



Spectrophotometric Determination of Isoniazid in Pharmaceutical Preparations Using Oxidative Coupling Reaction with 2,4-dinitrophenylhydrazine

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p-ISSN: 1608-9391

e-ISSN: 2664-2786

Article information

Received: 24/9/2022

Accepted: 27/3/2023

DOI: 10.33899/rjs.2023.178227

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ABSTRACT

A spectrophotometric method is proposed to determine isoniazid using oxidative coupling reaction. The proposed method depends on adding the oxidizing agent potassium periodate to the reagent solution 2,4-dinitrophenylhydrazine. Different optimum conditions are studied and explained based on various experiments and tests. The wavelength is (406) nm. Beer's law within the concentration (0.2-40) $\mu\text{g/ml}$, and with a molar absorptivity value of $0.2482 \times 10^4 \text{ l.mol}^{-1}.\text{cm}^{-1}$, while the recovery was (98.46%), and the relative standard deviation did not exceed 1.34. The method was successfully applied for the determination of isoniazid in its pharmaceutical preparations (medicine tablets).

Keywords: Spectrophotometric method, isoniazid, pharmaceutical preparations, 2,4-dinitrophenylhydrazine.

INTRODUCTION

Isoniazid Fig. (1), is a medicine known by its simple chemical structure, in which, pyridine and a hydrazine group are shared to constitute 4-cyanopyridine and hydrazine are reacted in an alkaline medium to prepare the isoniazid, and the chemical structure of isoniazid was shown in Fig. (1). (Sittig, 1988; Sycheva *et al.*, 1972).

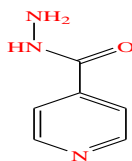


Fig. 1: Chemical structure of Isoniazid (Saleem, 2018)

Isoniazid is one of the most important drugs used to treat tuberculosis. It has a significant effect on germs and against the rapid multiplication of fungal bacteria (Zhang, 2005; Majeed *et al.*, 2022). It is worth noting that this drug was synthesized in 1912, thereafter, this medicine has been considered in the early 1950s as an anti-tuberculosis agent. This was reported by researchers from USA and Bayer in Germany (Bernstein *et al.*, 1952; Fox, 1952). After its discovery as an anti-tuberculosis drug, isoniazid was used for the treatment regimen for tuberculosis in 1952 (Blowers, and Cooke, 1954; Marshall *et al.*, 1952). Recently, a number of research studies have shown isoniazid derivatives with anti-tuberculosis efficacy, however, isoniazid remains at the fore as one of the best options available even 64 years after its discovery (Matei *et al.*, 2013; Martins *et al.*, 2014). In fact, it can clearly be noticed that isoniazid plays a significant role in the current treatment regimen and several clinical trials have shown its significance in new treatment plans under development (Rangaka *et al.*, 2014; Villarino *et al.*, 2015). Here are some properties of isoniazid:

- The molecular weight is 137.139 g/mol
- Molecular formula $C_6H_7N_3O$
- It is in the form of crystals or white crystalline powder
- Quickly soluble in water and soluble in alcohol, chloroform and ether.

2,4-Dinitrophenylhydrazine Fig. (2)

It is known as a Brady's reagent and indicated as DNPH or DNP, its color is a red to yellow in the form of crystalline, its chemical formula is $C_6H_3(NO_2)_2NHNH_2$ and its molecular weight is 198.14 g/mol. Its melting point is approximately 194°C. It is sparingly soluble in water or alcohol and somewhat soluble in inorganic acids. This can be used to detect aldehydes and ketones. It is sensitive to shocks and may result in a sharp explosion, so it must be handled with caution. The chemical structure of 2,4-dinitrophenylhydrazine is shown below (Tameed, 2006; Eisa, 2006; Saleem, 2022).

