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Colorimetric Estimation of Sulfathiazole and Application in Pharmaceutical Preparations

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Abstract

Sulfathiazole is determined by a simple and rapid visible spectrophotometric method. The method is depend on the oxidation of pyrocatechol by potassium periodate and coupling with sulfathiazole in the presence of 1N HCl, produce a coloured water soluble product that is stable and has a maximum absorption at 550 nm. Beer's law is obeyed over the concentration range of 2-24µg.ml-1, the molar absorptivity, Sandal's sensitivity index and detection limit were 0.6944×104 L.mol-1.cm-1, 0.0367µg.cm-2 and 0.0618 µg.ml-1 respectively. This method was applied successfully to the determination of sulfathiazole veterinary injection liquid solution (bioprime) .

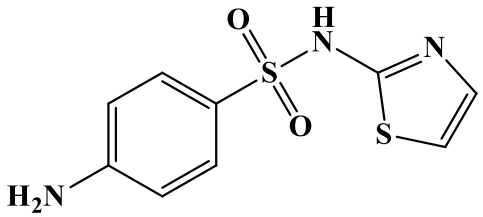
Keywords: Sulfathiazole, Pyrocatechol, oxidative coupling, Spectrophotometry.

1. Introduction

Sulfathiazole, scientific name is 4-amino-*N*-(thiazol-2-yl) benzene sulfonamide (Figure 1) [1], It is also called "norsulfazolum," "sulfanilamidothiazolum," "sulphathiatole," "sulfona–zolum," and "solfatiazolo." [2]. Sulfathiazole is a white or slightly yellowish, crystalline powder. Practically insoluble in water while slightly soluble in alcohol , It dissolves in dilute solutions of alkali hydroxides and in dilute mineral acids. Sulfathiazole belongs to the sulfonamide groups of chemotherapeutics. Despite the availability of numerous antibiotics, sulfonamide is still an important drug for therapeutic use, particularly in the treatment to treat urinary areas as well as resistance to infections .It also uses as antibiotic in veterinary applications [3,4].

Different methods were used for the determination of sulfathiazole such as spectrophotometric methods [5-7], HPLC methods [8-11] , the official method is

electrometrically titration [12], electrochemical method [13], chemiluminescence method [14] , FT-Raman technique [15] and gas-liquid chromatographic method (GLC) [16] .Potentiometric and Conduct metric methods [17,18] and anew complexes of sulfathiazole with Ni(II), Zn(II), Pd(II) and Ag(I) [19]. In this research the suggested method gives good results for estimation sulfathiazole in pure and drugs formulations which can be applicable to routine analysis of sulfathiazole and complies well with the validation requirements in the pharmaceutical industry. The study aimed at several axes: 1- Estimating the level of adropin for Iraqi cardiac disease and atherosclerosis patients, -2- Effect of the factor of obesity and other diseases (diabetes mellitus- type 2 (DM-2, and hypertensive), age and sex at the level of adropin. 3- And the relationship of adropin with the studied variables.



4-amino-N-(thiazol-2-yl) benzenesulfonamide

$C_9H_9N_3O_2S_2$ = 255.3 g/mole

Fig. (1) : Chemical structure of sulfathiazole

Different methods were used for the determination of sulfathiazole such as spectrophotometric methods[4-6], HPLC methods [7-10], the official method is electrometrically titration [11], electrochemical method [12], chemiluminescence method [13] FT-Raman technique [14] and gas-liquid chromatographic method (GLC) [15]. Potentiometric and Conduct metric methods[16,17] and anew complexes of sulfathiazole with Ni(II), Zn(II), Pd(II) and Ag(I) [18]. In this research the suggested method gives good results for estimation sulfathiazole in pure and drugs formulations which can be applicable to routine analysis of sulfathiazole and complies well with the validation requirements in the pharmaceutical industry.

2. Material and Methods

Apparatus

The UV Spectrophotometer was used (Shimadzu UV-1800 Double-beam) and matched cells of 1 cm are used.

Materials and Solutions

All chemicals used were of a high degree of purity, sulfathiazole standard material was prepared from purchased from Sigma Chemical Company (Germany) and used as received without any purification.

"Sulfathiazole stock solution (100 ppm) (3.9×10^{-4}M) : A 0.01g of sulfathiazole dissolving in 10ml of acetonitrile and the volume was diluted to 100 ml by distilled water in a volumetric flask".

