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Spectrophotometric Method for Determination of Nifedipine in Pure Form and its Pharmaceutical Preparation

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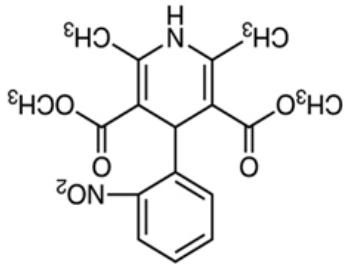
FOR nifedipine (NIF) determination in both its pure Form and in pharmaceutical dosage forms, a straightforward, quick, exact, and sensitive analytical method has been devised. To complete the reaction and obtain a colorful product bound to the amount of NIF, this approach uses a proton transfer between the NIF and 3-5 dinitro salicylic acid (DNSA) reagents in a basic medium while standing. This product's highest absorption wavelength is 359 nm. Over a concentration range of 1.5-12.5 ppm, a linear calibration curve with a correlation coefficient of 0.9991 was observed. The molar absorptivity was 4.522×10^4 $L.mol^{-1}.cm^{-1,}$ and Sandell's sensitivity index was equal to 0.010 $\mu g.cm^{-2}$. The limit of detection (LOD) and quantification (LOQ) values are 0.359 and 1.121 ppm, respectively. The suggested approach method was applied to estimate the NIF in the available pharmaceutical preparations (tablets). The results confirmed that the Method is successful by studying the recovery using the standard addition method.

Keywords: Nifedipine, Proton transfer, Determination, Spectrophotometry, 3-5 dinitro salicylic acid.

Introduction

Nifedipine (NIF) is a yellow crystalline substance, chemically called (3,5-dimethy l2, 6-dimethyl-4-(2-nitrophenyl)-1, 4-dihydropyridine-3,5-dicarboxylate), it is a could be a part of 1,4-dihydropyridine (1,4-DHP) subordinates,[1].

NIF is a slightly water-soluble compound, slightly soluble in acetonitrile, and soluble in isopropyl alcohol [2]. NIF belongs to a group of medicines primarily used to treat high blood pressure [3]. NIF could be a dihydropyridine calcium channel-blocking operator. It restrains the



Scheme. 1: NIF chemical composition of ($C_{17}H_{18}N_2O_6$)
M.Wt. = 346.335 g/mol

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transmembrane deluge of extracellular calcium particles into myocardial and vascular smooth muscle cells, causing dilatation of most coronary and systemic courses and diminishing myocardial contractility [4].

The sedate has been decided by various estimation procedures, such as high-performance liquid chromatography [5,6] and gas chromatographic methods [7,8]. Spectrofluorimetry [9,10]. Electroanalytical methods [11,12] and Voltammetric methods [13,14].

This is to make a basic, quick, particular, and touchy spectrophotometric strategy for deciding NIF's immaculate frame and pharmaceutical arrangements. Based on the proton exchange between NIF and 3-5 dinitro corrosive salicylic reagent in basic medium. This procedure can be utilized for schedule assurance of this medication.

Experimental

Apparatus

A double-beam UV-visible spectrophotometer (ShimadzuUV-Vis1800kouto-Japan)was utilized for all absorbance estimations utilizing 1.0-cm quartz cells.

Chemical reagents

All the chemical compounds utilized within the tests and tests were of a tall standard; it was not essential to channel them.

Prepare NIF stock solution 500 ppm: I used a volumetric flask to dissolve 0.05 g of pure NIF in 10 ml of distilled water while vigorously agitating and heating the mixture and dilute to 100 ml with the same solvent. A standard working solution (100 ppm) (2.887×10-4 mol.L-1) was created by properly diluting the stock solution.

3,5- dinitrosalicylic acid(DNSA) solution 100 µg. **ml^{-1}** is made by dissolving 0.01 g of DNSA powder in refined water and weakening it to 100 ml within the same dissolvable with a calibration vial.

Sodium carbonate 0.01 mol.L-1: This solution was already prepared.

Dosage Forms Procedure

NIF tablets (EPILAT retard) solution 100 ppm: This solution was made by weighing six tablets (20 mg NIF/tablet) and smashed well so that their combined weight reached 0.2131 g and then 0.0177 g weight of this powder, which is equivalent to 0.01 g of clean NIF. , after which it is broken into 10ml of refined water, mixed well, and heated gently until the material is completely disintegrated; at that point, sifted with channel paper, exchange the filter to a volumetric 100ml volume, complete the volume to check the refined water.