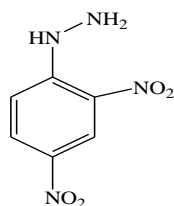


Fig. 2: Chemical structure of 2,4-DNPH

EXPERIMENTAL

Devices

1. The absorbance was measured and the absorption spectrum was drawn using a Shimadzu UV/vis 1800 Double-beam spectrophotometer-Japan.
2. Protopathic quartz cells 1 cm.
3. Weighing operations are carried out using a Sartorius type sensitive scale
4. Heating with an electro. mag type water bath

Substances and chemical solutions

All the chemicals used were of a high degree of purity and were produced by BDH Company, and their solutions were prepared as follows:

Isoniazid Standard Solution (100 µg/ml):

The solution was prepared with a weight of 0.0100 g of isoniazid (Samarra Pharmaceutical Laboratory - Iraq) and dissolved in distilled water and the volume was completed with distilled water up to the mark in a volumetric flask of 100 ml and kept in a dark colored flask where the solution is stable for at least one week from the date of preparation.

Potassium periodate solution KIO₄ (0.1%):

The solution was prepared with a weight of 0.1 g potassium periodate and dissolved in distilled water and then filled to the mark with distilled water using a 100 ml volumetric flask.

Reagent solution 2,4-dinitrophenylhydrazine (2×10⁻³) M:

The reagent solution was prepared by dissolving 0.0397 g of 2,4-DNPH reagent in 2 ml of concentrated sulfuric acid and the volume was filled with distilled water to the mark in a 100 ml flask.

Sodium hydroxide solution NaOH (approximately 1 M):

The base solution was prepared with a weight of 4 g of NaOH and dissolved in distilled water, then the volume was filled with distilled water to the mark in a 100 mL volumetric flask.

Pharmaceutical solution (100 µg/ml):

Each tablet contains 100 mg of isoniazid, five tablets have been crushing well, then weighing an equivalent to 0.0100 g of pure isoniazid was dissolved in distilled water with simple heating, then filtered and washing with distilled water. Finally, completing the final volume to the mark using a 100 ml volumetric flask, the concentration was equal to (100 µg/ml).

Solutions of surfactants (0.1%)

The solutions were prepared by dissolving 0.1 g of each one (Cetaflon CTAB, SDS and TritonX-100) in distilled water in a 100 mL volumetric flask.

Interferential solutions (5000 µg/ml)

The solutions were prepared by dissolving 0.5 g of each one the interfering substances used in distilled water, completing the volume to the mark, with distilled water in a 100 mL volumetric flask.

RESULTS AND DISCUSSION

The proposed method for the determination of isoniazid depends on adding the oxidizing agent potassium periodate to the reagent solution 2,4-DNPH. The wavelength is 406 nm.

Preliminary study

A number of preliminary tests were carried out using 25-mL volumetric flask, by adding 1 mL of (0.1%) potassium periodate to 1 mL of 2,4-DNPH reagent prepared at a concentration of 2×10⁻³ M, and then adding a volume of 2 mL isoniazid solution at a concentration of 100 µg/ml, 1 ml of 1

M sodium hydroxide solution was added, after diluting the solutions to the mark using distilled water and measured at 406 nm against blank solution.

Study of the optimum conditions for the reaction

The factors affecting the absorbance of the colored product were studied using 2 ml (100 µg/mL) of isoniazid in a final volume of 25 ml and absorbance measured at wavelength 406 nm to obtain a high sensitivity for the determination of isoniazid.

Studying the effect of reagent quantity

The effect of the quantity of 2,4-dinitrophenylhydrazine reagent on the absorption of the colored product was studied by adding increasing volumes (0.5-3) ml of 2×10^{-3} M reagent to a series of 25 ml volumetric flasks. Table (1) shows that a volume of 2 ml of the reagent gives the best value for absorbance, and therefore it was adopted in subsequent experiments.

Table 1: Effect of reagent quantity

ml of reagent (2×10^{-3} M)	0.5	1	1.5	2	2.5	3
Absorbance	0.087	0.194	0.226	0.233	0.228	0.215

Study of the effect of the type of oxidizing agent

Types of oxidizing agents were studied to choose the oxidizing agent that gives the best absorption value. 1 ml was taken from 0.1% of several oxidizing agents. It is noted in (Table 2) that potassium periodate gives the highest absorbance of the colored solution at the wavelength of 406 nm, so it was adopted as the best oxidizing agent in subsequent experiments.