"**Pyrocatechol solution (0.1%):** This solution was prepared by dissolving 0.1g of Pyrocatechol in 10ml of ethanol and dilute to 100 ml with distilled water in a volumetric flask.

"**Potassium periodate solution**", **(0.1%):** A 0.1g of KIO_4 was dissolved in amount of distilled water and the volume is completed to 100 ml in a volumetric flask . This solution was prepared by dissolving 0.1g of in 100ml of distilled water in a volumetric flask.

Hydrochloric acid solution (1N)

Recommended procedure

Aliquots of standard solution of sulfathiazole (50-600) µg were transferred into a series of 25 ml calibrated flasks, added 1 ml of potassium periodate solution (0.1%), 3 ml of pyrocatechol(0.1%) and 1 ml of Hydrochloric acid (1 N), dilute the solution to the mark with distilled water . The absorbance of the red-purple -colored product was measured at 550 nm against a reagent blank.

Procedure for veterinary injection

Ampoule of 100 ml Trisulphoprim contain 40mg/ml Sulfathiazole [provided from (Mobedco-vet) .The Arab Pesticides and Veterinary Drugs Mfg. Co. Made in Jordan] The content of ampoule was mixed well in 500 ml beaker. An 0.25ml which contains 10 mg of sulfathiazole was transferred into 100ml volumetric flask (the solution is equivalent to 1000 µg.ml^{-1} sulfathiazole) and diluted up to the mark with distilled water, and a aliquot of this solution was treated as described above for recommended procedure.

3. Results and Iscussıon

The absorption spectra of the reagent and its sulfathiazole complex are shown in (Figure.2). The maximum absorbance of the red-purple product is at 550nm which used in all subsequent experiments.

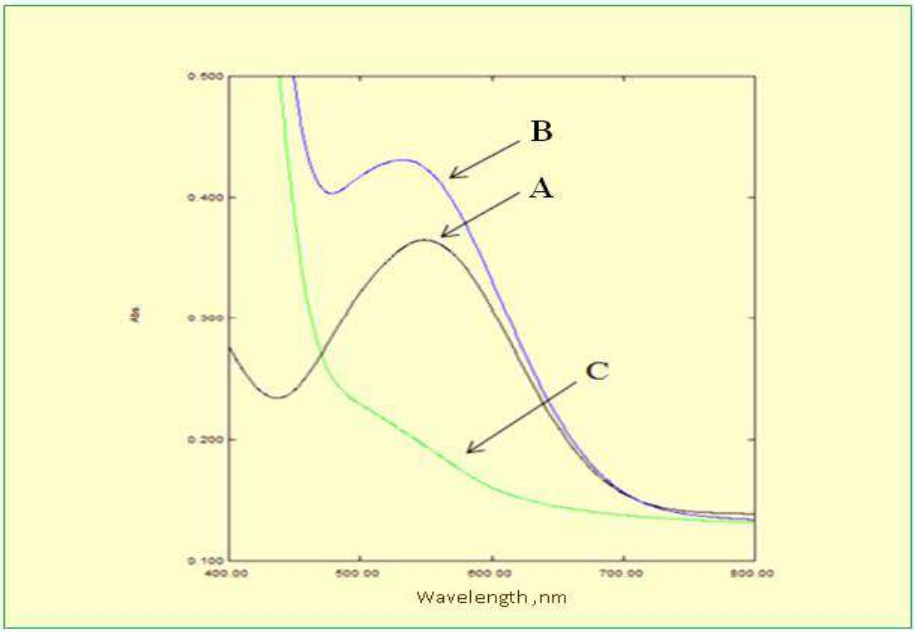


Fig.(2): Absorption spectrum of the colored product 12 µg Sulfathiazole (A) against blank, (B) the azo dye measue against D.W. and (C) blank against D.W.

Choice of acid and its amount

The preliminary experiments have shown that sulfathiazole can give high intensity of coloured dye with pyrocatechol in the presence of KIO_4 in acid medium, so that different types of acids are examined such as HCl, H_2SO_4, HNO_3 and CH_3COOH are examined and that HCl gave the best results with volume equal to 1 ml shown in (Table 1).

(Tab. 1) : Effect of HCl amount on absorbance

ml of HCl (1N)	0.5	**1.0**	1.5	2.0	3.0
Absorbance	0.267	**0.366**	0.345	0.312	0.270

Effect of pyrocatechol reagent concentration

Various volumes of pyrocatechol (0.1%) were tested , the results indicated that using 3ml of pyrocatechol solution was the optimum amount which gave the highest intensity of color (Table 2).

