Spectrophotometric Determination of Methyldopa in Pure and Pharmaceutical Preparations by the Oxidative Coupling Reaction with 1, 5-Diaminonaphthalene in the Presence of Ammonium Ceric (IV) Nitrate

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Abstract

Oxidative coupling reactions are one of the significant methods used in the estimation of certain drugs in pharmaceutical formulations. This study describes the development of a fast and sensitive spectroscopic method considering the determination of trace amounts of methyldopa in an acidic aqueous solution. The adopted method centered on oxidative coupling due to the drug Methyldopa with the reagent 1, 5-diaminonaphthalene in the presence of ceric (IV) ammonium nitrate as oxidizing agent to form a stable blue-colored product, giving the highest absorption at 730 nm. The product was appeared to agree Beer's law within the limits of (5.4 - 39.6) μg /ml, and a value of molar absorptivity equal to $4.79471\times10^4 L/mol.cm.Sandell'sindex, 0.0044\mu$g/cm^2, Relative standard deviation was in the range (0.5942 - 0.9687)%, whereas recovery average and determination factor were 99.908% and 0.9998. The proposed method has been successfully applied in the determination of Methyldopa in its pharmaceutical preparations.

1. Introduction:

Methyldopa is colorless or almost colorless crystals or white to yellowish-white fine powder; almost tasteless [1]. In the sesquihydrate form the pH value (saturated aqueous solution) is about 5.0. It is soluble in isopropanol, ethanol, and water [2].

Methyldopa is a catechol derivative (catecholamine) that is commonly used to treat hypertension. It is a centrally acting a-2-adrenoceptor agonist that decreases blood pressure and reduces sympathetic tone [3]. Methyldopa's activity varies between that of more potent antihypertensives like guanethidine and that of milder antihypertensives like reserpine.

Dihydroxyphenyl is a structural analogue of methyldopa, only the presence of a methyl group separates alanine (dopa) from other amino acids on the a-carbon side of the chain [4]. Common side effects include sleepiness. More severe side effects include red blood cell breakdown, liver problems Methyldopa was discovered in 1960[5]. It is on the World Health Organization's List of Essential Medicines [3]. Figure 1 shows the structural formula of methyldopa. Diverse analytical methods and procedures have been published relating the esstimation of methyldopa in bulk, pharmaceutical form or biological fluids. These methods include the Voltammetric [6], spectrophotometry [7], chromatography [8], flowinjection chemiluminescence [9], electrochemical oxidation, kinetic methods [10], and Electrochemical determination [11], 1,5-Diaminonaphthalene is an organic compound in the form of colorless crystals or a white powder [12] that fuses at 190 °C. It does not have a boiling point, but it sublimates [12] and the major use of this compound is in the manufacture of 1,5-

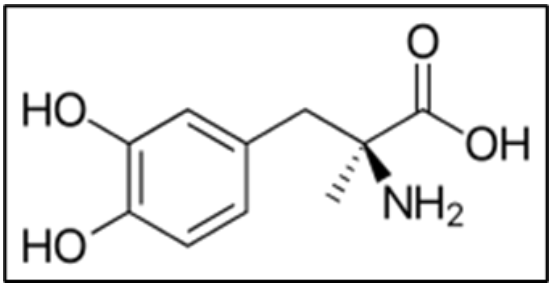

Figure 1. The structural formula of methyldopa. $(2S)-2-amino-3-(34-dihydroxyphenyl)-2-methylpropanoicacid$

naphthalene diisocyanate in the production of polyurethane elastomers. It is also used to the synthesis of dyes and pharmaceuticals [13].

2. Experimental:

2.1 Instruments:

UV-Visible Spectrophotometer –PG +92 were used to record absorbance measurements, water bath, sensitive Balance, pH meter.

Chemicals: The chemicals used on this study were of analytical grades and shown in Table 1.

Table 1. Used Chemicals.