Table 2: Effect of the type of oxidizing agent

Oxidant agent (0.1%)	Chemical formula	Absorbance
Sodium periodate	NaIO ₄	0.173
Potassium periodate	KIO ₄	0.241
Potassium dichromate	K ₂ Cr ₂ O ₇	0.112
Ammonium cerium (IV) sulfate dihydrate	Ce (NH ₄) ₄ (SO ₄) ₄ 2H ₂ O	0.015

Studying the effect of the amount of oxidizing agent

The effect of the amount of oxidizing agent was studied by adding different volumes (0.25-2.5) ml of potassium periodate at a concentration of 0.1% to the series of 25ml volumetric flasks. In (Table 3), it is obvious that the amount (1 ml) of the oxidizing agent gives absorbance of 0.238.

Table 3: Effect of the amount of oxidizing agent on the intensity of absorption of the colored product

ml of KIO ₄ (0.1%)	0.25	0.5	1	1.5	2	2.5
Absorbance	0.126	0.187	0.236	0.221	0.189	0.157

Study of the effect of base type

It has been shown from the initial experiments of the reaction in the preliminary study that the dye is formed only in the base medium, i.e., when the base is added. The use of sodium hydroxide gives the best absorption of the colored product, so it was approved in (Table 4).

Table 4: Effect of base type

Type of base (1M)	Absorbance
NaOH	0.240
KOH	0.228
Na ₂ CO ₃	0.175
NH ₄ OH	0.056

* 1 ml of base used

Study of the effect of the amount of base

The study was done by adding different volumes (0.25-2.5) ml of (1 M) of sodium hydroxide to the reaction mixture, and then measuring the absorbance of these solutions at wavelength 406 nm. It can be seen in (Table 5) that (1 ml) sodium hydroxide gives the highest absorbance of the colored product, so it was adopted in subsequent experiments.

Table 5: Effect of the amount of base on the colored product

ml of NaOH (1M)	0.25	0.5	0.75	1	1.5	2	2.5
Absorbance	0.141	0.193	0.212	0.234	0.227	0.217	0.205

Studying the effect of surfactants

The effect of adding different types of surfactants (positive, negative and neutral) on the reaction of product formation was studied, by taking increasing volumes of surfactant solutions (0.5-2) ml to the reaction solutions. The efficiency of these factors in improving the sensitivity of the method and the intensity of absorbance, but had a negative effect on the absorbance of the colored product, so it was ignored in subsequent experiments. Table (6) includes the effect of surfactant on the absorbance.

Table 6: Study of the effect of surfactant on absorbance

Surfactant 0.1%	Absorbance/ml of surfactant used			
	0.5	1	1.5	2
SDS	0.196	0.187	0.184	0.185
CTAB	0.178	0.181	0.183	0.143
Triton X-100	0.189	0.193	0.212	0.215
Without	0.236			

Studying the effect of the addition sequence

The effect of the addition sequence of the reactants on the absorbance of colored absorbance was studied. Therefore, different addition sequences of the reactants were conducted to choose the best sequence. It is noted from (Table 7) that the sequence (I) is the best sequence to form a colored product with the highest absorbance when the optimal conditions for the reaction are followed. Therefore, it was adopted this arrangement in subsequent experiments.

Table7: Effect of the addition sequence

Order number	Order of addition *	Absorbance
I	R +O+D+B	0.234
II	D +R+O+B	0.230
III	R +O+B+D	0.226
IV	D +B+R+O	0.185

*R=Reagent, O=Oxidizing, D=Drug, B=Base.

