark in a 100 ml volumetric flask, and Concentration $250 \mu\text{g}.\text{ml}^{-1}$ was prepared by taking 25 ml of the standard solution ($1000 \mu\text{g}.\text{ml}^{-1}$) and easing it in a volumetric flask of 100 ml and filled the volume to the mark with the distilled water.

Potassium hydroxide of Solution (0.1 Molar)

It was prepared by dissolving 0.561g of the substance in a little distilled water and completed the volume to the mark with the same solvent in a volumetric flask of 100 ml.

Solution of potassium permanganate (1×10^{-2} Molar)

It was prepared by dissolving 0.158g of potassium permanganate in an amount of distilled water (D.W) and filled the volume to the mark with the same solvent in a volumetric flask of 100 ml.

Solution of 1-Naphthylamine-4-sulfonic acid (2×10^{-2} Molar)

It was prepared by dissolving 0.446 g of the reagent in an amount of distilled water (D.W), and then filled the volume to the mark with the same solvent in a volumetric flask of 100 ml capacity.

Solution of pharmaceutical preparation (Tablets)

Ten tablets are weighed (40 mg/ Tablet by company SDI), and the grains are crushed well, then a certain weight of the powder is taken, which is equivalent to 0.1g of furosemide, depending on the type of tablets used, and it is dissolved in a little of methanol and then filtered to separate the insoluble components, if any, then transferred to a volumetric flask of 100 ml capacity, and completed the volume to the mark with distilled water and then 25 ml is taken of this solution and transferred to a volumetric flask of capacity of 100 ml and filled the volume to the mark with distilled water to obtain a solution of $250 \mu\text{g}.\text{ml}^{-1}$.

The method's general principle

Principle is the coupling of the reagent 1-naphthylamine-4-sulfonic acid with the drug furosemide and in the presence of the oxidizing agent potassium permanganate in an alkaline medium, a solution with a yellow color is formed, which gave the highest wavelength at 465 nm versus the blank solution.

3. Results and discussion

3.1. Preliminary study

• Studying the absorption spectrum of the drug only

The absorption spectrum of the $250 \mu\text{g}.\text{ml}^{-1}$ of drug was recorded against the methanol as a reference. This was done by transferring 2 ml of the $250 \mu\text{g}.\text{ml}^{-1}$ solution into a 25 ml volumetric flask and then filled the volume with methanol to the mark. The absorption spectrum was taken as in Figure (2), which shows that the drug gives absorption at the wavelength of 340 nm.

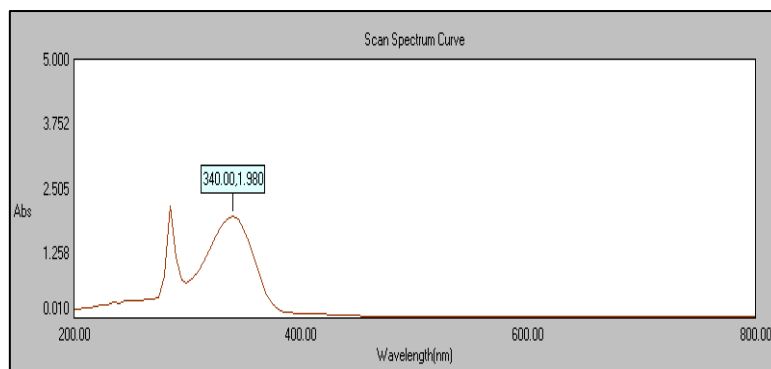


Fig. 2: Absorption spectrum of furosemide versus methanol

• Studying the absorption spectrum of the drug with the reagent 1-Naphthylamine-4-sulfonic acid

Where 2 ml of a solution of the drug with a concentration of $250 \mu\text{g}.\text{ml}^{-1}$ was mixed with 2 ml of a reagent solution of 2×10^{-2} M, 1 ml of potassium permanganate solution of 1×10^{-2} M, and 1 ml of a solution of potassium hydroxide of 0.1 M in a volumetric flask with a capacity of 25 ml, then filled

the volume to the mark with distilled water with shaking to homogenize the solutions. The absorption spectrum of the resulting yellow solution, was measured against the versus blank solution as a reference to obtain the wavelength and the highest value for the absorption was given at the wavelength of 465 nm, as in Figure (3).

