

Metronidazole concentration measured spectrophotometrically using a diazotization and coupling reaction with 5-sulphosalicylic acid

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<https://doi.org/10.54153/sjpas.2024.v6i4.760>

Article Information

Received: 11/11/2023

Revised: 10/12/2023

Accepted: 12/12/2023

Published: 30/12/2024

Keywords:

Spectrophotometric determination, Diazotization reaction, 5-Sulphosalicylic acid, Metronidazole.

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Abstract

A novel, straightforward, and sensitive spectrophotometric technique for determining Metronidazole has been developed; this method relies on the reduction of metronidazole in acidic media using iron powder and then preparing diazonium ion by adding an equivalent amount of sodium nitrite to the reduced metronidazole in acidic medium and waiting for 3 min. before adding the reagent (5-sulphosalicylic acid) to produce a red color product that gives the highest absorption at the wavelength 503 nm, and the linearity of Beer's law was in the concentration range from 0.5 to 10 µg/ml. The value of the determination coefficient for the standard curve was 0.9972, indicating statistically that it has excellent linear characteristics; the molar absorptivity was calculated and found to be 1.151×10^4 L.mol⁻¹.cm⁻¹, and Sandell's sensitivity value is 0.01485 µg/cm². The method has been successfully applied to the assay of Metronidazole in pure and pharmaceutical forms, as well as tablet and oral suspension.

Introduction:

Metronidazole [1-(2-hydroxyethyl)- 2- methyl-5-nitro-imidazole] has been employed as a curative drug for at least 30 years. It has strong antiprotozoal and bactericidal action. It has the following chemical composition, as displayed in Figure 1.

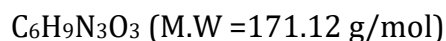
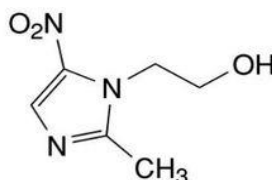


Fig. 1 Chemical composition of metronidazole [1].

In ensuring pharmaceutical quality and combating the issue of subpar pharmaceuticals, there is a pressing necessity for quick and comprehensive techniques for assaying such kinds of medications [2,3]. Different methods were reported for determining metronidazole most of which have been applied in the determination of Metronidazole in pharmaceutical formulations and as pure involving liquid chromatographic methods [4,5], gas chromatography-mass chromatography [6], gas chromatography-FID[7], thin-layer Chromatography [8,9]), HPTLC method [10,11], electrochemical methods [12,13] and voltammetric methods [14,15]. Spectrophotometric methods are still used in the determination of various drugs (16,17) to simplify the operation of the main apparatus, the spectrophotometer. The majority of the spectrophotometric techniques that have been published have drawbacks such as a narrow range for measurement, color product stability, lengthy reaction times, the use of non-aqueous systems, difficult heating or extraction, etc. Furthermore, most spectrophotometric methods reported are UV-spectrophotometric methods and are for mixed formulations (MNZ or TNZ with other medications), including prior reduction and then diazotisation and coupling or oxidation [18-23].

The innovation in the science of this method is that the reagents used in this approach are readily accessible, and their chemistry is already well-confirmed. The reaction that includes this reagent is simple, sensitive, and rapid in its range of results in comparison to alternative techniques [24].

Experimental

Instrumental

UV-1900I spectrophotometer (Japan) with 1 cm light path glass cells was utilized for all absorbance measurements. The ABS 120-4 electronic balance from KERN was employed for weighing all solid materials. Solution acidity measurements were conducted using the TRANSE BP 3001 pH meter.

Chemicals and prepared solutions

The chemicals used in this research were of high purity and did not require any purification process.

5-Sulphosalicylic acid solution (5-SuA, 0.4 %, w/v)

In order to prepare the solution, 0.4000 g of pure 5-Sulphosalicylic acid in 4 ml ethanol was used, and the volume was completed to the mark in a volumetric flask with 100 mL of distilled water.

Solutions of sodium nitrite (0.0006M)

0.0042 g of sodium nitrite was weighed and dissolved in distilled water, then passed on a 100 mL volumetric flask, and distilled water was added to bring the volume up to the required level.

Sodium hydroxide solution (1 M)

Sodium hydroxide solution (1M) concentration was prepared by diluting an ampoule of 10 mol/L (100 ml) with distilled water in a 1000 ml volumetric flask and then kept in a non-glass (plastic) container.

