Spectrophotometric Determination of Sulfathiazole Using 2,4 –dinitrophenylhydrazine as Coupling Reagent

Israa T. Humedy
Ismael M. Ali
Dept. of Chemical Engineering/ College of Engineering / University of Tikrit
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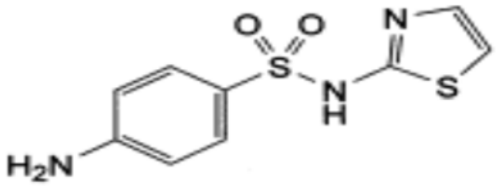
Abstract

A rapid, sensitive and selective spectrophotometric method was developed for determination of sulfathiazole (STHZ) in aqueous solution. The method is based on the oxidative coupling reaction with 2,4-dinitrophenylhydrazine (2,4-DNPH) in a basic medium (pH 10.9) in the presence of potassium periodate to produce an intense orange colour, soluble in water , stable product and absorbs at 492 nm. Beer's law was in the linear range 2.0-28.0 µg/ml of sulfathiazole, the molar absorptivity, Sandell's sensitivity index and detection limit were 1.1437 $\times 10^4$ liter. $mol^{-1}.cm^{-1}$,0.0223 $\mu g.cm^{-2}$ and 0.1274 µg/ml respectively. The RSD value was 0.75 - 1.12 % depending on the concentration. This method was applied successfully to the determination of sulfathiazole veterinary injection liquid solution (bio prime) with average recovery of not less than 100.38 % .

Keyword: Spectrophotometric, sulfathiazole, 2,4-DNPH reagent.

Introduction

The scientific name for the sulfathiazole[1] is:
4-amino-*N*-(thiazol-2-yl)benzenesulphonamide



The molecular structure for the sulfathiazole is C9H9N3O2S2 and the molecular weight of 255.3 g/mol [2]. Sulfathiazole is an organosulfur compound used as a short-acting sulfa drug.It is used for treatment on a regular basis and prevent injuries germ and the drug used as a narcotic and prohibitive for the growth of bacteria and it was used most commonly to treat urinary areas as well as resistance to infections as sulfathiazole used as antibiotics in veterinary applications. Side effects of the drug itching ,rash, allergy, edness of skin.[3,4]
Many different analytical methods were used for the determination of sulfathiazole such as spectrophotometric methods[5-7], high performance liquid chromatographic methods (HPLC)[8-14], flow-injection method[15-16], FT-Raman technique [17-18] , gas-liquid chromatographic method(GLC)[19], electrochemical method[20-21]and chemiluminescence method[22]. In this research a simple, accurate and sensitive spectrophotometric method for determining of sulfathiazole in pure form as well as in veterinary injection liquid solution (bio prime) based on the oxidative coupling using 2,4-dinitrophenylhydrazine (2,4-DNPH) in presence of potassium periodate in basic medium.

Experimental

Apparatus

Spectrophotometric measurements have been perform using shimadzu UV-Visible spectrophotometer UV-160, ultrasonic with water bath, UNISONICS, jenway pH meter 3310, Sartorius BL210 S AG and hot plate with magnetic stirrer (BIOSAN MSH 300).

Reagents and chemicals used

All chemicals and analytical reagents used in this research are of high purity.

Preparation of solutions

1-Standard sulfathiazole solution,1000 µg/ml^{-1}

The solution was prepared by dissolving 0.1 g of sulfathiazole in amount of distilled water and the volume is diluted to 100 ml with distilled water in a volumetric flask. 20 ml of this solution diluted to 100 ml with distilled water, to obtain a solution with a concentration of 200 µg/ml (7.834×10^{-4} M).This solution was prepared to be used not more than one month.
The absorption spectrum of this solution versus distilled water on Figure (1) shows that the a maximum absorption of this solution is 286 nm.

2- 2,4-dinitro phenylhydrazine reagent solution (2×10^{-3}M)

The solution was prepared by dissolving 0.1981g of 2,4-dinitro phenyl hydrazine in 5 ml of sulphuric acid and the volume is completed to 100 ml in a volumetric flask with distilled water, then 20 ml of this solution diluted to 100 ml with distilled water to obtain a solution with a concentration of (2×10^{-3}M).

3-Potassium periodate solution (5×10^{-3}M)

A 0.1150g of potassium periodate was dissolved in amount of distilled water using ultrasonic and the volume is completed to 100 ml in a volumetric flask with distilled water.