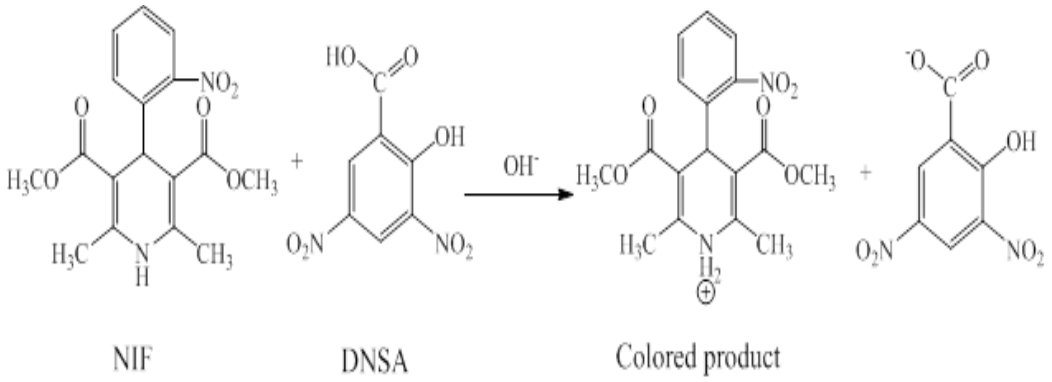
Initial procedure

1.0 ml of 100 µg.ml^{-1} NIF added to a 10 ml volumetric flask, then 1.0 ml of 100 µg.ml^{-1} DNSA solutions, and 0.5 ml of 0.01 M Na_2CO_3. Over a brief period of shaking, we noticed that the product gets the peak absorption at a wavelength of 359 nm and remains stable at room temperature.

Results and Discussion

Fundamentals of chemical reactions

The main reaction in the proposed Method includes one step that can be explained by the transfer of a proton from the reagent (DNSA) to NIF in a basic medium and result in a spectrally traceable solution at 359 nm and proportional to the NIF concentration as shown in **Scheme 2:**



Scheme 2

Optimum conditions for the reaction

The following experiments were carried out in 10 ml volumetric flasks with 25 µg of NIF, measuring absorption for the colored product at 359 nm.

Effect of 3-5 dinitrosalicylic acid reagent amount:

The impacts of DNSA amount were considered by changing the included volume and keeping other variables steady. It was found that 1.0 ml of 100 µg.ml-1 of the DNSA reagent gave the most noteworthy assimilation. Subsequently, it was utilized in consequent tests based on the discoveries in (Fig. 1).

Effect of basic medium:

The effect of number of basic solutions at 0.01 M for each one has been studied on the absorption of the product was investigated, and it was discovered that Na_2CO_3 was the best acid with the highest absorption value, as illustrated in (Table 1). Furthermore, the optimal amount of Na_2CO_3 was examined, and 1.0 ml of 0.01 M Na_2CO_3 obtained the best absorbance (Fig. 2):

Surfactant Effect

Utilizing distinctive sorts of surfactants and diverse sums of each sort at 1% concentration, it was found that these substances don't positively impact the absorption of the colored product. Still, there's a negative impact when adding these solutions, as appeared in (Table 2).

The Influence of Addition Sequence

The order of adding the reaction components was tested to see if it affected the color intensity of the product by applying several addition sequences. The results of this study are shown in Table 3.

Depending on the comes about of Table 3, the expansion arrangement number 2 was received, which is reagent + basic + drug and beneath the same conditions. The other orders of the groupings were of moo absorbance.

Effect of time and temperature:

The Method was carried out at different temperatures in arrange to decide the impact of temperature on the absorbance as well as to study the stability of the colored product, and it was found that the assimilation was steady for at slightest an hour at room temperature (25°C ± 2), as appeared in Fig. 3:

Final absorption spectrum

In a 10 ml volumetric flask at the optimum conditions, 1 ml of 100 ppm of the DNSA reagent solution was added, and then added 1 ml of a 25 ppm NIF solution, followed by the addition of 1.0 ml of 0.01 M Na_2CO_3 solution. After completing the volume to the mark with distilled water, we stand for a few minutes. The absorbance spectra scan of the resulting colored solution was measured against the blank at 359 nm. Figure 4 shows the final absorption spectrum of that product.