(Tab. 2): Effect of pyrocatechol amount on absorbance

ml pyrocatechol solution (0.1%)	Without	0.5	1.0	2.0	**3.0**	4.0
Absorbance	0.192	0.274	0.326	0.358	**0.363**	0.335

Effect the amount of KIO_4

The effect of various volumes (0.5-4.0 ml) of potassium periodate (0.1%) on the color intensity has been studied. A 2.0 ml of KIO_4 was the optimum amount which gave the greatest color intensity (Table 3).

(Tab. 3) : Effect of KIO_4 on absorbance

ml of KIO_4 sol. (0.1%)	Without	1.0	**2.0**	3.0	4.0	5.0
Absorbance	0.191	0.306	**0.361**	0.363	0.325	0.284

The Effect of Time on Oxidation of Sulfathiazole

The results in (Table 4) showed that 5 minutes was needed to give complete oxidation process before dilution with distilled water.

(Tab. 4):The effect of time on oxidative process.

Time , minutes	0	**5**	10	15	20	25	30
Absorbance	0.259	**0.365**	0.349	0.328	0.313	0.309	0.289

Time and Stability Period

The experimental results are shown in (Figure.3) indicating that maximum absorbance is obtained immediately and remains constant for at least 2 hours.

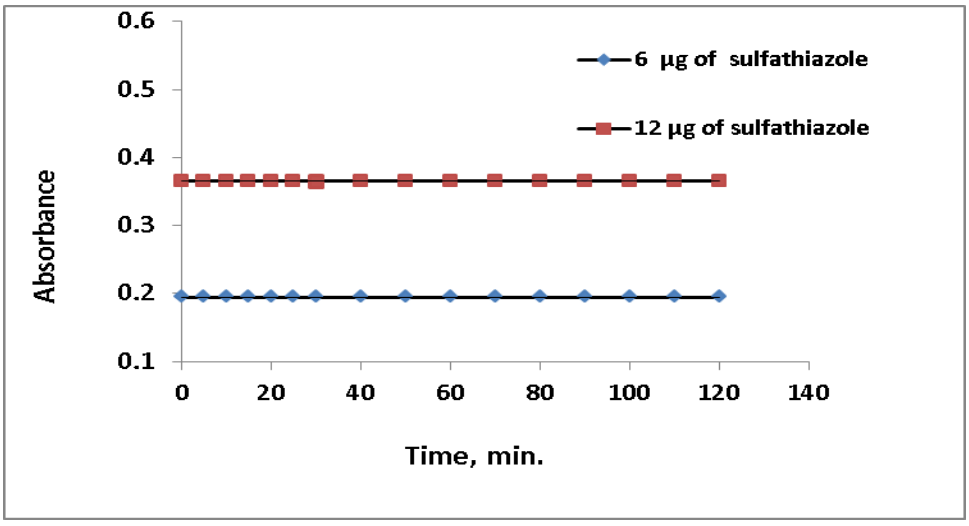


Fig. (3): Effect of time and stability period

Order of addition

The optimum orders of reagent additions be followed as given under the recommended procedure (Table 5).

(Tab. 5) : Order of addition

Order of addition*	Order No.	Abs.
S + OX + H + PC	I	0.364
S + OX + PC + H	II	0.345
OX + S + H + PC	III	0.052
OX + S + PC + H	IV	0.259
H + S + OX + PC	V	0.265
H + OX + PC + S	VI	0.185

* Sulfathiazole (S), Pyrocatechol (PC), HCl (H) , KIO_4 (OX).

Effect of Surfactants

Various kind of surfactants were studied such as cationic (CPC and CTAB), anionic (SDS) and non-ionic (Triton X-100). The experimental data reveal that the presence of surfactants gave no useful effect. Therefore, omitted in this study experiments.

Beer's law

Employing the condition described under recommended procedure, a linear calibration curve for sulfathiazole with the concentration range of 2-24µg.ml^{-1} (Figure 4). Linear regression equation : y= 0.0272x +0.0167 (R^2=0.998, n=7) where y is the absorbance and x is the concentration in µg.ml^{-1}. The apparent molar absorptivity referred was found to be 0.69441×10^4 L.mol^{-1}.cm^{-1} and sandall's sensitivity was 0.0367 µg.cm^{-2} , while (L.O.D) and (L.O.Q)[20] are found to be 0.0618 µg.ml^{-1} and 0.2061 µg.ml^{-1}, respectively.