4-Interference solutions 1000 µg / ml

A 0.1000 g of each foreign compounds was dissolved in distilled water then the volume is completed to 100 ml in a volumetric flask with distilled water.

5-Sodium hydroxide solution, (approximate concentration 1.0 M)

The solution was prepared by dissolving 4.0 g of sodium hydroxide in 100 ml of distilled water in a volumetric flask, and the solutions for the lowest concentration are prepared with dilution.

6- Solution of STHZ injection formulation (1000 µg/ml)

Veterinary injection liquid solution (bio prime) (Bioagripharm GmbH-germany), every 1.0 ml contains 40 mg of sulfathiazole, the solution was prepared as follows:

The solution was prepared by taking an equivalent of 0.100 g from sulfathiazole and the volume has been completed to 100 ml with distilled water to obtain a solution with a concentration of 1000 µg/ml. A solution of 200 µg/ml is prepared by dilution of 20 ml of the above solution by distilled water in a volumetric flask of 100 ml.

Preliminary investigations

A 1 ml of 2,4- DNPH reagent is added to 1.5 ml of standard STHZ solution in the presence of 1 ml of potassium periodate solution in basic medium using 0.5 ml of 1.0 M sodium hydroxide then diluted with distilled water in a 25 ml volumetric flask to produce an orange color product. Absorption spectrum of the colored dye against its corresponding blank reagents shows maximum absorption at 492 nm in contrast to blank reagent.

Results and discussion

Optimization of the experimental conditions

The effect of various variables on the absorption intensity of 1.5 ml of sulfathiazole solution (200µg/ml), 1.0 ml of (2,4-DNPH) and 1.0 ml of KIO_4 in alkaline medium(0.5 ml,1.0M NaOH) was studied to establish the optimum conditions.

Selection of the oxidizing agent

The study was conducted by adding 1.0 ml of different types of oxidizing agents (5×10^{-3}M) to 1.0 ml of 2,4-dinitro phenyl hydrazine solution (2×10^{-3}M) and 0.5 ml of sodium hydroxide solution(1.0 M)such as: ammonium ceric sulphate dihydrate, N-bromosuccinimde potassium hexacynoferrate(III), potassium periodate, potassium iodate and ferric sulphate, the results showed the potassium periodate solution gives a higher intensity for colored product at 492 nm compared with other oxidizing agents used , so this oxidizing agent was selected in subsequent experiments.

Effect of pH

The effect of pH was studied by adding 0.1-2.5 ml of 1.0 M sodium hydroxide solution. The best pH is found to be in the range of 9.8–11.2, so the pH of 10.9 and 1.5 ml of sodium hydroxide solution was adopted in subsequent experiments, the results are shown in Table (1).

Effect of the amount of oxidizing agent

The study was conducted to select the best amount of oxidizing agent KIO_4 (5×10^{-3}M) by adding different volumes (0.3-2.5 ml) of oxidizing agent to volumetric flasks containing 1.5 ml of STHZ (200 µg/ml) and 1.0 ml of 2,4-DNPH reagent (2×10^{-3}M), then addition of 1.5 ml of 1.0 M sodium hydroxide and the volume was completed to 25ml with distilled water. The results shown in the Table (2) indicate that the volume of 1.2 ml of oxidizing agent KIO_4 (5×10^{-3}M) is the optimum amount because of highest absorbance, so it was selected in subsequent experiments.

Effect of the amount of coupling reagent

The effect of the amount of coupling reagent was studied by adding different volumes (0.3-2.5ml) of reagent solution (2×10^{-3} M) to the volumetric flasks containing 1.5 ml of sulfathiazole (200µg/ml) and 1.2 ml of oxidizing agent KIO_4 (5×10^{-3}M), then the addition of 1.5 ml of 1.0 M sodium hydroxide and the volume is completed to 25ml with distilled water, the results are shown in Table (3), it is clear that the volume of 2.0 ml of 2,4-DNPH reagent (2×10^{-3}M) is the optimum amount because it gave the highest absorption. So it is used in subsequent experiments.

Order of additions

The effect of different orders of addition on the absorption of the colored product was studied.The results are shown in Table (4) indicate that the addition (STHZ + 2,4−DNPH + KIO_4 + OH^-) achieves a higher absorption of colored product. So it was adopted in subsequent experiments.