Solution of hydrochloric acid (1M).

In a 100 ml volumetric flask, 8.47 ml of concentrated HCl is diluted to the appropriate level using distilled water.

Reduced Metronidazole solution (100 µg/ml, 0.00058 M):

The preparation of this solution involved dissolving 0.0100 g of pure Metronidazole in 10 ml deionized water, adding 0.4 g of Fe-powder, and then adding 5 ml of concentrated HCl. The mixture was then allowed to sit at room temperature for 20 minutes before being filtered through filter paper, and the volume was filled up with distilled water to the volumetric flask's mark.

Reduced Pharmaceutical preparation solutions (100 µg/ml)

The preparation of pharmaceutical formulations involved weighing and mixing three tablets of metronidazole (100 µg/tablet) from Iraqi and French companies. Subsequently, the equivalent of 0.0128 g of powder for the Iraqi product and 0.0139 g for the French product, both corresponding to 0.0100 g of pure metronidazole, was weighed. Each sample was then reduced by adding 10 ml of deionized water, 0.4 g of Fe-powder, and 5 ml of concentrated HCl, followed by leaving the mixture to stand for 20 minutes. The solutions were then filtered using filter paper, and the volumes were adjusted to the mark of two volumetric flasks with distilled water.

Results and Discussion

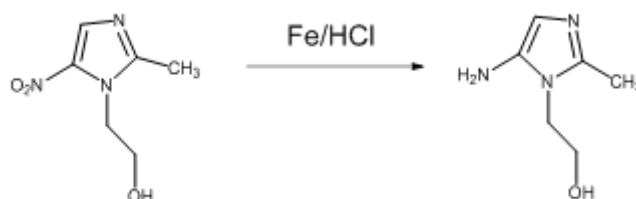
Preliminary study

This method includes the formation of diazonium ion by adding an equivalent amount of sodium nitrite in the presence of hydrochloric acid to reduce metronidazole and then waiting 3 min before adding 5-Sulphosalicylic acid in the presence of sodium hydroxide (1M). The colored product gives the highest absorption spectrum at the wavelength of 503 nm, so it was used in the subsequent measurements.

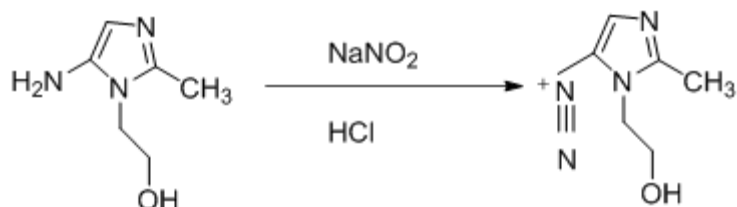
Principle of the method

The reaction included three steps:

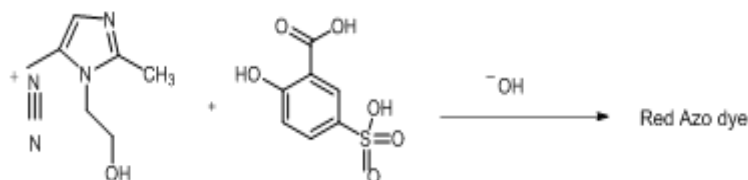
- 1- The reduction of MZ via using a powder of iron in an acidic medium



- 2- The diazotisation of R-MZ by adding equivalent amount of sodium nitrite to produce diazonium salt.



3- The coupling with 5-Sulphosalicylic acid in alkaline medium to produce coloured azo dye



All the factors affecting research have been done on the colored product's absorption.

Choice coupling agent

For the reaction with diazotized reduced metronidazole (0.5 ml) in the acidic medium, various coupling agents are used. 5-Sulphosalicylic acid produces a more sensitive reaction in an acidic media, as indicated by Table 1's results.

Table 1: Coupling agent selection.

Reagents 0.1%	Absorbance
Resorcinol	Blank darker than sample
Histidine	Uncoloured solution
2,5- dimethyl phenol	0.063
5-Sulphosalicylic acid	0.3887
4,4- dihydroxy diphenyl sulphone	Uncoloured solution

Effect of 5-Sulphosalicylic acid amount

The investigation has been done on the effects of varying 5-Sulphosalicylic (5-SuA) acid amounts on dye color intensity, as shown in Table 2.