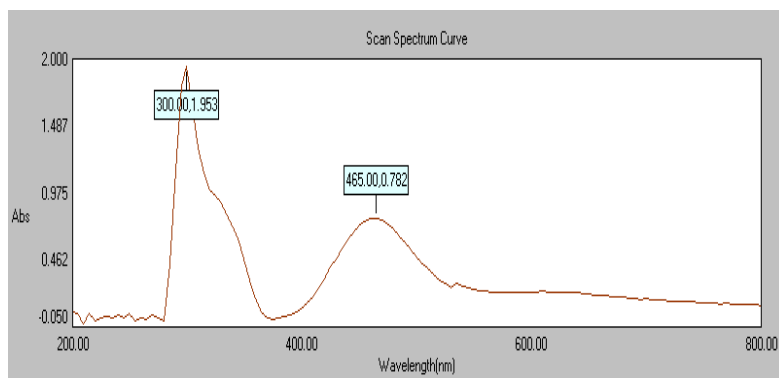


Fig. 3: Absorption spectrum between the drug and the reagent in the presence of potassium permanganate

3.2. Choosing the best reagent

1 ml of each solution were taken from the used reagents, 2 ml of furosemide solution at a concentration of $250\mu\text{g}\cdot\text{ml}^{-1}$, 1 ml of potassium permanganate solution as an oxidizing agent, and 1

ml of potassium hydroxide solution of 0.1 M and the results shown in Table No. (1). we note that the 1-naphthylamine-4-sulfonic acid reagent gave the highest absorption of the colored product formed at 465 nm versus the blank solution.

Table 1: Choosing the best reagent

2×10^{-2} M of Reagent	Variable	Absorbance	λ_{max}
1-Naphthylamine-4-sulfonic acid	SB	0.782	465
	BW	0.147	408
1,5-Diaminonaphthalene	SB	0.513	580
	BW	0.139	497
4-aminobenzenesulfonic acid	SB	0.443	438
	BW	0.108	400

SB: It symbolizes the absorption spectrum of Furosemide solution compared to a blank solution

BM: It symbolizes the absorption spectrum of the blank solution compared to Methanol.

3.3. Volume impact of the coupling reagent:

Impact of the volume of reagent 1-Naphthylamine-4-sulfonic acid on the intensity of absorption was studied. Where a series of volumes were taken from the used (1.3-3) ml reagent with a concentration of 2×10^{-2} M, 2ml of the furosemide solution of $250\mu\text{g}\cdot\text{ml}^{-1}$, 1ml of the oxidizing agent solution, and 1ml of potassium hydroxide solution of 0.1 M. It was found that adding 2ml of the reagent was the best to give it the highest absorption. Results are shown in the Table (2).

Table 2: Impact of volume reagent

2×10^{-2} M of Reagent, ml	Absorbance	
	BW	SB
1.3	0.138	0.717
1.5	0.150	0.752
1.8	0.166	0.764
2	0.148	0.782
2.3	0.160	0.752
2.5	0.173	0.738
2.8	0.186	0.692
3	0.198	0.674

3.4. Choosing the best oxidizing agent:

Several experiments were carried out to find the best oxidizing agent to form the colored product. Several Solutions of oxidizing agents were used of 1×10^{-2} M, each with a volume of 1 ml, and 2 ml of 1-Naphthylamine-4-sulfonic acid solution and added 1 ml of the potassium permanganate solution were has a concentration of 1×10^{-2} M in a volumetric flask with a capacity of 25 ml. It was noted that the best

oxidizing agent was potassium permanganate, which gave a maximum absorption. Results are in the Table (3).