Effect of oxidation time

The color intensity reached maximum, after drug it reacted with 2,4-DNPH and KIO_4 for 10 min in basic medium, therefore, a 10 min is sufficient for the oxidation to be completed, so it is adopted in the subsequent experiments. The color obtained was stable for 70 min and the results are shown in Table (5).

Effect of temperature

The effect of temperature (5-60°C) on the absorption of the colored product was studied. The results are shown on Figure (2) indicate that the optimum temperature is (25°C) because it gave the best absorption. So it is used in subsequent experiments.

Effect of the solvents

The effect of the solvents on the formed colored product was studied, the dilution was carried out by different organic solvents instead of water. The results are shown in Table (6) indicate that the water is a good medium for reaction and gives good absorption value at the wavelength of 492 nm and due to its availability, it has been used as the best solvent in the subsequent experiments.

Final absorption spectra

The spectrum of the colored product by coupling of 1.5 ml of sulfathiazole solution (200µg/ml) with 2.0 ml of 2,4-DNPH (2×10^{-3}M) in the presence of 1.2 ml of KIO_4 (5×10^{-3}M) in basic medium (1.5 ml,1M NaOH) (pH 10.9) and temperature 25°C against its corresponding reagent blank show a maximum absorption at 492 nm in contrast to the blank reagent. The spectra are shown on Figure (3).

Procedure for construction of calibration curve

To a series of volumetric flasks (25ml), 0.25-3.5ml of (200 µg/ml) of sulfathiazole were transferred, 1.2ml of KIO_4 (5×10^{-3} M) and 2.0 ml of 2,4-DNPH reagent (2×10^{-3}M), 1.5 ml of 1.0M sodium hydroxide solution(pH 10.9)were added at 25°C . After that the solutions were left for 10 min to complete the reaction, then the volumes were completed to the mark with distilled water. The absorbance was measured at 492 nm against the blank reagent. Figure (4) illustrates that the calibration curve is linear over the concentration range of 2.0 -28.0 µg/ml while higher concentrations show a negative deviation from Beer's law. The molar absorptivity value is 1.1437×10^4 liter. $mol^{-1}.cm^{-1}$and the Sandell's sensitivity index 0.0223µg/cm^2.

Accuracy and precision

Accuracy and precision were studied by measuring absorption at 492 nm for two different concentrations of the drug within the limits of Beer's law, the average recovery (99.88 %) and the relative standard deviation (< 1.12 %) indicate that the method is of high accuracy and precision . The results are shown in Table (7).

Detection limit

Detection limit was calculated by measuring the absorption for the lower concentration 2 µg/ml at optimal conditions at 492 nm. The results are shown in Table (8).

The nature of the formed product

To know the nature of the formed orange color complex (stoichiometry of drug with the reagent), Job's method and molar ratio method were applied. In both methods, the concentration of each of the standard STHZ solution and 2,4-DNPH reagent solution is equal to 7.834×10^{-4} M. In Job's method, in a series of volumetric flasks (25 ml), different volumes of the drug solution ranging from 1-9 ml and different volumes (9-1 ml) of reagent solution were mixed. A 1.2 ml of potassium periodate (5×10^{-3} M) and 1.5 ml 1 M of sodium hydroxide solution were added and volumes were completed to the mark with distilled water. The absorbance was measured at 492 nm against the blank reagent. The results as it Figure (5) show that the ratio is 1:1.

In molar ratio method, 1.5 ml of the standard drug solution (7.834×10^{-4} M) in a series of volumetric flasks (25 ml) were transferred and different volumes 0.25 – 4.0 ml of 2,4-DNPH reagent solution, 1.2 ml of potassium periodate (5×10^{-3}M) and 1.5 ml 1.0 M of sodium hydroxide solution were added. The volumes were completed to the mark with distilled water and the absorbance was measured at 492 nm against the blank reagent. Molar ratio was found to be 1:1. The results are shown in Figure (6) which is in agreement with the Job's method results . Scheme 1 shows the formed complex structure.