Chemicals	M. wt (g/mol)	Chemical Formula	Assay %
Methyldopa	211.217	$C_{10}H_{13}NO_4$	Pure
Ceric(IV) ammonium nitrate	548.26	(NH4)2Ce(NO3)6	98
1,5-Diaminonaphthalene	158.204	$C_{10}H_6(NH_2)2$	98
Hydrochloric acid	36.5	HCl	97

2.2 Reagents and Solutions:

The solution of Methyldopa (1000) μg/ ml: This solution was prepared via dissolving 0.1000 g of methyldopa in a certain amount of hot distilled water and the volume was made up to the point of the sign in a 100 ml volumetric flask using the same solvent. Dilution method was employed to prepare lower concentrations of this solution. Then the solution was diluted to a of 300 μg/ml by taking 30 ml of Methyldopa (1000) μ g/ml and 70 ml of distilled water in a 100 ml volumetric flask . HCl solution (0.5 M) was prepared on diluting 4.24 ml of concentrated HCl (11.8 N) in 100 ml volumetric flask in size by distilled water.

The oxidizing agent ceric (IV) ammonium nitrate$(1\times 10^{-2}M)$: This solution was prepared by dissolving 0.548 g of ceric (IV) ammonium nitrate in a definite volume of distilled water and transferred to 100 ml volumetric flask to be completed with the same solvent to the mark.

1, 5- Di-aminonaphthalene $(1\times 10^{-2}M)$ solution: The present solution was prepared by dissolving 0.158 g of 1,5- diaminonaphthalene in 25 ml of hot distilled water and diluting it in volumetric flask to 100 ml with the same solvent.

2.3 Pharmaceutical Preparations of Methyldopa:

pharmaceutical preparation containing methyldopa, found as Aldomet tablet 250 mg supplied by Samarra Pharmaceuticals Corporation (S D I-IRAQ).

The tablets solution was prepared by grinding 10 tablets (4.38)g; 0.0526 g of this quantity was taken and dissolved in a little quantity of hot water in a 100 ml volumetric flask. The resulting solution was filtered and the residue remained washed successively with hot distilled water. The volume of the filtrate was completed to the limit of the mark using distilled water to acquire a solution with a concentration of 1000μg/ml. 30 ml of the prepared solution was poured in 100 ml volumetric flask and the volume supplemented to the limit by distilled water has a solution of 300 μg/ml.

2.4 Preliminary Study:

On the addition of 1.5 ml of the reagent (1, 5-diaminonaphthalene) with a concentration of 1×10^{-2} M to 1 ml of 1×10^{-2}M of the oxidizing agent (ceric(IV) ammonium nitrate) and 2 ml of 300 μg/ml solution of methyldopa a blue product was formed in the presence of 1 ml, 0.5 M HCl. The absorption values versus a blank solution were measured after dilution of the reaction mixture with distilled water in (25 ml) volumetric flask to the sign. The highest absorption value has been recorded at 730 nm, whereas no absorption was recorded at this wavelength for the blank.

3. Results and Discussion:

The optimum reaction conditions study:

Methyldopa drug was mixed with the reagents (1, 5- diaminonaphthalene) and (Ceric (IV) ammonium nitrate) in an acidic solution as an oxidizing agent throughout this work For a final volume of 25 ml. The resulting blue-colored solution has the highest absorption at 730 nm compared to the blank solution.

3.1 Effect of the Best Coupling Reagent:

1.5 ml, 1×10^{-2} M of each of the reagent solutions mentioned in the table below and 1ml, 1×10^{-2} M of the oxidizing agent solution (ceric (IV) ammonium nitrate) were mixed with 2 ml of 300 μg/ml methyldopa solution and (1 ml) of 0.5 M HCl.Table 2 exhibits the results.

Table 2 obviously reveals that highest absorbance value of the coloured product at 730 nm against the blank solution was recorded in the case of the reagent 1, 5-Diaminonaphthalene. No absorption due the blank solution was recorded at this wavelength.

3.2 Effect of the Coupling Reagent Quantity:

To study the effect of the coupling reagent quantity, varied volumes (0.4-2) ml of 1×10^{-2} M of the adopted reagent

Table 2. The effect of Coupling Reagent Type.

Reagent 1×10^{-2} M	Variable	Absorbance	λ_{max}
1,5-Diaminonaphthalene	SB	0.530	730
	BW	0.073	500
o-Aminophenol	SB	0.213	495
	BW	0.082	430
p-Aminophenol	SB	0.276	489
	BW	0.097	415
Resorcinol	SB	0.196	515
	BW	0.088	420
4- Aminobenzenesulfonic	SB	0.317	485
	BWv	0.085	439

1,5-Diaminonaphthalene was mixed with 1 ml, 1×10^{-2} M of ceric (IV) ammonium nitrate, 2ml of 300 μg/ ml methyldopa and 1 ml, 0.5 M of hydrochloric acid solution for each volume case. The volume 1.5 ml of the coupling reagent solution gave the highest absorbance; accordingly the optimal volume was used in the following trials. The obtained results are shown in Table 3.