Table 3: oxidizing agent choosing

1×10^{-2} M of Oxidizing agent	Absorbance		$\lambda_{\text{max}}(\text{nm})$
	Blank	Sample	
Potassium permanganate	0.147	0.782	465
Potassium persulfate	0.132	0.620	432
Potassium Iodate	0.118	0.513	485
Ammonium ferric sulfate	0.102	0.496	502

3.5. Volume effect of oxidizing agent:

effect of the volume of oxidizing agent solution with a concentration of 0.1 M on the intensity of absorption was studied, as different volumes (0.3-2) ml were used and it was found that 1 ml of potassium permanganate solution is the best which gives highest absorption. Results are in the Table (4).

Table 4: Volume effect of oxidizing agent

1×10^{-2} M of KMnO_4 , ml	Absorbance	
	BW	SB
0.3	0.102	0.440
0.5	0.125	0.554
0.7	0.138	0.675
1	0.146	0.782
1.3	0.127	0.643
1.5	0.114	0.590
1.7	0.101	0.478
2	0.087	0.427

3.6. Choosing the best base:

1 ml of different types of bases with a concentration of approximately 0.1 M were used and their impact

on the intensity of absorption was studied. Results are shown in the Table (5).

Table 5: Choosing of best base

Base Solution 1×10^{-1} M	KOH	Na_2CO_3	$\text{Ca}(\text{OH})_2$	NaOH
Absorbance	0.782	0.665	0.570	0.427

From the above table, we note that the potassium hydroxide gave the highest absorption and therefore it was chosen in the subsequent experiments.

3.7. Volume impact of the base:

Different quantities of the base used were added of potassium hydroxide 0.1M to find out the optimum amount, it was found that 1 ml is the best which gives highest absorption. Results are shown in the Table (6).

Table 6: volume impact of the base

1×10^{-1} M of KOH, ml	Absorbance	
	BW	SB
0.3	0.133	0.485
0.5	0.137	0.531
0.7	0.142	0.668
1	0.146	0.781
1.3	0.140	0.690
1.5	0.130	0.568
1.7	0.123	0.531
2	0.116	0.456

3.8. Sequence of additions Impact:

It was found that the best addition sequence which gives the highest absorption in (D+R+O+B). Results are shown in the Table (7).

Table 7: sequence of additions Impact

No.	Order of Addition	Absorbance	
		BW	SB
I	D+R+O+B	0.147	0.782
II	O+R+D+B	0.132	0.664
III	R+B+O+D	0.118	0.583
VI	B+O+D+E	0.106	0.479

Table 9: Impact of Temperature

Temp. °C	15	20	25	30	35	40	45	50	55	60	65	70
Absorbance	0.573	0.642	0.782	0.760	0.731	0.701	0.684	0.652	0.625	0.572	0.548	0.492

3.11. The Product Stability:

The stability was studied using different time periods at room temperature (25°C) to know the stability of the formed product, This study was carried out by taking 2 ml of furosemide solution with a concentration of $250 \mu\text{g} \cdot \text{ml}^{-1}$ representing concentration $20 \mu\text{g} \cdot \text{ml}^{-1}$ and adding 1 ml of solution potassium permanganate with a concentration of 1×10^{-2} M, then add 2 ml of 1-Naphthylamine-4-sulfonic acid reagent solution with a concentration of 2×10^{-2} M, then add 1 ml of potassium hydroxide solution of 0.1 M in a volumetric flask of 25 ml, then complete the volume to the mark with distilled water, The absorption value of the formed colored product was observed to be stable for 60 minutes. Which is sufficient time to complete many of measurements. The results shown in Table (10).

Potassium permanganate (O), reagent 1-Naphthylamine-4-sulfonic acid(R), Furosemide (D), and base (B).

3.9. Impact of time:

The time was studied by taking a series of volumetric flask with a capacity of 25 ml containing 2 ml of furosemide solution at a concentration of $250 \mu\text{g} \cdot \text{ml}^{-1}$, then adding to it 2 ml of the reagent solution of 1-Naphthylamine-4-sulfonic acid with a concentration of 2×10^{-2} M and then added 1 ml was from the solution potassium permanganate at a concentration of 1×10^{-2} M then was added to it 1ml of potassium hydroxide solution a concentration of 0.1M, and the solutions were left for different periods of time after which they were diluted with distilled water to the mark. Then the absorbance of the solutions was measured at the wavelength of 465 nm against their blank solutions. The results are shown in the Table (8).