Calculation of the stability constant

Stability constant for the dye formed was calculated according to mole ratio method under the optimum conditions. For the reaction between a drug and reagent giving ML complex, degree of dissociation and stability constant was calculated [23, 24]:

$$M + L \longrightarrow ML$$

$$K = \frac{[ML]}{[M][L]} \longrightarrow \frac{[C\,(1-\alpha)]}{[\alpha\,C][\alpha\,C]}$$

If the α degree of dissociation and C concentration of colored product,so:

$$K = \frac{[C\,(1-\alpha)]}{\alpha^2\,C^2} \longrightarrow \frac{1-\alpha}{\alpha^2 C}$$

$$\alpha = \frac{Am - As}{Am}$$

Am = is the greatest value of the absorption

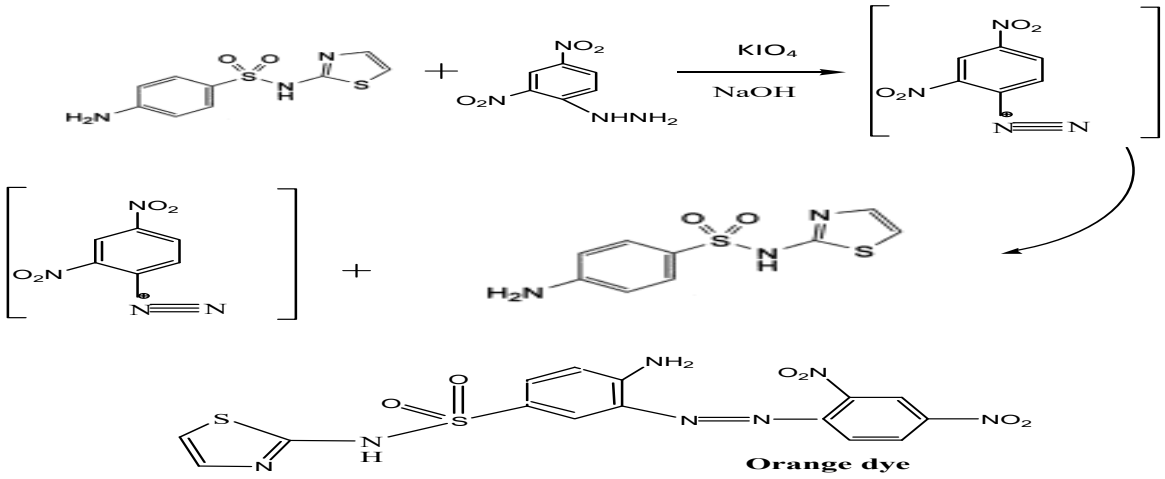
As = absorption value at the equivalence point (when the ratio of product 1:1)

The stability constant was calculated for the value of As is taken (0.458) when the volume of both (drug and reagent) is 1.5 ml of 7.834×10^{-4} M, while the value of A_m (0.482) is taken at volume of 4 ml for 7.834×10^{-4} M reagent. The value of the stability constant, is found to be 4.89×10^{5} liter.mol^{-1} for colored product STHZ - 2,4-DNPH under optimal experimental conditions and this indicates that the product is of high stability.

Effect of interferences

In order to test the efficiency and selectivity of the proposed method, the effect of some foreign substances that usually present in dosage forms was studied by taking volumetric flasks (25 ml) containing 1.5 ml of sulfathiazole (200 µg / ml) , then different volumes (2.5 , 5 ,7.5 ml) of foreign substances (1000 µg / ml) were added resulting in a final concentration of (100, 200 ,300 µg / ml). The optimum conditions were applied and the volumes were completed to the mark with distilled water.The absorbance was measured at 492 nm versus blank reagent and recovery were calculated. The results showed that there is no interferences ,Tablet (9).

The proposed equation for reaction can be written as follows:



Scheme (1)

Applications

This method was applied for the determination of STHZ in its pharmaceutical formulation bio prime injection (40 mg).

Direct method

In this method, different volumes (0.25, 0.5ml) of a pharmaceutical formulation solutions (200 µg/ml) were transferred to 25 ml volumetric flasks and the resulting concentrations (2, 4 µg/ml) were treated as in construction of calibration curve. The absorbance was measured at 492 nm for six times. Recovery and RSD were calculated and Table (10) shows the efficiency and success of the developed method for the determination of STHZ in its pharmaceutical formulation, the average recovery is 100.38 %.

Standard additions method

To prove that the developed method is free from interferences , method of standard additions is applied for determining of STHZ in its pharmaceuticals. Different volumes (0.25, 0.5ml) of a pharmaceutical formulation solutions (200 µg/ml) were transferred to six volumetric flasks (25 ml) for each volume, then increasing volumes (0.25-2.0 ml) of 200 µg/ml of STHZ standard solution were added with leaving the sixth flask without addition. The solution was treated as in construction of calibration curve. The absorbances were measured at 492 nm (Figure 7) the measured concentration was calculated from the

equation of the straight line and the results shown in Table (11) indicate that method of standard additions is in consistent with the direct method within the acceptable range of error, indicating that the method is satisfactory and free from interferences. The average recovery is 101.13%.