Study of the effect of oxidation time on absorption

The time required to complete the oxidation process was studied by taking a series of 25 ml volumetric flasks containing the specified amount of reagent solution with the oxidizing agent, leaving the solutions for different periods of time ranging (0-15) minutes, and then adding 2 ml of isoniazid at a concentration (100 µg/ml), and adding the known amount of the base, then diluting it to the mark limit with distilled water, and measuring the absorbance at the wavelength 406 nm. Table (8) includes the effect of oxidation time on the absorbance.

Table 8: Effect of oxidation time

Time (Min.)	0*	3	5	7	10	15
Absorbance	0.217	0.241	0.235	0.231	0.229	0.224

* Immediately

The results in (Table 8) show that the effect of oxidation process needs 3 minutes to be completed.

Studying the effect of temperature and time on the stability of the resulting dye

The effect of time and temperature on the stability of the formed product was studied by tracking the absorbance of the resulting-colored product in different periods of time and at different temperatures, the colored product is being stable after 10 minutes.

Table 9: The effect of temperature and time on the stability of the resulting compound

Time, (Min.)	Absorbance / Temperature (°C)		
	0	R.T (18 °C)	40
Immediately	0.217	0.217	0.218
5	0.220	0.225	0.220
10	0.224	0.235	0.224
15	0.224	0.234	0.221
20	0.223	0.234	0.221
25	0.222	0.234	0.223
30	0.222	0.234	0.224
35	0.222	0.235	0.224
40	0.221	0.235	0.222
50	0.221	0.235	0.219
60	0.222	0.234	0.210
75	0.221	0.234	0.206
90	0.221	0.234	0.206
Over night	0.228		

Final absorption spectrum

After the optimum conditions were established, the final absorption spectrum Fig. (3) of the product formed from the reaction of isoniazid with 2,4-DNPH reagent in the presence of potassium periodate in a basic medium.

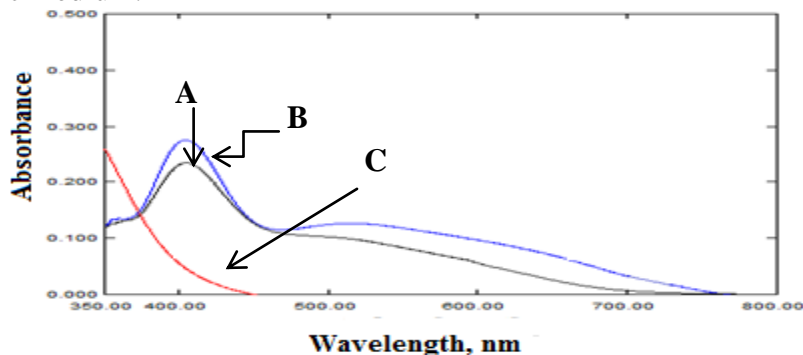


Fig. 3: Final Absorption spectrum of 100 µg/25 mL isoniazid measured against (A) blank (B) distilled water (C) blank versus distilled water

Approved working method and standard curve

After studying and fixing the optimal conditions for the determination of isoniazid, the standard curve was prepared by taking increasing volumes of isoniazid solution at a concentration of 100 $\mu\text{g/mL}$ into a series of 25 ml volumetric flasks containing 2 ml of 2,4-dinitrophenylhydrazine at a concentration of 2×10^{-3} M. and the oxidizing agent is 1 ml (0.1%), then add 1 ml of the base (1 M), then complete the volumes with distilled water to the mark, and then measure the absorbance of the solutions against the blank solution after 10 minutes of dilution at the wavelength 406 nm and by drawing the absorbance against the concentration of the isoniazid solution, the standard curve was obtained as shown in Fig. (4), which follows Beer's law for the range of concentrations (0.2-40) $\mu\text{g/ml}$. The value of the molar absorptivity indicates that the method has good sensitivity, and the value of the determination coefficient indicates that the standard curve has excellent linear characteristics.