Calibration curve:

In several 10 ml standard battels at ideal experimental conditions, 1.0 ml of 100 µg.ml^{-1} DNSA reagent was added to each one, followed by 1.0 ml of 0.01 M Na_2CO_3 and increasing volumes of 25 µg.ml^{-1} NIF solution to cover the range

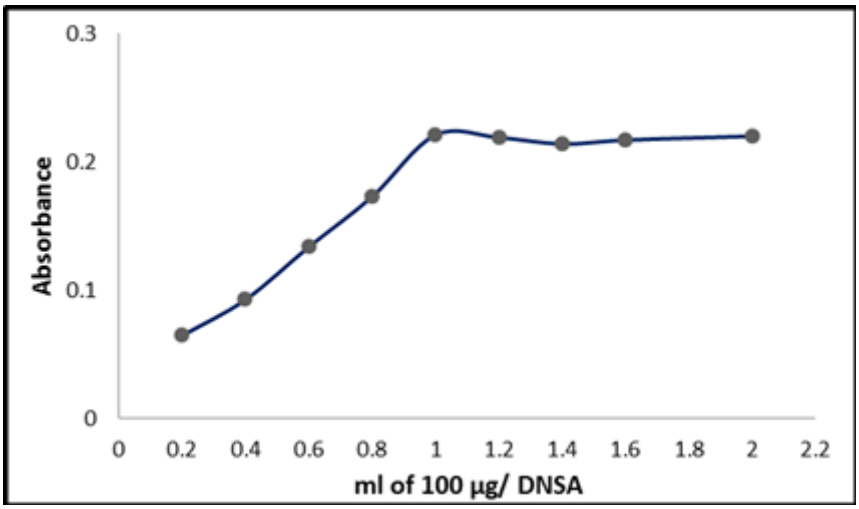


Fig.1. Oxidant agent quantity

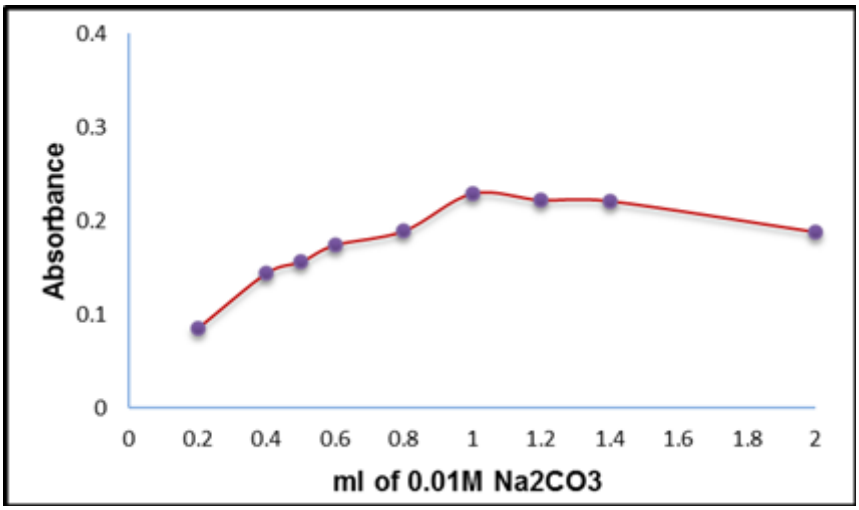


Fig. 2. Effect of H_2SO_4 amount

TABLE 2. Effect of surfactants

Surfactant	Absorbance/ml of Surfactant				
0.1 M	**0.0**	**0.5**	**1**	**2**	**3**
Triton X 100	**0.227**	0.212	0.205	0.197	0.190
SDS		0.218	0.209	0.181	0.169
CTAB		0.211	0.217	0.202	0.206
CPC		0.210	0.211	0.209	0.200

TABLE 3. Effect of Sequence of additions

NO.	Sequence of additions	Absorbance
1	NIF + DNSA+ Na_2CO_3	0.223
2	**DNSA + Na_2CO_3 + NIF**	**0.231**
3	NIF + Na_2CO_3+ DNSA	0.211