Conclusions

The results obtained confirm that the proposed method is simple, rapid and of good sensitivity for the determination of sulfathiazole. The method is based on oxidative coupling between sulfathiazole and 2,4-dinitrophenylhydrazine reagent in presence of potassium periodate in basic medium to form orange colored dye which is water soluble, stable and shows a maximum absorption at 492 nm. This method does not require temperature control, nor use of organic solvents, or solvent extraction and it can be applied successfully for determination of sulfathiazole in veterinary pharmaceuticals formulation with recovery of not less than 100. 38%.

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Table (1) Effect of base

ml of 1.0 M NaOH	Absorbance	pH
0.3	0.198	5.4
0.5	0.251	7.9
0.7	0.278	8.6
1.0	0.322	9.8
1.2	0.358	10.4
1.5	**0.377**	**10.9**
2.0	0.345	11.2
2.5	0.294	11.6

Table (2) Effect of the amount of oxidizing agent.

ml of KIO_4 (5×10^{-2}M)	0.3	0.5	0.7	1.0	1.2	1.5	2.0	2.5
Absorbance	0.218	0.287	0.349	0.378	0.421	0.384	0.361	0.355

Table (3) Effect of the amount of coupling reagent.

ml of 2,4-DNPH ($2x10^{-3}$M)	0.3	0.5	0.7	1.0	1.2	1.5	2.0	2.5
Absorbance	0.271	0.331	0.395	0. 422	0.438	0.454	0.489	0.463

Table (4) Order of additions.

Order number	Order of addition	Absorbance
I	STHZ+2,4−DNPH + KIO_4+OH^-	0.489
II	STHZ+ KIO_4+2,4−DNPH + OH^-	0.475
III	2,4-DNPH + KIO_4+STHZ+ OH^-	0.156
IV	STHZ+ OH^-+ KIO_4+2,4-DNPH	0.108
V	2,4-DNPH + OH^-+ KIO_4+ STHZ	0.242

Table (5) Effect of oxidation time.

Time(min)	5	10	15	20	25	30	35	40	50	60
Absorbance	0.267	0.490	0.489	0.489	0.490	0.488	0.488	0.487	0.485	0.485

Table (6) Effect of the solvents.

Solvent	Water	Ethanol	Methanol	Aceton	Isobutanol	Propanol
Absorbance	0.489	0.264	--------	0.335	0.173	0.284
λ_{max}, nm	492	485	turbid	500	503	490

Table (7) Results of accuracy and precision.

Conc. of STHZ(ppm)	RSD%	Recovery*%	Average recovery%	RE%
4	1.12	99.47	99.88	-0.53
8	0.75	100.29		0.29

*** Average of six determinations**

Table (8) Detection limit.

Concentration µg/ ml	$\overline{X}$ (Absorption)	S	D.L µg/ ml
2	0.075	0.00153	0.1274

*** Average of ten determinations**

Table (9) Effect of interferences.

Foriegn compound	Recovery (%) of 12 µg . ml^{-1} of STHZ per µg . ml^{-1} foreign compound added		
	100	200	300
Starch	99.53	98.76	100.15
Glucose	100.25	99.35	99.85
Fructose	100.29	98.13	96.95
Maltose	97.39	97.54	98.18
Sucrose	100.66	100.39	102.02

Table (10) Direct method for determination of STHZ in bio prime injection.

STHZ present µg/ml	STHZ measured µg/ml	RE,%	RSD,%	Recovery*,%
2	2.03	1.50	1.03	101.50
4	3.97	-0.75	1.36	99.25

*** Average of six determinations**

Table (11) Results of standard additions method.

Type of Drug	STHZ present µg/ml	STHZ measurd µg/ml	RE%	Recovery, (%)
Injection	2	2.05	2.5	102.50
	4	3.99	-0.25	99.75