Table 8: The impact of time

Time/min	2	5	10	15	20
Absorbance	0.781	0.672	0.548	0.530	0.518

The above table shows that 2 minutes is enough time to complete the oxidation and conjugation process, so this time was used in the following experiments.

3.10. Impact of temperature:

The formed product was studied using different temperatures, and it was found from the results that the absorbance remains stable within the range (15-70) degrees Celsius, and the absorbance decreases at high temperatures, and it was found that the colored product was gives the highest absorption at room temperature (25 °C). Results are shown in the Table (9).

Table 10: The Stability of the Reaction Product

20 $\mu\text{g} \cdot \text{ml}^{-1}$ of FUR	
Time (min)	Absorbance
5	0.697
10	0.729
15	0.761
20	0.782
30	0.781
40	0.782
50	0.781
60	0.781
70	0.767
80	0.749

3.12. Spectrum of ultimate absorption:

The final absorption spectrum was measured after fixing the optimal conditions using 2 ml of furosemide solution at a concentration of $250 \mu\text{g} \cdot \text{ml}^{-1}$, 2ml of reagent solution of 1-Naphthylamine-4-sulfonic acid at a concentration of 2×10^{-2} M, 1ml of potassium permanganate at a concentration of 1×10^{-2} M, and 1ml of potassium hydroxide at a

concentration of 0.1M, and complete the volume to the mark in a 25ml volumetric flask with distilled water, then final absorption spectrum of the yellow product was measured against the blank solution, it was found that it gives the highest absorption at the wavelength 465 nm .As shown in Figure (4).

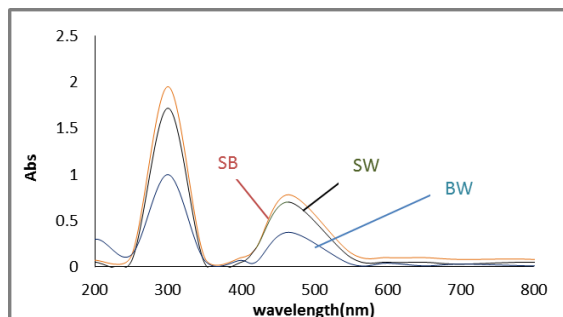


Fig. 4: Final absorption spectrum for the determination of furosemide

SB: It symbolizes the absorption spectrum of Furosemide solution versus the blank solution.

SW: It symbolizes the absorption spectrum of Furosemide solution versus the distilled water

BW: It symbolizes the absorption spectrum of the blank solution versus distilled water.

The optimal conditions for the determination of furosemide are summarized in the Table (11).

Table 11: Summary of optimum condition

Experimental Condition	
λ_{max}	465 nm
Amount ml of 1×10^{-2} M potassium permanganate	1ml
Amount ml of 2×10^{-2} M 1-Naphthylamine-4-sulfonic acid	2ml
Potassium hydroxide of 0.1M	1ml
Temperature	25 C ^o

3.13. Calibration Curve:

Increasing volumes (0.3-2.3 ml) of Furosemide solution at a concentration of $250 \mu\text{g} \cdot \text{ml}^{-1}$ were added to volumetric flask 25 ml capacity containing 2 ml

Table 12: The accuracy and compatibility

Diusemide 40 mg/ Tablets	Diusemid 40mg/ Tablets Measured	Recovery, R %	Average of Recovery, R %	RSD %
3	3.01	100.333	99.948	0.641
9	8.9	.98.888		0.580
16	16.1	100.625		0.812

3.15. Detection Limit (LOD) and quantitation limit (LOQ):

Detection limit and quantitation limit were studied and found that the LOD ($3s.d/\text{slope}$) and the LOQ ($10s.d/\text{slope}$) were 0.971 and $3.237 \mu\text{g} \cdot \text{ml}^{-1}$, respectively.