Table 2: Effect of 5-sulphosalicylic acid amount.

5-SuA amount (ml)	A / μg MZ in ml			R^2
	2.0	5.0	10.0	
0.75	0.1115	0.3176	0.3741	0.802
1.0	0.1147	0.3880	0.4782	0.829
1.5	0.1212	0.4677	0.6655	0.9128
2.0	0.1252	0.4711	0.5938	0.8399

From the results in Table 2, it can be observed that 1.5 ml of 0.4% 5-Sulphosalicylic acid solution is the more appropriate quantity that produces the highest determination coefficient (R^2) values as well as to the highest absorbance of produced azo dye.

Setting the optimum conditions for the determination of metronidazole:

Subsequent experiments were performed using 1 mL of 100 $\mu\text{g}/\text{mL}$ of the reduced solution of pure metronidazole in a final 10 mL volume, and the absorbance of the colored product at the wavelength of 503 nm was measured against its blank solution.

Effect of acid types and their amounts

The effect of acid types and amounts used for the diazotisation of reduced metronidazole was studied by adding 0.5 ml of different types of acids HCl, H₂SO₄, HNO₃, CH₃COOH and H₃PO₄. Based on the results, which indicate that HCl has the maximum absorbance, Table 3 presents the measurements that used HCl in the following investigations.

Table 3: Effect of acid type.

Type of acid (0.5 ml, 1 M) with (10.0 µg/ml of Med)	A	pH
HCl	0.6647	1.64
H ₂ SO ₄	0.4215	1.87
HNO ₃	turbid	1.94
CH ₃ COOH	0.5858	1.85
H ₃ PO ₄	0.1129	1.94

The study's results on hydrochloric acid is displayed in Table 4.

Table 4: Effect of acid amount.

ml of hydrochloric acid	A
0.1	0.3878
0.2	0.5345
0.3	0.6636
0.5	0.6329
0.7	0.6060
0.9	0.3646

The volume of 0.3 mL produced the maximum product absorbance, as shown by the data in Table 4. Hence, it was utilized in the experiments that followed.

Effect of base types and their amounts

The effect of different types of bases at the concentration of 1 M and a volume of 0.5 ml on the absorbance of the formed product was studied by adding 1.5 mL of 5-Sulphosalicylic acid reagent and 1.0 ml of reduced solution of metronidazole with a concentration of 100 µg/mL. The obtained results are illustrated in Table 5.

Table 5: Selection of the base type

0.5 ml of base	A
NaOH	0.6658
KOH	0.6080
Na ₂ CO ₃	0.5411

Then the optimum amount of sodium hydroxide was studied as shown in Table 6.

Table 6: The optimal amount of sodium hydroxide.

ml of sodium hydroxide	A
0.1	0.3434
0.2	0.4696
0.3	0.5412
0.5	0.6659
0.7	0.5427
0.9	0.4174

Based on Table 6's results, the 0.5 mL volume gave the highest absorbance of the colored product, therefore it was used in subsequent experiments.

Effect of the sequence addition of components

In order to choose the best sequence of the reacting components, the sequences shown in Table 7 were chosen with the addition of the reaction components in the quantities mentioned previously.

Table 7: The effect of the sequence of adding reaction components.

SEQ.	order	A
I	MZ + HCl + NaNO ₂ + NaOH + 5-Sulphosalicylic acid	0.5229
II	MZ + NaNO ₂ + HCl + 5-Sulphosalicylic acid+ NaOH	0.6645
III	MZ + NaNO ₂ + 5-Sulphosalicylic acid+ HCl + NaOH	0.3212
IV	MZ + NaNO ₂ + NaOH + 5-Sulphosalicylic acid + HCl	0.6184
V	MZ + HCl+ NaOH+ NaNO ₂ + 5-Sulphosalicylic acid	0.1436

MZ : Metronidazole

The results of Table 7 show that sequence II was chosen to give maximum absorbance.

The influence of time on a colored product's stability

It was investigated how time affected the stability of colored products by taking two different concentrations 4 and 8 µg/L and proceeding according to the procedure mentioned before, the absorbance was read every 5 or 10 minutes for 60 minutes Table 8.