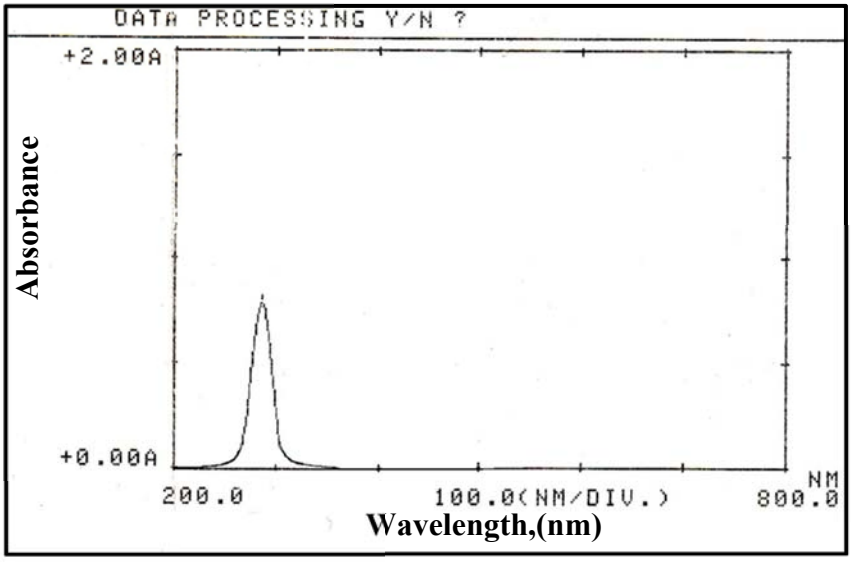

Figure (1) :absorption spectrum of the sulfathiazole versus distilled water

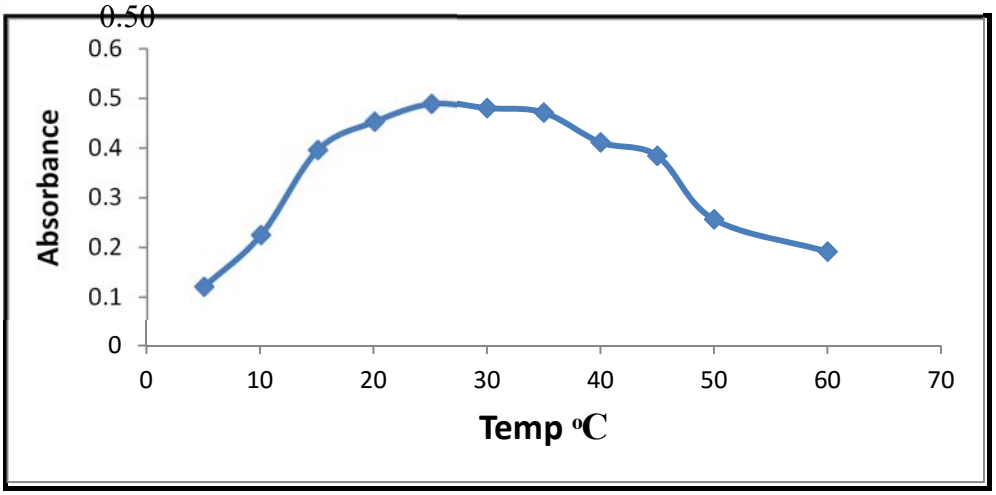

Figure (2): Effect of temperature on the absorption of the colored complex

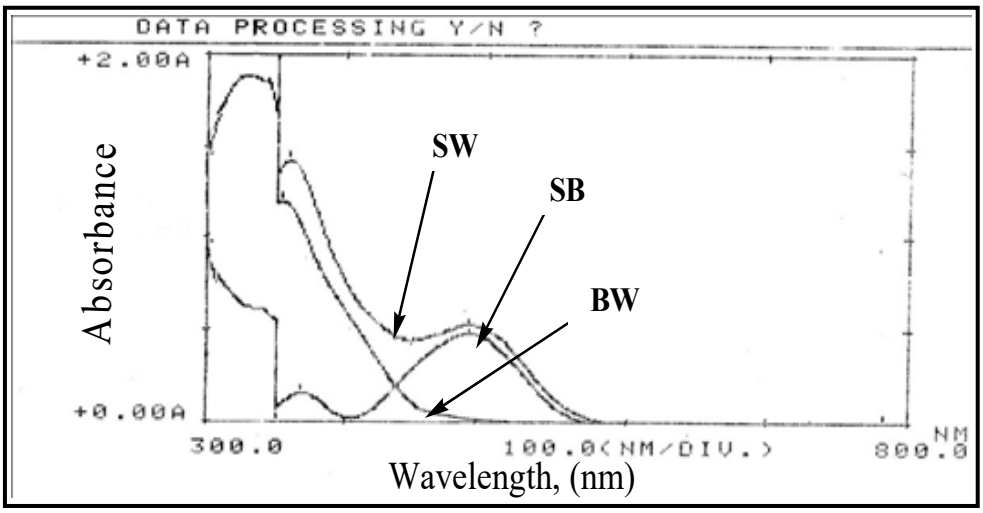

Figure (3): Final absorption spectrum of the colored complex (from 200 µg/ml Sulfathiazole).