3.16. The nature of colored product: Two continuous change approaches (Job's method) and (molar ratio method) were used to determine the nature of the colored product formed and the source of the drug's connection with the reagent. In both methods, the concentration of the furosemide solution and the reagent solution 1-Naphthylamine-4-sulfonic

acid is the same concentration 2×10^{-2} M, 1 ml of potassium permanganate solution of 1×10^{-2} M, and 1 ml of potassium hydroxide with a concentration of 0.1M. Then the absorbance of all solutions was measured against the blank solution at a wavelength of 465 nm. Results indicated in Figure (5) that follows law Beer's within the limits of concentration from ($3 - 23 \mu\text{g} \cdot \text{ml}^{-1}$) of furosemide solution, and the molar absorption coefficient was calculated its value was $1.065 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ and Sandell's sensitivity was calculated and found to be equal to $0.0310 \mu\text{g} \cdot \text{cm}^{-2}$, and the correlation coefficient is 0.9994.

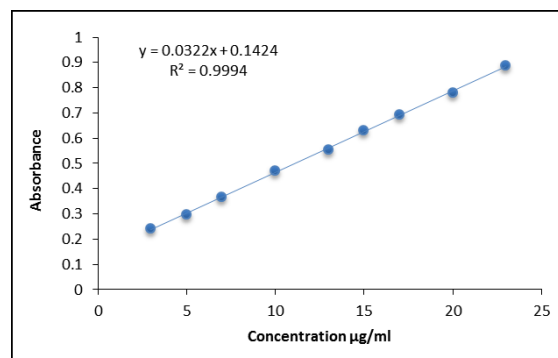


Fig. 5: Calibration curve for the determination of furosemide when reacted with 1-Naphthylamine-4-sulfonic acid in the presence of oxidizing agent potassium permanganate

3.14. Method validation:

Three different concentrations of furosemide with a concentration of $250 \mu\text{g} \cdot \text{ml}^{-1}$ were used to verify the accuracy and precision of the method represented by Relative Error RE%, Recovery percentage (R %), and Relative standard Deviation RSD%. By taking an average of six readings for each of them, the recovery rate was 99.999% and the relative standard deviation (0.641-0.812%), and results in the Table (12) are shown, meaning that the method is of high accuracy and has good agreement.

acid is the same concentration 2×10^{-2} M. In the Job method, a series of volumetric bottles with a capacity of 25 ml was taken. Different volumes of the drug solution were placed, ranging from (1-9) ml to bottles containing decreasing volumes of the reagent (9-1 ml) and and diluted with distilled water to the limit of the mark, and measurement of the absorption of these solutions at 465 nm in comparison to their blank solutions. Figure (6) shows that the correlation ratio between the drug and the reagent is 1:1.

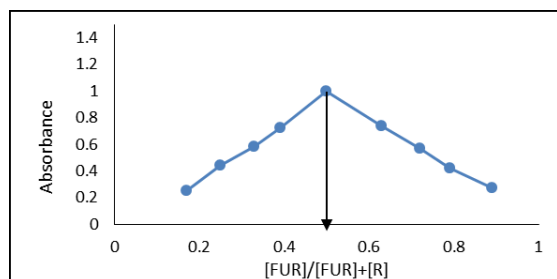


Fig. 6: Shows method of continuous changes (Job method)

to ensure that the reaction ratio between the furosemide and the reagent is 1:1, the molar ratio method was used where 1 ml of the drug solution was placed in a series of 25 ml volumetric bottles, different volumes of the reagent (0.2-2.9 ml) were added to it, and the rest was supplemented with the addition of solutions in the optimal volumes, then completed with distilled water, diluted to the mark. The absorption of these solutions was measured at wavelength 465 nanometers against the blank solution for each of them. With the method of continual changes, it was discovered that the molar ratio agrees with the blank solution. As shown in figure (7).