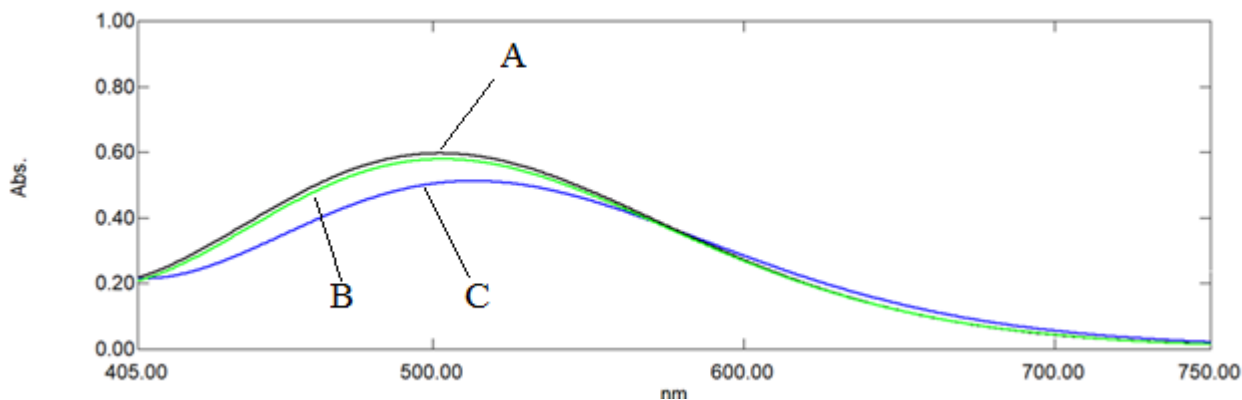
Table 8 : The influence of time on the azo dye's stability after formation.

Sample µg/ml	Absorbance / minute standing time							
	0	5	10	20	30	40	55	60
4	0.2786	0.2804	0.2816	0.2821	0.2830	0.2838	0.2845	0.2849
8	0.5392	0.5411	0.5418	0.5424	0.5432	0.5441	0.5451	0.5455

The results above indicated that the formed azo dye was stable for 60 minutes.

Effect of solvents

Various solvents were used for the dilution to find out which of them gave the highest absorption of the formed azo dye; from the results, water gave the higher absorptivity, followed by acetic acid and ethanol. The results are shown in Figure 2 and Table 9.



Black Curve(A) = Water , Green Curve (B)= Acetic Acid , Blue curve(C) = Ethanol

Fig. 2 The effect of solvents on spectrum of formed azo dye.

Table 9: Effect of solvents.

Solvents	A	λ	ϵ (l.mol ⁻¹ .cm ⁻¹)
Water	0.6652	503	1.032 x 10 ⁴
Methanol	Turbid	-----	-----
Acetone	Turbid	-----	-----
Ethanol	0.512	512	0.882 x 10 ⁴
Acetic acid	0.579	503	0.998 x 10 ⁴

The optimum conditions

The optimum conditions extracted from previous experiments shown in the Table 10

Table 10: The optimum conditions

Optimization condition	The value or selected material
Reagent used	Sulphosalicylic acid-5
Amount of Reagent	1.5 ml
Base used	Sodium hydroxide
Amount of NaOH (ml , M)	0.5 , 1
Acid used	Hydrochloric acid
Amount of HCl (ml , M)	0.3 , 1
Temperature	Room Temp.(24±2 °C)
Solvent used	Water
λ_{max}	503 nm

Final absorption spectrum

The final spectrum shown that the best wavelength is 503 nm this shows in Figure 3.