SB : Absorption spectrum of colored complex versus blank.
SW: Absorption spectrum of colored complex versus distilled water.
BW: Absorption of blank versus distilled water.

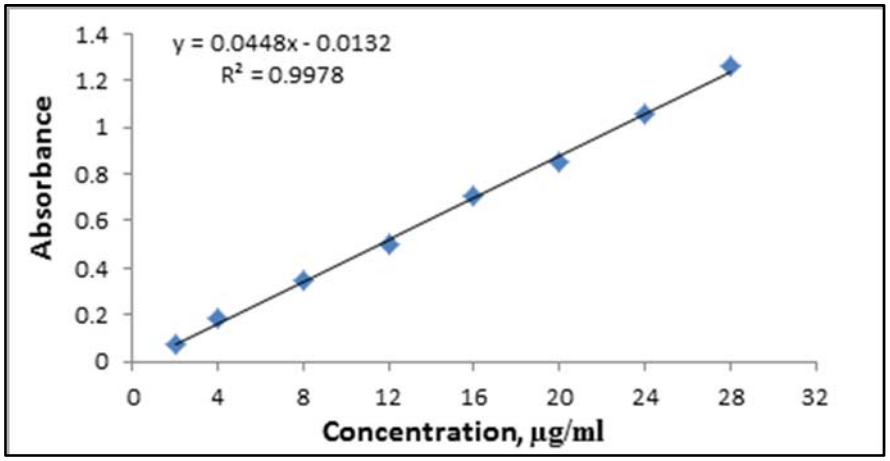

Figure (4): Calibration curve for determination STHZ by oxidative coupling with 2,4-DNPH reagent.

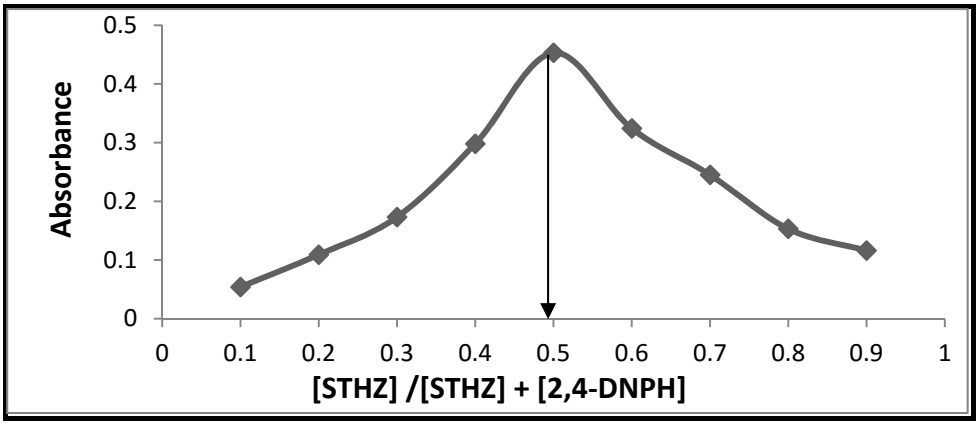

Figure (5): Job's method of formed product by oxidative coupling of STHZ(7.834×10^{-4}M) with 2,4-DNPH reagent (7.834×10^{-4} M).