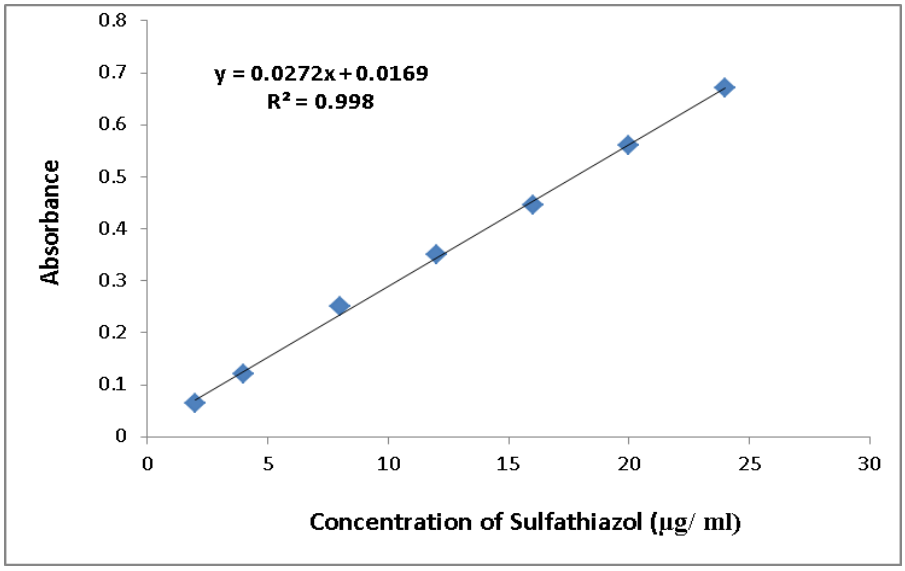


Fig. (4) : Calibration graph of sulfathiazole.

Composition of the Reaction Product

Job's method of the continuous variations[21]. (Figure 5) indicate that the structure of 1:1 sulfathiazole- pyrocatechol at 550 nm. The oxidation of pyrocatechol by KIO4 to obtain 1,2 –benzoquinone and then reacted with sulfathiazole to produce stable Red – purple product according the following suggested mechansim [22], as in (Figure 6).

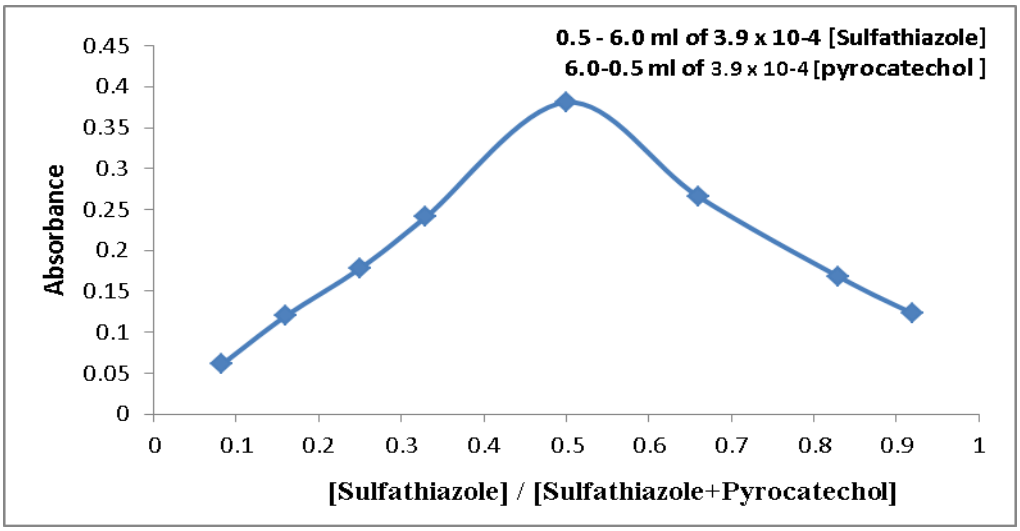


Fig. (5) : Job's plot for sulfathiazole- pyrocatechol coloured product.