Table 3. Effect of the Coupling Reagent Quantity.

ml of the reagent 1×10-2 M	Absorbance	
	SB	BW
0.4	0.072	0.262
0.6	0.080	0.397
0.8	0.092	0.425
1.0	0.188	0.456
1.2	0.208	0.509
1.5	0.244	0.531
1.8	0.181	0.485
2.0	0.097	0.462

SB: The Uv-visible absorption spectrum of Methyldopa solution against blank solution.
BW: The Uv-visible absorption spectrum of the blank solution versus distilled water.

3.3 Oxidizing Agent Optimization:

1 ml of several oxidizing agents, mentioned in Table 4, with a concentration of 1×10^{-2} M were added to 1.5 ml of the reagent solution (1,5-Diaminonaphthalene), 2 ml Methyldopa (300 μg/ ml) in a volumetric flask of 25 ml completed to the mark by distilled water. The absorbance for every specimen versus the blank solution was measured. Ceric (IV) ammonium nitrate, which showed the highest absorbance of the product at 730 nm, was adopted as a best oxidizing agent. The results are shown in Table 4.

Table 4. Test from best Oxidizing Agent.

Oxidizing agent 1×10^{-2} M	Absorbance		$\lambda max(nm)$
	Blank	Sample	
Ceric(IV) ammonium nitrate	0.091	0.531	730
Potassium peroxydisulfate	0.040	0.235	512
Ammonium per Sulphate	0.073	0.198	500
Ammonium ferricSulphate	0.039	0.104	410

3.4 Choosing the Acid used for Coupling Reaction:

1 ml, 1M of various acids varying in their acidity strength was employed and their influence on the value of absorption of the coloured product solution was studied. Table 5 shows the summary of the results obtained. The concentration of the used methyldopa was (300 μg/ ml , 2 ml.

Table 5. Effect of the Type of Acid used in the Coupling.

Acid solution used 1 M	HCl	H_2SO_4	HNO_3	H_3PO_4	CH_3COOH
Absorbance	0.525	0.402	0.423	0.394	0.289

3.5 Influence of the Quantity of Acid used:

Varied volumes of the utilized acid (HCl) were employed in the formation of the reaction mixture. It was found that 1 ml of the hydrochloric acid (pH value: 2.56) exhibited the maximum absorption reading (0.531), therefor this volume was adopted in the successive experiments. The outcomes are shown in Table 6.

3.6 Sequence of Addition:

It was noticed that, in some cases, the addition sequence of the added solutions had an influence on the value of the absorbance due to the resulting coloured product; hence numeral of trials was held using different succession of addition keeping the volumes and concentration of the used substance not suitable. Results obtained from Table 7 show that the addition order (III) has the highest absorbance value of the coloured product. Accordingly this order was followed in successive , Ceric (IV) ammonium nitrate (O), 1, 5-Diamino naphthalene (R), Methyldopa (D), as well as hydrochloric acid, HCl (A). experiments:

3.7 The Effect of Temperature:

Variation of the temperature of mixture solution was held at the range (5-50) c. Invariable and maximum values of absorbance of the resulting blue-colored solution were noted at

Table 6. Effect of the Quantity of Acid used in the Coupling.

ml of 1 M HCl	Absorbance BW	Absorbance SB	pH
0.2	0.108	0.291	2.67
0.4	0.109	0.405	2.64
0.6	0.098	0.471	2.61
0.8	0.103	0.524	2.58
1	0.104	0.531	2.56
1.2	0.102	0.511	2.55
1.4	0.095	0.474	2.53
1.6	0.082	0.385	2.51
1.8	0.071	0.318	2.5
2	0.103	0.276	2.48

Table 7. Effect of Order (Sequence) of Addition.

Order Number	Order of Addition	Absorbance BW	Absorbance SB
I	D+ O+ A+R	0.041	0.438
II	A +D +R+ O	0.020	0.323
* III	O + R + D +A	0.052	0.530
IV	R +A +D +O	0.037	0.392

the range of temperatures (20-30) Celsius, and accordingly, the temperature 25°C was chosen to be the optimum temperature. The results are given in the Table 8.

Table 8. Effect of Temperature.