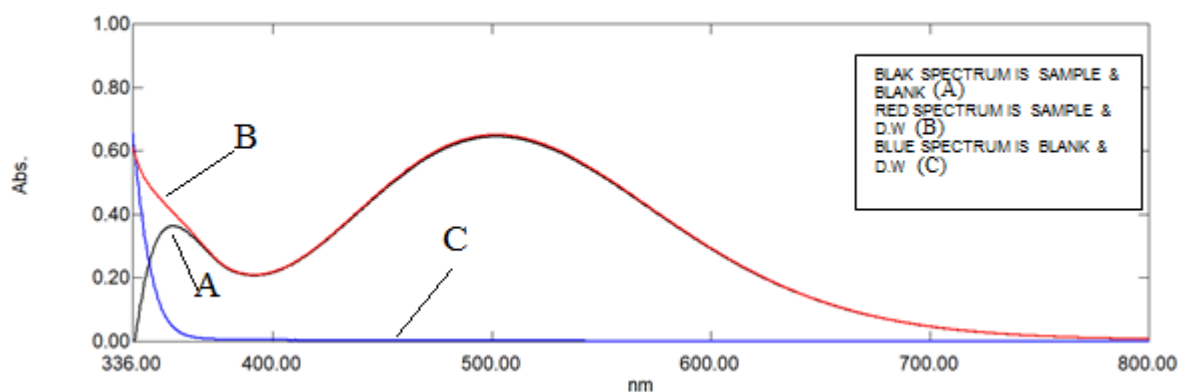


Fig. 3 The absorption spectrum of reduced metronidazole proceeds under the proposed method.

Metronidazole determination process and calibration curve

Following the best conditions for determining metronidazole were established, the standard curve was prepared by adding 0.3 ml of HCl to increasing volumes of the reduced solution of metronidazole with the same amount of NaNO₂ at the same concentration added to flasks of 10 ml, then waiting for 3 min. and adding 1.5 ml of 5-Sulphosalicylic acid reagent and finally 0.5 ml of sodium hydroxide solution (1M), then complete the volume to 10 ml with distilled water. As seen in Figure 2, the absorbance was measured at a wavelength of 503 nm. Beer's law was in the concentration range from 0.5 to 10 µg/ml, after calculations, 1.15x10⁴ l/mol.cm and 0.01455 µg/cm² were found to be the molar absorptivity and Sandell's index, respectively.

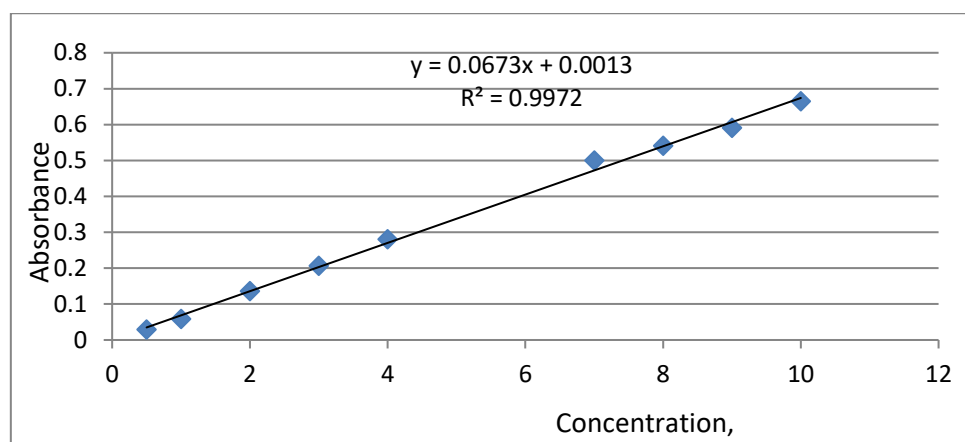


Fig. 4 Standard curve of metronidazole by using the proposed method.

Precision and accuracy

A calibration curve including two distinct concentrations of metronidazole was determined in order to verify its accuracy and precision. The method's good outcomes are demonstrated in Table 11.

Table 11: The calibration curve's accuracy and precision.

Concentration of metronidazole (µg/ml)		Recovery* %	RSD%
Present	Found		
4	3.997	99.92	0.252
8	7.995	99.93	0.203

*Average for four determinations.

The nature of the formed colored product

The two methods of continuous variation and mole ratios [25] were applied to study the molar structural ratio of the azo dye formed between diazotised reduced metronidazole(D-RMZ) and 5-SuA at a concentration of 1.833×10^{-2} mol./l for each solution the results shown in Figure 5.