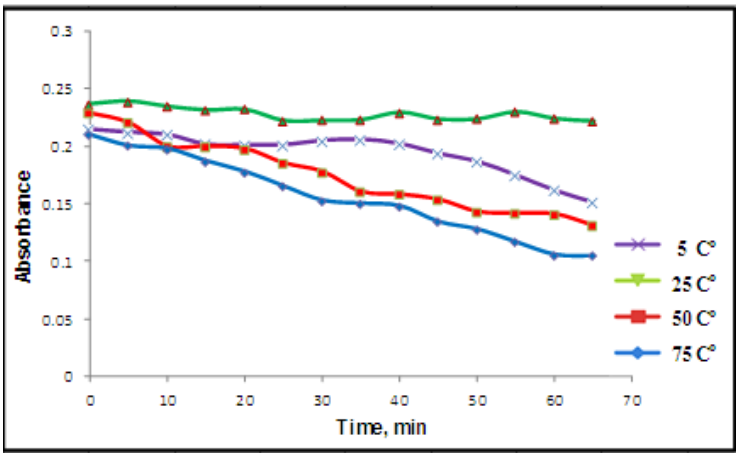


Fig. 4. Spectrum of absorption of 25 µg NIF VS (A) D.W, (B) blank, (C) blank VS D.W.

of 1.0-25 µg.ml^{-1}. After completing the volume of all flasks to the mark by distilled water. The absorbance was taken against the blank at 359 nm for all solutions. Fig. 5 shows the standard curve that draws from applying the absorbance values. The range of concentrations following Beer's law was 1.0-15.0 ppm with a molar absorption of 3.574×10^4 L.mol^{-1}.cm^{-1} and Sandell's index of 0.0096 µg.cm^{-2}.

Accuracy and precision:

To identify the accuracy and precision of the proposed method, NIF is determined using three concentrations of the working solution (25 µg.ml^{-1}), and the process is repeated several times for each concentration. The study's findings are presented in Table 4, which demonstrates the reliability and accuracy of the suggested Method.

Application of the procedure:

The proposed method was applied to one of the available drugs (tablets), where the amount of L in it was successfully estimated. The results of the application listed in (Table 5) gave that the Method is of good accuracy and acceptable for application to medical preparations.

Selectivity of the suggested Method:

Using two different quantities of drug solution (25 µg.ml^{-1}), the standard addition method [15], was applied. Then the standard curves were plotted, and the recovery was calculated for each concentration, as shown in (Fig. 6) and (Table 6). The results proved that the proposed method has an acceptable selectivity.

Conclusion

Based on the proton transfer between the NIF molecule and the DNSA reagent molecule, producing a colored solution proportional to the concentration of NIF, as its absorption is monitored at the wavelength of 359 nm. This drug has been appreciated in pharmaceutical preparations, and the proposed method has acceptable results, accuracy, selectivity, and good sensitivity.

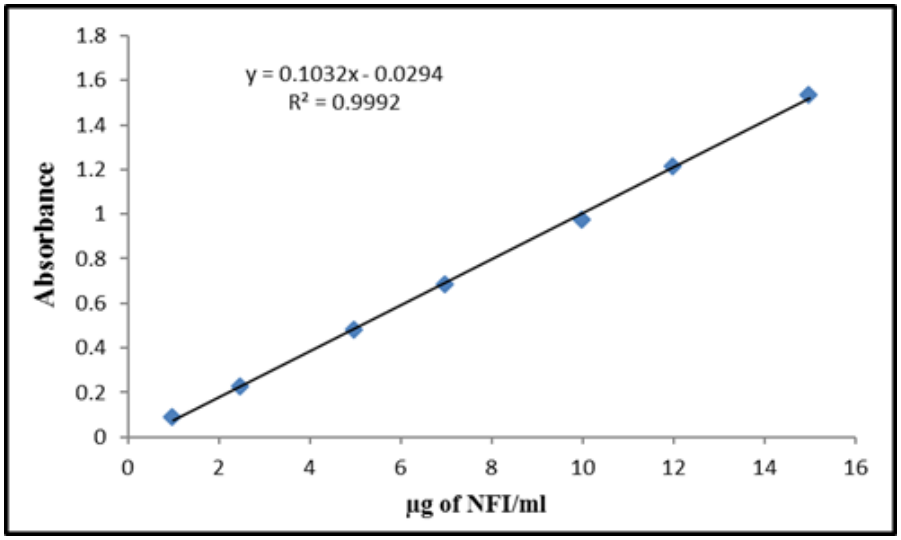


Fig. 5. Calibration Curve

TABLE 4. Accuracy and precision

Present amount of NIF µg/ml	Found amount of NIF µg/ ml	Recovery %	Relative error, %*	Relative standard deviation, %*
5	4.84	96.8	-3.2	±2.04
8	7.86	98.25	-1.75	±1.37
12	11.49	95.75	-4.25	±0.83