Temperature, c	5	10	15	20	25	30	35	40	50
Absorbance	0.489	0.526	0.533	0.531	0.532	0.512	0.386	0.259	0.185

3.8 Stability of the Reaction Product

Study of the stability of product made from ($300\mu g/ml$) Methyldopa solution, was conducted on measuring Absorbance of colored product versus the blank solution at various time intervals at a temperature of 25 °C. A volume of (0.3) ml of the solution was taken and the final concentration of drug MD was (3.6) $\mu g/ml$. Results are displayed in Table 9. The results exhibited in the above table reveal that the formed product remains stable for 90 min and this period of time is practically very sufficient to accomplish various measurements.

3.9 Final Absorption Spectrum:

final absorption spectrum was recorded following establishing the optimum conditions by mixing 1 ml, 1×10^{-2} M of the oxidizing agent solution (ammonium ceric (IV) nitrate), (1.5 ml, 1×10^{-2} M) of the reagent solution (1, 5-Diamino naphthalene), 2ml of 300 μg / ml of Methyldopa solution, with 1 ml, 0.5M HCl in a volumetric flask (25 ml) and diluting the resulting mixture to the mark with distilled water. The absorbance of the blue product has been measured versus the blank solution and set to be 730 nm. The spectrum is shown in Figure 2.

Table 9. Effect of Temperature.

μg/ml of MD	Absorbance / min. Standing time								
5	10	15	20	25	30	60	90	120	
3.6	0.528	0.528	0.529	0.529	0.527	0.527	0.528	0.529	0.402

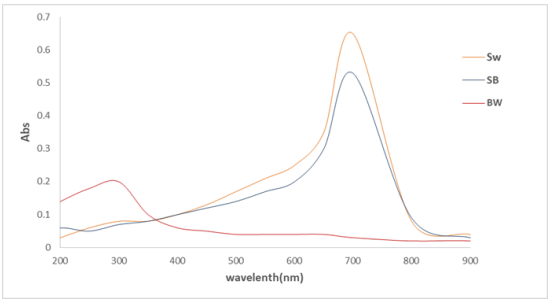

Figure 2. The Absorption Spectrum.

SW: The Uv-visible absorption spectrum of Methyldopa solution versus distilled water.
SB: The Uv-visible absorption spectrum of Methyldopa solution against blank solution.
BW: The Uv-visible absorption spectrum of the blank solution versus distilled water.

The validate working method and calibration curve: Following verification of the optimum conditions for the adopted procedure, the standard curve is depicted as follows:

Increasing aliquots (3.3- 0.45) ml of 300 μg /ml Methyldopa solution were added to a set of volumetric flasks (25 ml) including (1.5 ml) of the reagent 1, 5-Diamino naphthalene 1 x 10-2 M and (1 ml) of the oxidizing agent ammonium ceric (IV) nitrate and 1 ml of 0.5 M HCl solution. After completing the volume of all of the prepared solutions to the mark with distilled water, their absorbance versus the blank solution at 730 nm were measured. Figure 3 designates that the standard curve obeys Beer's- Lambert law in the range of concentrations (5.4 – 39.6)μg /ml of Methyldopa solution . with a determination coefficient (0.9998), molar absorptivity (4.7945×10^4) L mol^{-1} cm^{-1} and Sandell's index (0.0044) μg. cm^{-2}. These results clearly indicate that the method is highly sensitive.

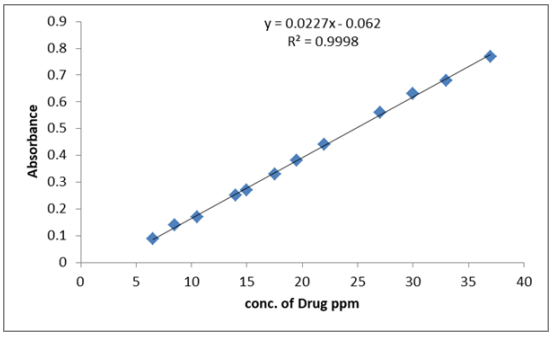

Figure 3. Calibration curve.