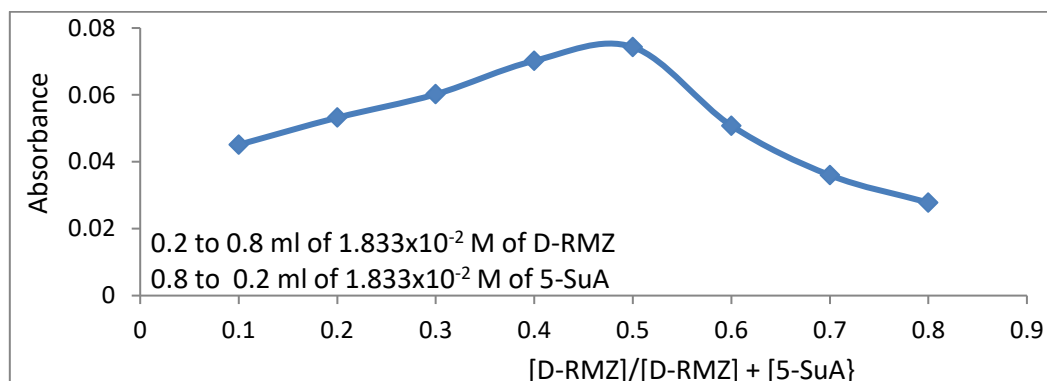


Fig. 5 The plot of the continuous variation method for azo dye.

From Figure (5), we infer that the reaction ratio is 1:1, and to verify that this ratio is correct, the mole ratio method was applied. Figure 6 shows the mole ratio plot obtained from adding increasing volumes from 0.25 to 2.0 ml of 5-SuA solution at a concentration of 1.833×10^{-2} mol./l to a constant volume of 1 ml of 1.833×10^{-3} mol./l of metronidazole solution.

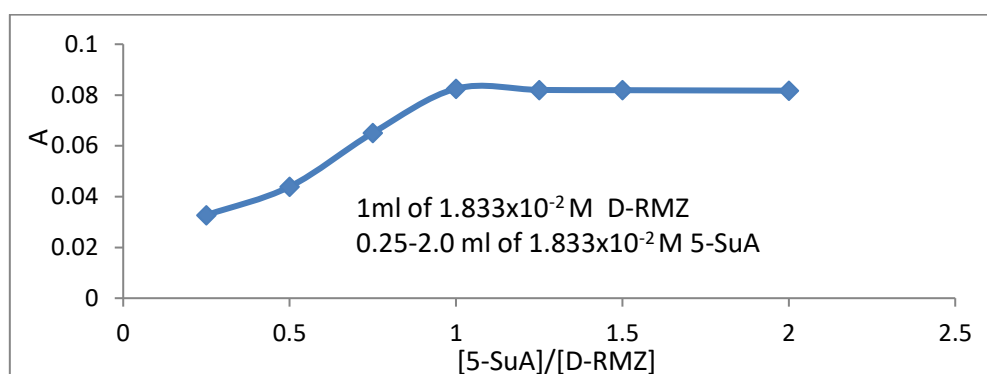


Fig. 6 The plot of the mole ratio method for the azo dye.

Based on the results of Figures 5 and 6, the proposed chemical structure for the formed azo dye was suggested as below in Figure 7 the connection to the o-position to the hydroxyl group because the p- position is occupied by another group(26).

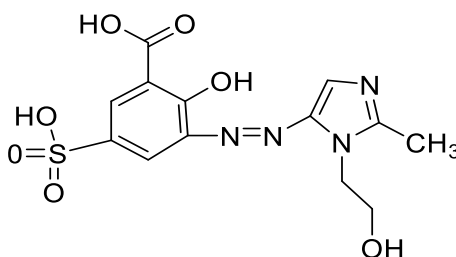


Fig. 7 The suggested chemical structure of coloured azo dye.

Analytical application

The results of using the suggested method to measure the amount of metronidazole in various pharmaceutical formulations are displayed in Table 12 below.

Table 12: Outcomes of using the suggested approach to calculate the amount of metronidazole in formulations.

Pharmaceutical preparation	Concentration of Metronidazole ($\mu\text{g/ml}$)		Recovery%	RSD%	Drug continent (mg)
	Present	Found			
Metronidazole 500mg/ Tablet/ SDI/Iraq	4	4.007	100.17	0.624	500.85
	8	7.992	99.90	0.238	499.50
Metronidazole 500mg/ Tablet/ SANOFI/France	4	4.001	100.02	0.459	500.10
	8	8.005	100.06	1.668	500.30
Nidazole 200mg/5ml/ The Arab Pharmaceutical / Jordan	4	4.009	100.22	0.315	200.44
	8	7.995	99.93	0.381	199.86

Estimation with the standard addition method

To demonstrate the interference-free nature of the suggested approach. The standard addition method was applied by adding 4 and 5 $\mu\text{g/mL}$ metronidazole for each one of the three pharmaceutical preparations to six sets of 10-mL volumetric flasks; each set contained various amounts of standard solution of metronidazole (0, 1, 2, 3, 4 and 6 $\mu\text{g/mL}$). The results obtained are shown in Figures 8-10 and Table 9.