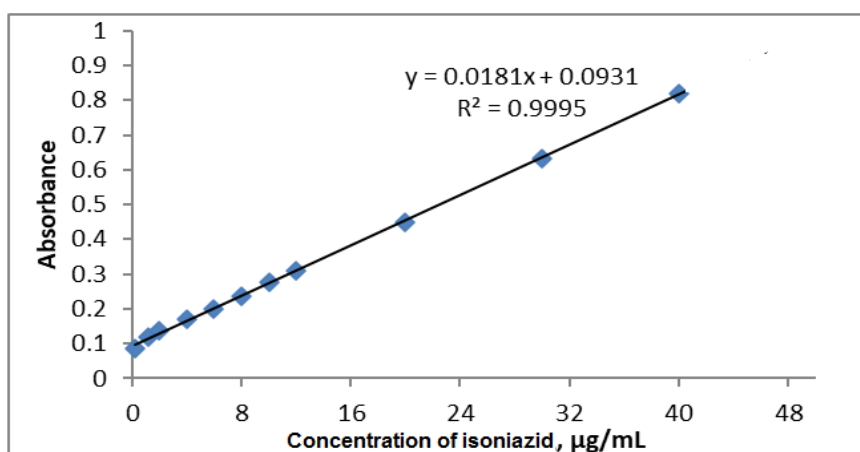


Fig. 4: Standard curve for the determination of isoniazid according to the proposed method

The molar absorptivity and Sandell's sensitivity were also calculated, and the results are shown in (Table 10).

Table 10: Results of statistical values and linear specifications of the proposed method

Linearity range ($\mu\text{g/ml}$)	0.2-40
Molar absorptivity ($\text{l.mol}^{-1}.\text{cm}^{-1}$)	0.248×10^4
Sandell's sensitivity ($\mu\text{g/cm}^2$)	0.0552
Determination Coefficient	0.9995

* For ten determinations of blank solution

Accuracy and compatibility of the method

The accuracy and compatibility of the method were studied by taking replicates of three different concentrations of isoniazid solution (2, 4 and 8 $\mu\text{g/ml}$) and they were dealt with the approved method of work by following the optimal conditions. The results shown in (Table 11) show that the method has good accuracy, as the average recovery was (98.46%) and good agreement.

Table 11: Accuracy and compatibility of the method

Amount taken ($\mu\text{g/ml}$)	Amount measure ($\mu\text{g/ml}$)	Recovery (%)	*Average recovery	* Error (%)	*RSD (%)
2	1.983	99.15	98.46	-0.85	1.22
4	3.98	99.50		-0.50	1.34
8	7.74	96.75		-3.25	0.81

* Average of five determinations

Studying the nature of the generated product:

The continuous variation method (Job's method) was used to find out the ratio of conjugation between the drug compound (Isoniazid) and the reagent 2,4-DNPH, so that solutions of the drug compound and the reagent were prepared with equal concentrations (7.292×10^{-4}) molar, from which a number of solutions were prepared by mixing volumes of the drug compound and the reagent so that the final volume was constant. (6 ml) and by following the optimal conditions and diluting to the mark with distilled water in volumetric flask of 25 ml, the absorbance of the solutions was measured at the wavelength 406 nm versus the blank solution, as it was noted that the formed dye consists of a molar ratio (1:1) of (the medicinal compound: reagent) as in Fig. (5).