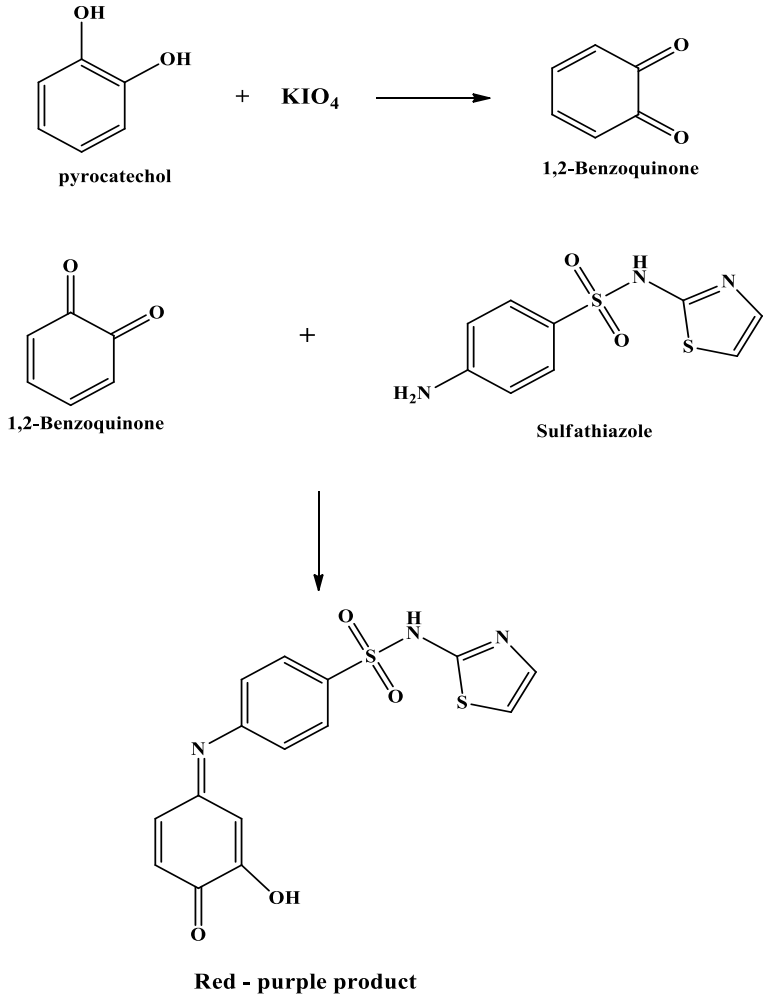


Fig. (6) :Mechanism between pyrocatechol by KIO_4 and 1,2 –benzoquinone.

Analytical Application

The experimental results in (Table 6),it has been applied to estimate sulfathiazole in drug formulation (veterinary injection). On applying proposed procedure a good recovery, accuracy and precision are obtained.

" **(Tab. 6) : Determination of sulfathiazole in pharmaceutical formulations**"

Drug	Label amount(mg)	Found by proposed method(mg)*	Recovery%
Trisulphoprim contain 40mg /ml Sulfathiazole [The Arab Pesticides and Veterinary Drugs Made in Jordan]	40mg/ ml	40.05	100.125

*mean value of five determinations

Comparison of Method

Several analytical variables those have been explained in (Table 7) shows the comparison between the proposed and method in the literature.

(Tab. 7). Comparison of method

Analytical parameters	Suggested work	Literature Method[21]
Type of reaction	Oxidative coupling	Oxidative coupling
Reagent	pyrocatechol	2,4-dinitro phenyl hydrazine
λmax(nm)	550	492
Beer's law range(µg/ml)	2.0-24.0	2.0-28.0
Molar absorptivity ($1.mol^{-1}.cm^{-1}$)	0.6944×10^4	1.1437×10^4
R^2	0.9980	0.9978
LOD(µg/ml)	0.0618	0.1274
Nature of the dye	1:1	1:1
Applications	veterinary injection liquid solution (bio prime)	veterinary injection liquid solution (bio prime)

4. Conclusions

The proposed method rapid, precise and accurate determination of sulfathiazole depend on the reaction of sulfathiazole with pyrocatechol by using of KIO_4 is developed. and all reagents utilized in the proposed methods are cheap, readily available , Moreover the procedures do not involve any critical reaction conditions or tedious sample preparation The recommended method is well suited for the assay of sulfathiazole in veterinary injection liquid solution (bio prime).

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