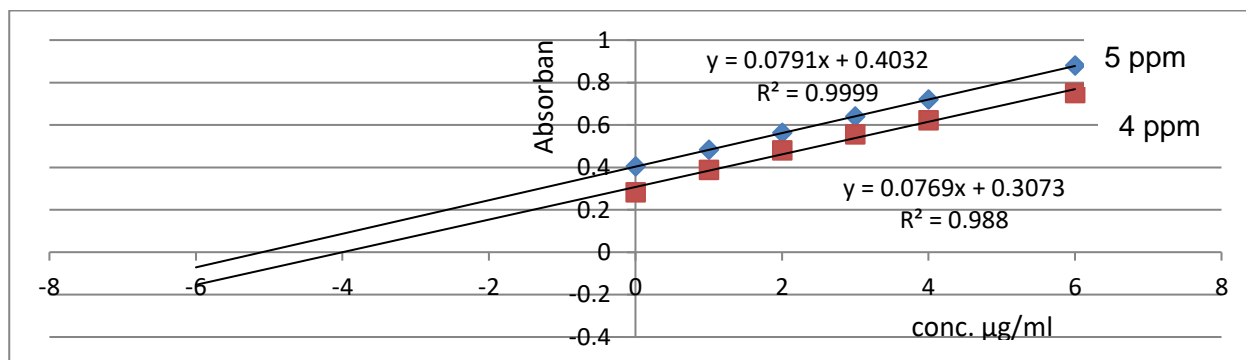


Fig. 8 The plot of the standard addition method to find how much of metronidazole in tablets (SDI - Iraq).

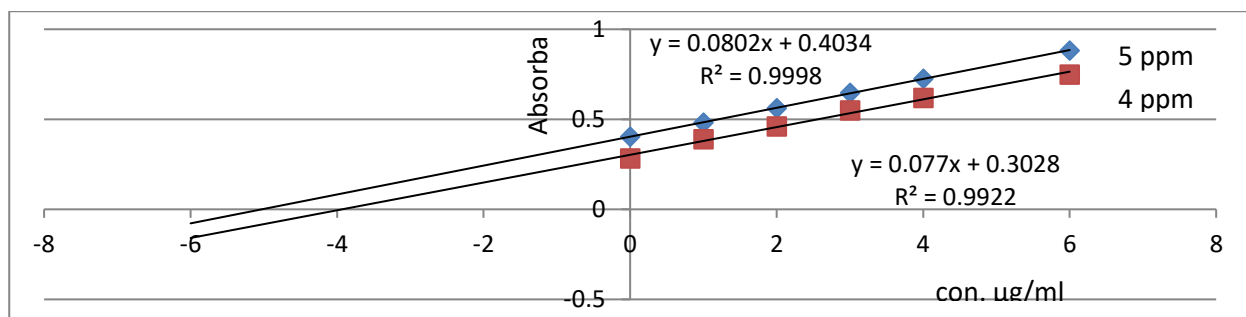


Fig. 9 The plot of the standard addition method to find how much of metronidazole in tablets (SANOFI- France)