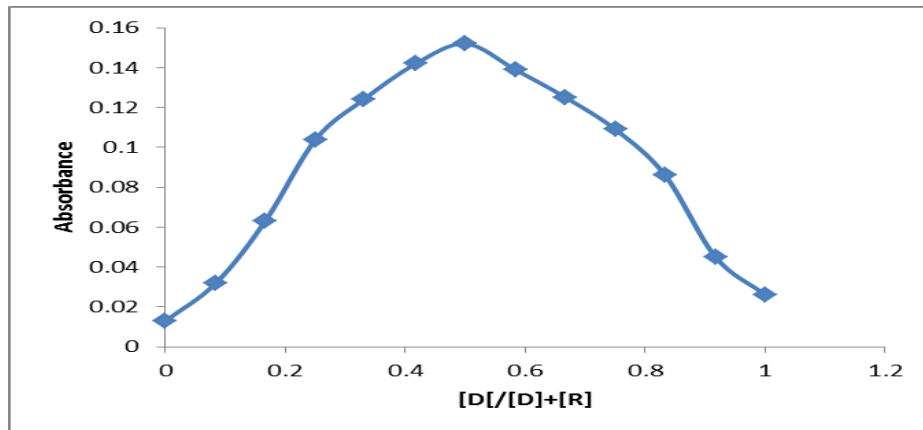


Fig. 5: Continuous variation method curve for isoniazid with 2,4-DNPH

In order to verify the conjugation ratio, the mole ratio method (Nameer 2022) was applied to know the nature of the dye resulting from the reaction of isoniazid with 2,4-DNPH reagent. A fixed volume of the drug compound at concentration of (7.29×10^{-4}) M was placed in a series of volumetric flasks of increased volumes (0.1-3 ml) of the reagent solution with a concentration of (7.29×10^{-4}) M and treated according to the optimal conditions, then the absorbance of the solutions was measured at the wavelength 406 nm after dilution with distilled water to the mark in volumetric flasks of 25 ml capacity against the blank solution, it was proved the molar ratio of the formed azo colored product (1:1) as in Fig. (6).

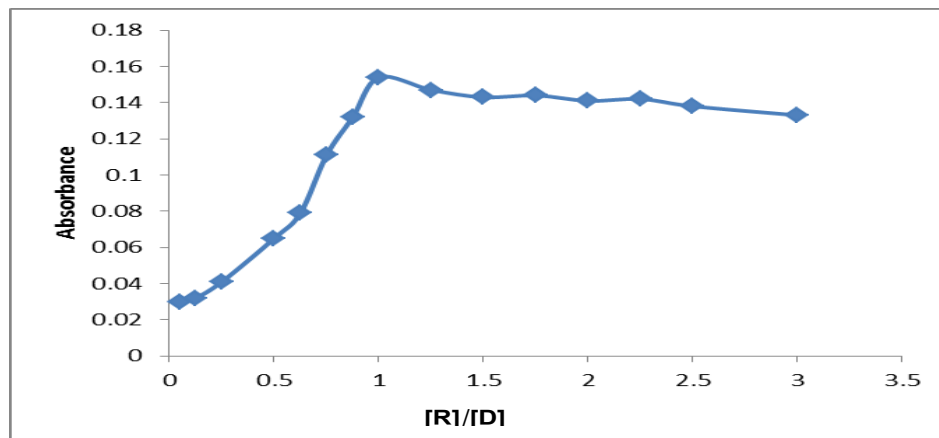
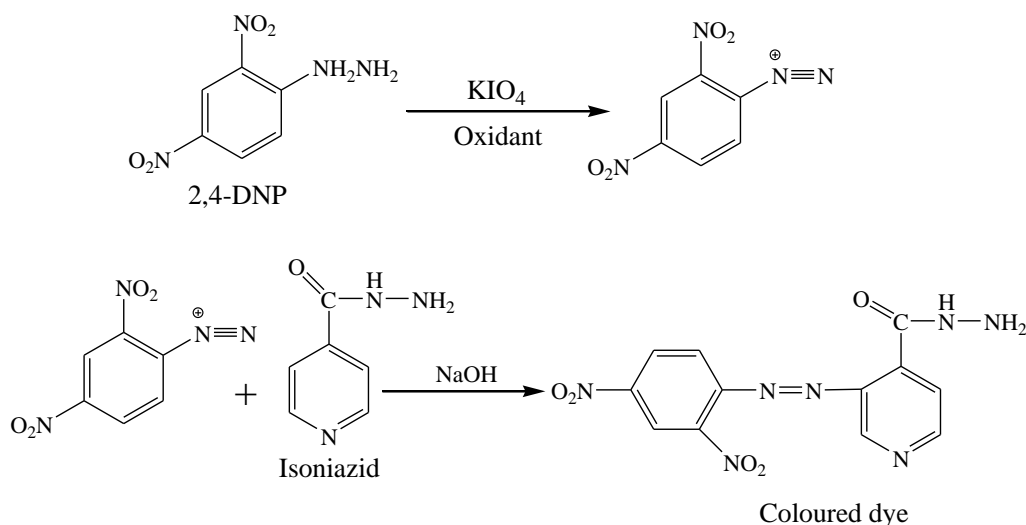