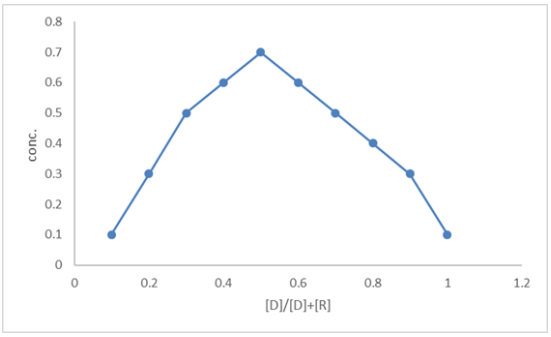

Figure 4. The Plot of Job's Method for Determination of Methyldopa.

4. Accuracy and Precision :

Accuracy being expressed by Relative Error (RE%), Relative Standard Deviation (RSD) and Recovery Percentage (Rec%) are also measures of the method's precision. RSD% was calculated, using three concentrations of methyldopa (5, 13, 32.5 μg/ml). The results obtained are exhibited in Table 10 which shows good accuracy and precision relating the adopted method.

The precision of the approach was calculated using three concentrations of methyldopa (5, 13, 32.5 g/ml) and was given as Relative Error (RE percent), Recovery Percentage (Rec percent), and Relative Standard Deviation RSD percent. Table 10 illustrates the data collected, which show good accuracy and precision in relation to the employed procedure.

Table 10. The accuracy and Precision of the Method.

Conc. of methyl dopa	Conc. of methyl dopa Measured	E%	Recovery %	Average of Recovery %	RSD %
5.8	5.9	1.724	101.724		0.5942
13	12.7	97.692	99.932	0.8724	
32.5	32.6	0.38	100.38		0.9687

*Each value of absorption exhibited is an average of five readings.

Continuous variation method (Job's) as well as molar ratio methods were applied to identify the sort of the established product as well as the ratio regarding the drug's adherence to the reagent [14]. In the both methods, the concentration for each of Methyldopa as well as the reagent solutions was held equal to 1×10^{-2} M. In (Job's) method, series of volumetric flasks (25 ml) were utilized and different volumes of the drug solution ranging from (0.1-0.9) ml placed in the flasks containing decreasing volumes of the reagent (0.9-0.1) ml. The remains of the other components were kept at the optimum amount as mentioned before. The absorption values for each solution were measured at 730 nm against blank solution. Figure 4 Shows clearly that the ratio is 1: 1. In order to ensure that the linking ratio between methyldopa and the reagent (1, 5-diaminonaphthalene) is 1:1, Molar ratio method was employed, in which 2 ml, to 1×10^{-2} M of the drug compound solution was transferred to a series of 25 ml volumetric flasks the drug compound solution was transferred to a series of 25 ml volumetric flasks containing various volumes of the reagent solution (0.2- 2) ml, 1×10^{-2} M, while the additives were kept at their optimal sizes and diluted with distilled water to the point of the mark. The absorption of the resulting solutions was measured against a blank solution at the wavelength of 730 nm. Figure 5 clearly shows that the ratio between the drug (methyldopa) and the reagent 1,5- diaminonaphthalene) is 1: 1; therefore the consistence of molar ratio method with continuous Variation method was confirmed.

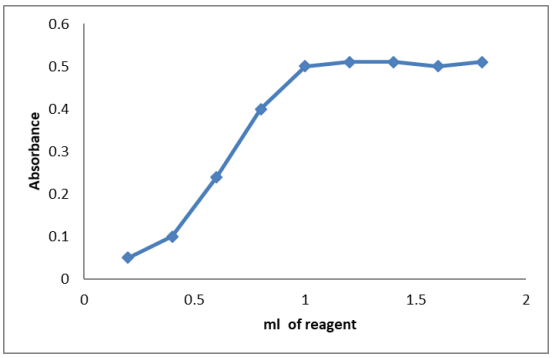

Figure 5. The Scheme of Application of Molar Ratio Method.

Table 11. The Results of Direct Method.

Conc. of Methyldopa	Conc. of Methyldopa Measured	E%	Recovery %	Average of Recovery %	RSD %
8	8.1	1.25	101.25		0.7718
15	14.89	-0.74	99.26	100.076	1.0260
25	24.93	-0.28	99.72		1.3045

Consequently, the proposed reaction steps are shown in the equations below. The reagent (1, 5-diaminonaphthalene) first oxidized by the oxidizing agent (ceric (IV) ammonium nitrate) followed by its coupling with the drug (Methyldopa) in an acidic medium to form blue- colored Complex (Scheme) ; Suggested equation for the reaction [17].