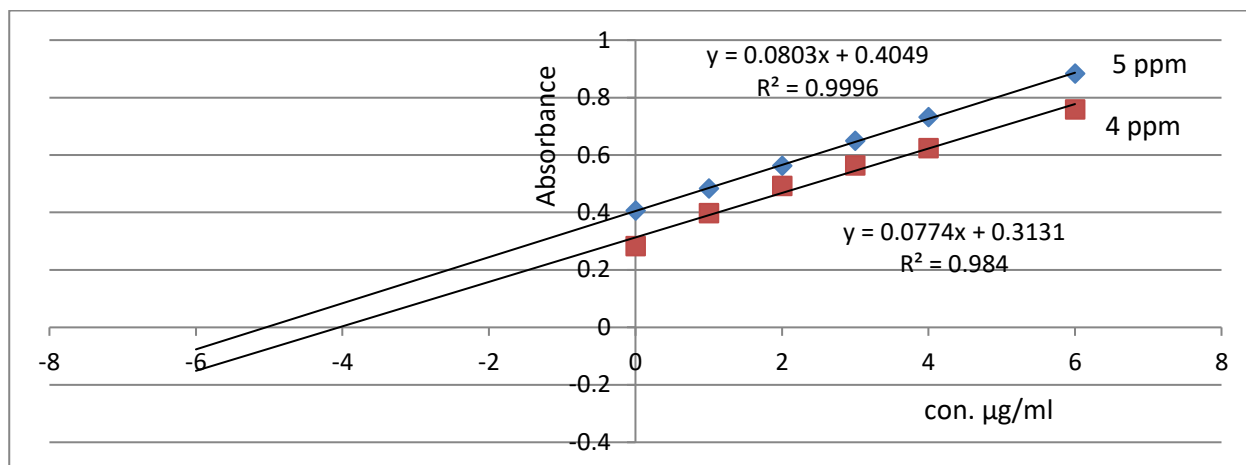


Fig. 10 The plot of the standard addition method to find how much of metronidazole in oral suspension (The Arab Pharmaceutical -Jordan)

Table 13: The result extracted from Figures. 8-10.

Pharmaceutical preparation	Certified Value (mg)	Amount Present (µg/ml)	Amount measured (µg/ml)	%Rec.	Drug content (mg)
Metronidazole/ SDI/ Iraq	500mg / Tablet	4	3.996	99.90	499.50
		5	5.097	101.94	509.70
Metronidazole/ SANOFI/ France	500mg / Tablet	4	3.932	98.30	491.50
		5	5.029	100.58	502.90
Nidazole/ The Arab Pharmaceutical/ Jordan	200mg /5ml/Oral suspension	4	4.045	101.12	202.20
		5	5.042	100.84	201.68

Conclusions

Based on the obtained results, we can confidently conclude that the proposed method excels in terms of accuracy, ease of use, cost-effectiveness, selectivity, and applicability for the spectrophotometric determination of metronidazole in both tablet and suspension forms of pharmaceutical formulations. The proposed new method was characterized by the addition of equivalent amounts of nitrite and metronidazole; thus, we did not need to add sulphamic acid or urea to destroy the excess nitrite. The outcomes achieved were within acceptable limits, affirming the method's reliability and suitability for the intended purpose

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لتقدير الطيفي للميترونيديازول عن طريق تفاعل الأزوتة والاقتران مع الكاشف حامض السلفوساليسيليك

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معلومات البحث:

تاريخ الاستلام: 2023/11/11

تاريخ التعديل: 2023/12/10

تاريخ القبول: 2023/12/12

تاريخ النشر: 2024/12/30

الكلمات المفتاحية:

التقدير الطيفي، تفاعل الأزوتة حامض

السلفوساليسيليك، ميترونيديازول

معلومات المؤلف

الايمل:

الموبايل:

الخلاصة:

تم تطوير طريقة طيفية جديدة وحساسة وبسيطة لتقدير الميترونيديازول ، تعتمد هذه الطريقة على اختزال الميترونيديازول بمسحوق الحديد في وسط حامضي باستخدام حامض الهيدروكلوريك ثم تحضير أيون الديازونوم بإضافة نترتيت الصوديوم إلى الميترونيديازول المختزل في الوسط الحامضي وليقترن مع الكاشف (5-حامض السلفوساليسيليك) للحصول على صبغة أزوية وردية اللون تعطي أعلى امتصاص عند الطول الموجي 503 نانومتر، وكانت خطية قانون بير في مدى التركيز من 0.5 إلى 10 ميكروغرام/مل، وقيمة معامل التقدير للمنحنى القياسي 0.9972 مما يشير إحصائياً إلى أنه يتمتع بخصائص خطية ممتازة، وتم حساب معامل الامتصاص المولاري وكانت قيمته 1.1518×10^4 لتر/مول. سم وقيمة حساسية ساندل هي 0.01485 ميكروغرام/سم² تم تطبيق الطريقة بنجاح على تقدير الميترونيديازول في صورته النقية والأشكال الصيدلانية الاقراص والمعلق الفموي.