*Average of five determinations

TABLE 5. Results of the application

Pharmaceutical drug	The present amount of NIF µg/ml	Found the amount of NIF µg/ml	Recovery %	Relative error, %*	Relative standard deviation, %*
NIF 50 mg/tablet (Egypt)	4	3.90	97.5	-2.5	±0.833
	11	10.88	98.91	-1.09	±0.906

* The mean of three determinations

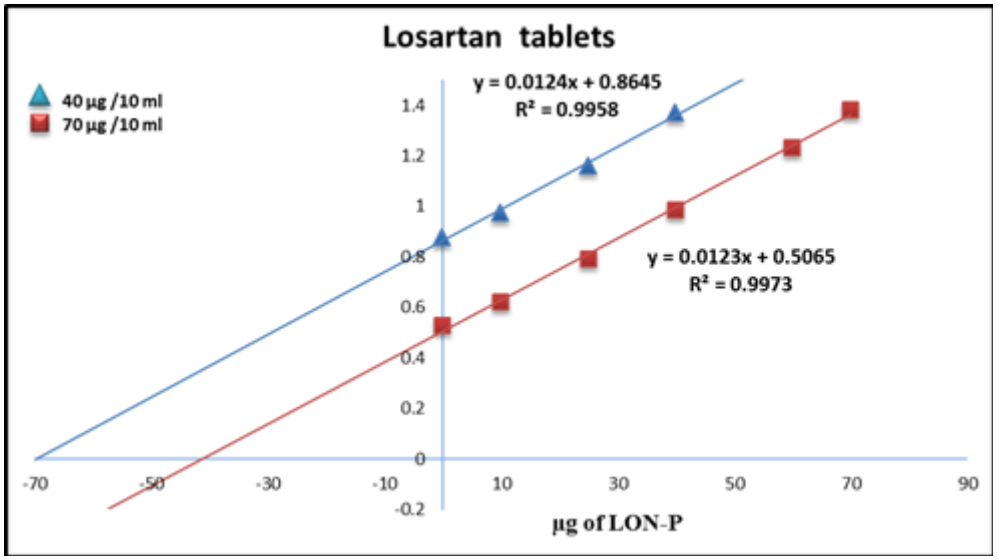


Fig. 6. Standard addition curves for estimation of NIF in pharmaceutical drug

TABLE 6. Results of the standard addition method

Pharmaceutical preparation	Amount of NIF presence µg/10 ml	Amount of NIF measured µg/10 ml	Recovery %
NIF 100 mg/tablet (Egypt)	4	4.04	101.12
	7	7.10	101.47

Conflicts of interest

There is no conflict of interest.

Formatting of funding sources

There is no funding entity.

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الطريقة الطيفية لتقدير نيفيديبين في شكل نقي ومستحضره الصيدلاني

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لتحديد نيفيديبين (NIF) في كل من شكله النقي وفي أشكال الجرعات الصيدلانية ، تم ابتكار طريقة تحليلية مباشرة وسريعة ودقيقة وحساسة. لإكمال التفاعل والحصول على منتج ملون مرتبط بكمية NIF ، يستخدم هذا النهج نقل البروتون بين NIF وكواشف حمض الساليسيليك ٣-٥ DNSA) dinitro) في وسط أساسي أثناء الوقوف. أعلى طول موجي امتصاص لهذا المنتج هو 359 نانومتر. على مدى تركيز 1.5-12.5 جزء في المليون ، لوحظ منحنى معايرة خطية بمعامل ارتباط 0.9991. كانت الامتصاصية المولية 4.522 × 104 L.mol-1.cm-1 ، وكان مؤشر حساسية ساندل يساوي ٠,٠١٠ µg.cm-٢. قيم حد الكشف (LOD) والقياس الكمي (LOQ) هي 0.359 و 1.121 جزء في المليون ، على التوالي. تم تطبيق طريقة النهج المقترحة لتقدير NIF في المستحضرات الصيدلانية المتاحة (أقراص). أكدت النتائج أن الطريقة ناجحة من خلال دراسة الاسترداد باستخدام طريقة الإضافة القياسية.

الكلمات المفتاحية: نيفيديبين ، نقل البروتون عزم ، قياس الطيف الضوئي ، 5-3 دينيترو حمض الساليسيليك.