Figure 6 shows the proposed interaction mechanism for oxidative coupling for methyldopa

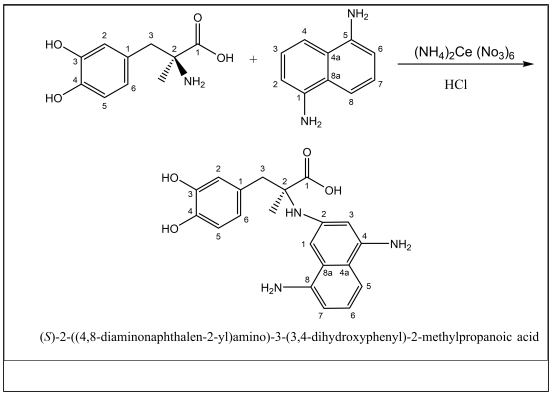

Figure 6. The Proposed Interaction Mechanism .

5. Applications:

The method can be applicable in the subsequent pharmaceutical preparation including Methyldopa-Aldomet tablet.

5.1 Direct Method:

Three various concentrations of the preparation solution (Tablets) (8, 15, and 25 μg/ ml) mentioned in the preparation paragraph were taken and treated according to the same steps used in preparing the calibration curve. Their absorbance values were measured at the wavelength 730 nm versus the blank solution. The average of (5) readings for each concentration besides the recovery and RSD% was calculated and exhibited in Table 11.

The results exhibited in Table 11 show the efficacy of the proposed method in esstimation of Methyldopa in the pharmaceutical preparation. Average of recovery % was 100.076

6. Conclusions:

A spectroscopic method was devised as an easy, simple, rapid, precise and highly sensitive Methyldopa determination established on the oxidative coupling reaction using ceric (IV) ammonium nitrate with the reagent 1, 5-diminaphthalene in an acidic medium of hydrochloric acid at a concentration of 0.5 M. The blue-colored product gave higher absorption at the wavelength of 730 nm following the Beer-Lambert law in the range (5.4-39.6) μg/ ml with a determination coefficient (0.9998). The molar absorption was(4.7947×10^4) liters mol^{-1} cm^{-1} and Sandell's index $(0.004)\mu$g. cm^{-2}. The method has been successfully applied in estimating Methyldopa in pharmaceutical preparations (tablets), and the recovery average (99.908)%.

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Data Availability Statement: All of the data supporting the findings of the presented study are available from corresponding author on request.

Declarations:

Conflict of interest: The authors declare that they have no conflict of interest.

Ethical approval: The manuscript has not been published or submitted to another journal, nor is it under review.

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التقدير الطيفي للمثيل دوبا بواسطة تفاعل الاقتران التأكسدي مع الكاشف 5,1– ثنائي امينو نفثالين بوجود العامل المؤكسد نترات السيريك الامونياكي

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الخلاصة

تتضمن الدراسة تطوير طريقة طيفية سريعة وحساسة لتقدير كميات ضئيلة من المثيل دوبا في محلول مائي حامضي، تعتمد الطريقة على الاقتران التأكسدي للعقار مع الكاشف 5,1– ثنائي امينو نفثالين بوجود العامل المؤكسد نترات السيريك الامونياكي لتكوين ناتج ازرق اللون ذائب بالماء مستقر، يعطي أعلى امتصاص عند الطول الموجي 730 نانوميتر ويخضع لقانون بير بحدود (5.4 – 39.6) مايكروغرام وبلغت الامتصاصية المولارية 4.79471×10^4 لتر مول. سم ، ودلالة ساندل 0.0044 مايكروغرام /سم ^{2} الانحراف القياسي النسبي بين %(0.5942 – 0.9687) ومعدل الاسترجاعية %99.908 ، معامل التقدير 0.9998 وطبقت هذه الطريقة المقترحة بنجاح لتقدير المثيل دوبا في مستحضراته الدوائية.

الكلمات الدالة: ميثيل دوبا؛ مطيافية الاشعة فوق البنفسجية والمرئية؛ تفاعل الإقتران التأكسدي.

التمويل: لا يوجد.

بيان توفر البيانات: جميع البيانات الداعمة لنتائج الدراسة المقدمة يمكن طلبها من المؤلف المسؤول.

اقرارات:

تضارب المصالح: يقر المؤلفون أنه ليس لديهم تضارب في المصالح.

الموافقة الأخلاقية: لم يتم نشر المخطوطة أو تقديمها لمجلة أخرى، كما أنها ليست قيد المراجعة.