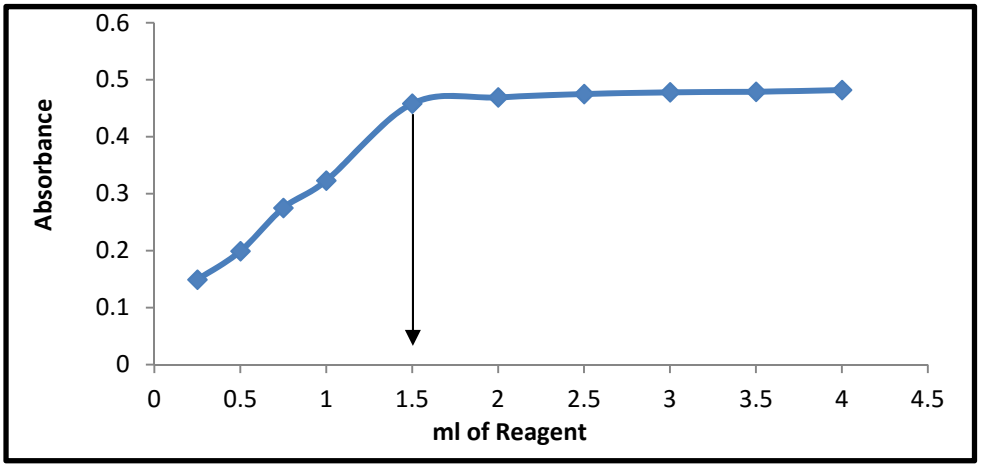

Figure (6): Molar ratio method of formed product by oxidative coupling of STHZ(7.834× 10^{-4} M) with 2,4-DNPH reagent(7.834× 10^{-4} M).

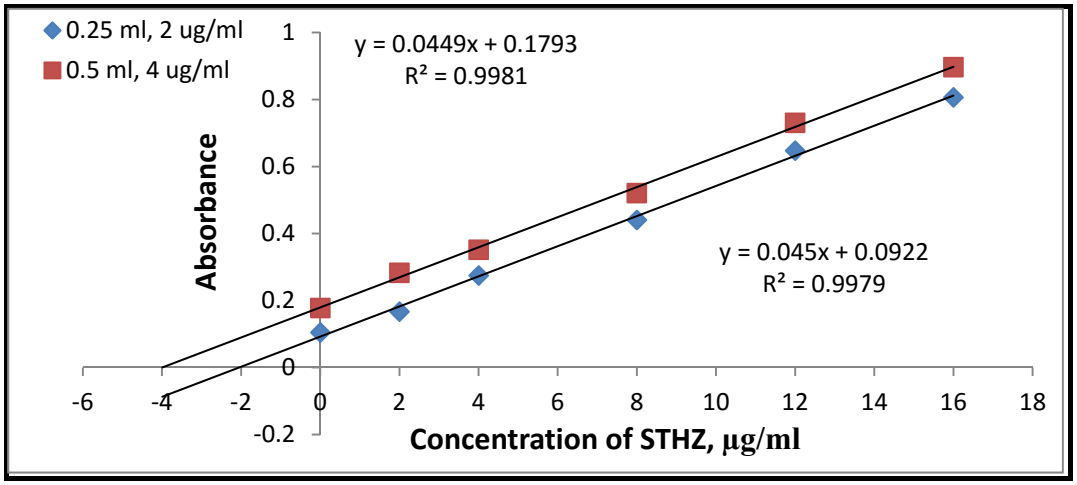

Figure (7): Standard additions curve for the determination of STHZ in injection.

التقدير الطيفي للسلفاثايازول باستعمال كاشف الاقتران 4,2- ثنائي نيتروفنيل هيدرازين

اسراء طالب حميدي
اسماعيل محسن علي
قسم الهندسة الكيمياوية/كلية الهندسة/جامعة تكريت
استلم في:6/كانون الأول/2015 ،قبل في:1/اذار/2016

الخلاصة

يتضمن البحث تطوير طريقة طيفية سريعة وحساسة وانتقائية لتقدير عقار السلفاثايازول في المحلول المائي.تستند هذه الطريقة على تفاعل الاقتران التأكسدي مع الكاشف 4,2- ثنائي نيتروفنيل هيدرازين في وسط قاعدي بوجود العامل المؤكسد بيريودات البوتاسيوم لتكوين ناتج برتقالي اللون ذائب في الماء ويعطي أعلى امتصاص عند الطول الموجي 492 نانوميتر. كانت حدود قانون بير في مدى التراكيز 2.0 - 28.0 مايكروغرام/مل من السلفاثايازول. والامتصاصية المولارية 1.1437 × 10^4 لتر.مول$^{-1}$.سم$^{-1}$ ودلالة ساندل 0.0223 مايكروغرام .سم$^{-2}$. وتراوحت قيمة الانحراف القياسي النسبي 0.75 - 1.12 % ، وحد كشف 0.1274 مايكروغرام/مل . تمّ تطبيق هذه الطريقة بنجاح لتقدير السلفاثايازول في محلول سائل الحقن البيطري (بايوبرايم) وبمعدل استرجاعية ليست اقل من 100.38 %.

الكلمات المفتاحية: الطريقة الطيفية, السلفاثايازول, 4,2- ثنائي نيتروفنيل هيدرازين.