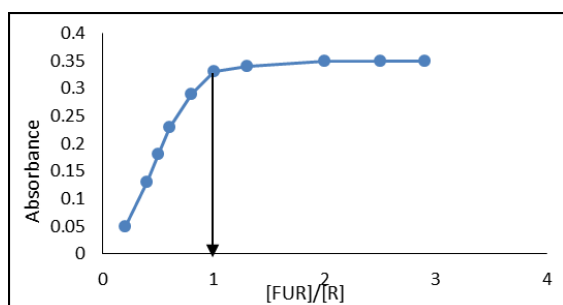
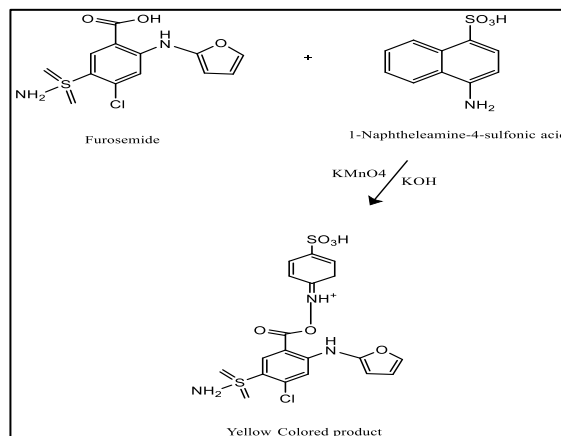


Fig. 7: show the molar ratio method between the drug and reagent

The results obtained from the oxidative coupling reaction of furosemide were based with 1-Naphthylamine-4-sulfonic acid in the presence of potassium hydroxide in an alkaline medium to form a yellow colored product, which gave the maximum absorption at a wavelength of 465 nm versus the blank solution, where the chemical equation is as follows.



4. Applications

The method can be used to test the following pharmaceutical formulations (Tablets), each of which contains 4mg of furosemide.

Three different concentrations (5, 10, 15 $\mu\text{g}/\text{ml}$) of the preparations solution (Tablets) were taken, and then the absorptions was measured at a wavelength of 465 nm versus the blank solution by the same steps followed when preparing the calibration curve, and the average of five was calculated Measurements for each concentration, and results in the Table (13) was indicated.

Table 13: The direct method

Diosemid 40mg (Tablet)	Diosemid 40mg (Tablets) easured	Relative Error, RE%	Recovery %	Average of Recovery %	RSD %
5	4.97	-0.6	99.400	98.18	0.552
10	9.80	-2	98.000		0.475
15	14.57	-2.9	97.130		0.361

Table (13) shows that the proposed method was successful in recognizing the furosemide-containing pharmaceutical product. The average recovery value was 98.18 percent in this case.

5. Conclusions

Many reagents were used as oxidative coupling for furosemide, as well as many methods were used to determine this drug in its free form and in its pharmaceutical preparation But in this proposed

method was used a suitable coupling reagent (1-Naphthylamine-4-sulfonic acid) in the presence of an oxidizing agent and in an alkaline medium for the purpose of using this method as a routine method for the determination of this drug. Where this method gave a linear relationship with a recovery rate of 99.948%, so it turns out that this method is a simple, fast and highly sensitive.

6. References

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التقدير الطيفي للفوروسيميد في المستحضرات الصيدلانية بواسطة تفاعل الإقتران التأكسدي

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الملخص

يتضمن البحث تطوير طريقة بسيطة وسريعة وحساسة لتقدير الفوروسيميد (FUR) في المستحضرات الصيدلانية. تعتمد الطريقة على تفاعل الإقتران التأكسدي بين فوروسيميد والكاشف 1-نفتيل أمين-4-حامض سلفونيك بوجود برمنغنات البوتاسيوم كعامل مؤكسد لتكوين ناتج أصفر اللون قابلة للذوبان في الماء لها أقصى امتصاص عند 465 نانومتر. يخضع لقانون بير في النطاق التركيز من 3 إلى 23 مايكروغرام.مل⁻¹ بامتصاص مولاري 1.065×10^4 لتر.مول⁻¹.سم⁻¹، ودلالة ساندل 0.0310 مايكروغرام.سم⁻². على التوالي، معامل التقدير 0.9994 بمعدل الإسترجاع 99.948%. كان حد الكشف وحد الكمي 0.971 مايكروغرام.مل⁻¹ و 3.237 مايكروغرام.مل⁻¹. الإنحراف القياسي النسبي من 0.641 إلى 0.812%. تم تطبيق الطريقة المقترحة بنجاح لتقدير الفوروسيميد في المستحضرات الصيدلانية (أقراص).