Fig. 6: Mole ratio of isoniazid with 2,4-DNPH reagent

Suggested chemical reaction

The proposed chemical reaction equation for the determination of isoniazid by the oxidative coupling reaction between isoniazid and 2,4-DNPH to form the colored product with a 1:1 mole ratio is as follows:



Study the effect of the interferences

In order to examine the selectivity of the proposed method as well as the possibility of its application to pharmaceutical preparations, the effect of a number of which substances in the method of estimation of isoniazid was studied, and the results are shown in (Table 12).

Table 12: The effect of the interferences

Additive	Recovery (%) of 100 μg /25 ml of isoniazid/ μg interference added				
	100	300	500	700	1000
Sucrose	101.53	100.34	99.65	98.77	98.29
Glucose	101.36	99.48	97.61	97.78	97.90
Starch	100.55	99.68	99.17	98.80	98.14
Gelatin	100.36	99.48	99.14	99.65	98.51

It is noted from the above table that there is no interference from the additives even when they are present in high quantities, and this indicates the possibility of applying the method successfully in the estimation of isoniazid in pharmaceutical preparations without any interference.

Application of the method

To find out the possibility of estimating isoniazid in pharmaceutical preparations by the developed method applied to the tablets, the results shown in (Table 13) confirm the success of the method for the determination of isoniazid in the studied pharmaceutical preparations.

Table 13: Result of determination isoniazid in pharmaceutical preparations

Pharmaceutical preparation	Amount taken $\mu\text{g/ml}$	Amount measured $\mu\text{g/ml}$	Recovery* %	Error* %	Relative standard deviation*%	Drug Content Found(mg)
T.B.Zide, 100mg (Egypt)	2	1.95	97.89	-2.11	1.32	97.50
	4	4.016	100.21	+0.21	1.04	100.40
	8	8.027	100.03	+0.03	1.011	100.33
Isoniazid, 100mg (S.D.I-Iraq)	2	2.016	100.22	+0.22	1.15	100.80
	4	3.972	99.17	-0.83	1.45	99.30
	8	8.005	100.25	+0.25	1.15	100.06

*Average of five determinations.

Efficiency of the proposed method

For the purpose of clarifying the efficiency and accuracy of the proposed method and that it is free of interferences, the standard addition method was applied to the pharmaceutical preparation of isoniazide. It is noted from the results shown in Fig. (7) and included in (Table 14) that the method has good selectivity and that it is free of interferences.

Table 14: Result of determination isoniazid in the pharmaceutical preparation by the standard addition method

Pharmaceutical preparation	Isoniazid measured $\mu\text{g/ml}$	Isoniazid present $\mu\text{g/ml}$	Recovery*, (%)
Izsozide 100mg (SDS-Iraq)	4.053	4	101.325
	8.014	8	100.175

*Average of five determinations

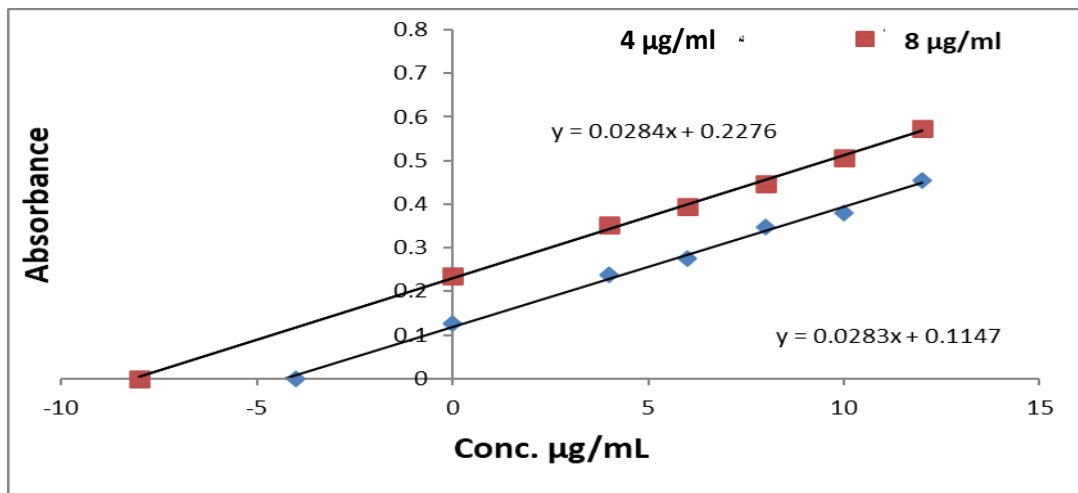


Fig. 7: Standard addition method curve for the determination of isoniazid in the pharmaceutical preparation

Comparison of the proposed method:

The proposed method for the determination of isoniazid was compared by another spectrophotometric method through some analytical specifications as shown in (Table 15).

Table 15: Comparison of some analytical variables of the proposed method with other spectroscopic methods

Analytical parameters	Present method	Literature method (Adegoke, 2019)	Literature method (Adegoke, 2019)
Reagent	2,4-Dinitrophenylhydrazine	Salicylaldehyde	p-Nitroaniline
Determination coefficient	0.9995	0.9935	0.9930
λ_{\max} (nm)	406	405	420
Beer's law range ($\mu\text{g.ml}^{-1}$)	0.2-40	13.436 -53.744	6.212 – 37.272
Molar absorptivity ($\text{l.mol}^{-1}.\text{cm}^{-1}$)	0.248×10^4	1.597×10^3	4.04×10^3
Color of the dye	Yellow	Yellow	Yellow
Nature of the dye	1:1	1:1	1:1

CONCLUSION

The proposed method was used to estimate the current in the determination of trace amount of isoniazid, using the oxidative coupling reaction. Through the optimal conditions that were identified and confirmed in the current work, it was found that the method is accurate, simple, inexpensive, and does not need pre-extraction steps or high temperatures. The method was successfully applied for the determination of isoniazid in its pharmaceutical preparations (tablets).

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التقدير الطيفي للايزونازايد في المستحضرات الصيدلانية باستخدام تفاعل الاكسدة والاقتران مع الكاشف

4,2-ثنائي نيتروفينيل هيدرازين

شيماء ميسر نايف

فرع الكيمياء الحياتية/ كلية الطب/ جامعة الموصل

داؤود حبو محمد

قسم الكيمياء/ كلية التربية للبنات/ جامعة الموصل

الملخص

يتضمن هذا العمل تطوير طريقة طيفية حساسة لتقدير الايزونازايد وذلك باستخدام تفاعل الاقتران التأكسدي بمدى تركيز (0.2 - 40) مايكروغرام/ ملتر من الايزونازايد. وتعتمد الطريقة على تفاعل الايزونازايد مع كاشف 4,2-ثنائي نيتروفينيل هيدرازين في الوسط القاعدي، ويعطي التفاعل ناتجاً اصفر اللون عند الطول الموجي 406 نانومتر. وقد بلغ معامل الامتصاص المولاري 0.2482×10^4 لتر. مول⁻¹. سم⁻¹ ومعدل نسبة الاسترجاعية 98.46% والانحراف القياسي النسبي لا تتجاوز قيمته 1.34%. وقد طبقت الطريقة بنجاح في تقدير الايزونازايد في المستحضر الصيدلاني بشكل اقراص.

الكلمات الدالة: المطيافية، الايزونازايد، المستحضرات الصيدلانية، 4,2-ثنائي نيتروفينيل هيدرازين.