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Spectrophotometric Determination of Sulphamethoxazole by Cerium ammonium nitrate with Arsenazo III in pure and pharmaceuticals preparation

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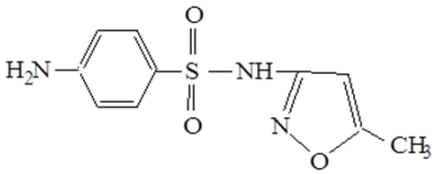
Abstract

An easy, accurate and sensitive spectrophotometric method used to determine sulphamethoxazole in pure and pharmaceutical preparation. The proposed method depends on using Arsen azo III as detector. Here we adopted on oxidative coupling reaction of sulphamethoxazole by cerium ammonium nitrate with Arsen azo III in the presence of sulfuric acid ,the dye is made of orange color, and gave higher absorption when 495 nm. Beers law is obeyed in the concentration of (1 – 7 µg.ml^{-1}). The molar absorptivity is (2.1 * 10 ^{4}) L.mol $^{-1}$.cm^{-1}, a sandal sensitivity of (0.012) µq.cm^{-2}. Limit of detection (LOD) (0.00849) µg.ml^{-1} ، Limit of Quantitation (LOQ) (0.0283) µg.ml^{-1} . The method gave a successful determination for Sulphamethoxazole in pharmaceutical preparations and the value of recovery % was better than (100.56 %).

Keywords: Sulphamethoxazole (SFMX) medication, Arsenazo III, Spectral determination.

INTRODUCTION

Sulphamethoxazole (SFMX) is member of the sulfonamide family of antibacterial and molecular formula $C_{10}H_{11}N_3O_3S$, and the synthetic formula of the drug as in Scheme [1]. White and yellowish white colored, its use has been limited by the development of resistance and it is now used mainly as a mixture with trimethoprim [1-3]. Mixture of sulphamethoxazole and trimethoprim which is known as co-trimoxazole is used to treat of bacterial infections e.g.: middle ear infections. Its main uses now are in Pneumocystis carinii pneumonia, toxoplasmosis, and nocardiosis. Gastrointestinal disturbances (mainly nausea and vomiting) [4, 5]. Literature survey indicated that few analytical methods have been reported for analysis Sulphamethoxazole. They include some spectrophotometric method [6-12], HPLC [13-20], flow injection analysis [21] and micellar electro kinetic capillary chromatography (MEKC) [22]. In this research , we succeeded in developing coupling agent for sensitive and selective spectrophotometric determination of the sulphamethoxazole drug oxidative drug by cerium ammonium nitrate in Acidic medium to form Ce^{+3} which react with Arsenazo III, to formation of coloured products complex.



Scheme 1: The chemical structure of sulphamethoxazole.

MATERIALS AND METHODS

Apparatus

AUV / Visible Spectrophotometer (1800 Shimadzu), Spectrophotometric UV1100 and 1 cm matched quartz cells was used for absorption measurements, Electronic balance (Sartorius AG Germany).

Reagents

* Stock Solution (1000 µg.ml^{-1}) Sulphamethoxazole (samara – Iraq) dissolution (0.1 gm) Sulphamethoxazole by (10 ml) pure ethanol and then diluted with distilled water to 100 ml.
* Stock Solution cerium ammonium nitrat dissolution (0.1gm) in distilled water and diluting to mark in 100 ml the bottle size.
* Arsenazo III was prepared by dissolving (0.1gm) in distilled water and diluting to mark in 100 ml the bottle size.
* Sulfuric acid H_2SO_4 (0.5 M) was prepared by take (2.72 ml) from concentration sulfuric acid and diluting distilled water to mark in 100 ml the bottle size.

Procedure for assay of Sulphamethoxazole (SFMX) in pharmaceutical preparations tablets

Metheprim tablets:

The medications used were from, the state company of drugs industrial and medical application Ninawa (N.D.I) and used for the analysis: one tablet contains 80 mg trimethoprim and 400 mg SFMX. (20) Tablets were powdered and mixed thoroughly. An amount equivalent to 100 mg of powdered was then dissolved in 5 ml ethanol and complete to 100 ml with distill water. This solution was diluted quantitatively to form concentration in the range of calibration curve.

Oral solution:

Co – trimoxazole suspension from pharaonia pharmaceuticals Egypt , Alexandria , this drug contain 40 mg trimethoprim and 200 mg SFMX each 5 ml of drugs contain 200 mg SFMX o.25 ml Transfer to 100 ml The bottle size dissolve in 5 ml ethanol and filtered and diluted up to the mark with distilled water .

Recommended procedures:

Into a series of 25 ml Volume Bottles, transfer an Increase the size of Sulphamethoxazole sol. (100 µg.ml^{-1}) to cover the range of calibration curve (1 – 7) µg.ml^{-1}, added (3 ml) cerium nitrate Alamouniakih (100µg.ml^{-1}) and shake well. Added (3 ml) Arsenazo III (100 µg.ml^{-1}). Added (0.4 ml) of H2SO4 (0.5 M), diluted by distilled water, and wait 30 min warmly the room (25 Ċ). And measurement of absorption at (495) nm opposite the blank prepared in the like method without a presence Sulphamethoxazole.

RESULTS AND DISCUSSION:

Sulphamethoxazole drug react with cerium ammonium nitrate and Arsenazo III in the presence of sulfuric acid as acidic media where the dye is made of orange color measured at 495nm **Figure(1).**

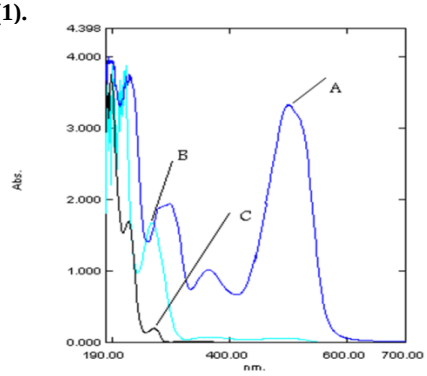


Figure 1:(A) Arsenazo III Vs. distilled water. (C): Sulphamethoxazole Vs. distilled water. (B): dye product Vs. Blank

Optimization of the action steps

The effect of various parameters on the absorption intensity of the dye formed was studied and the reaction conditions are optimized. The factors affecting colour development, reproducibility, sensitivity and conformity with Beers law were investigated with Sulphamethoxazole.

Effect of cerium* ammonium *nitrate volume:

The optimum conc. of cerium nitrate solution was found to be 3 ml cerium ammonium nitrate (100 µg.ml^{-1}) for Sulphamethoxazole. **Figure 2**

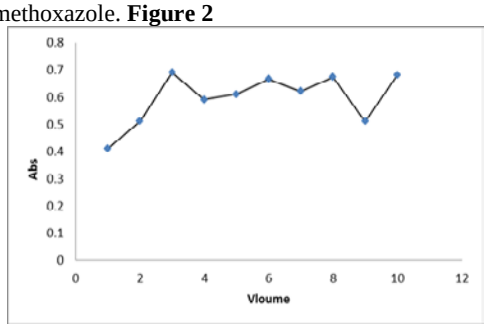


Figure 2: Effect of cerium ammonium nitrate volume

Effect of coupling agent:

The effect of the conc. of coupling reagent, has been adding 1 – 10 ml 0f (100 µg.ml^{-1}) of Arsenazo III. And found its stability in color were formed with 3 ml of Arsenazo III solution for Sulphamethoxazole in final volume of 25 ml. **Figure 3**

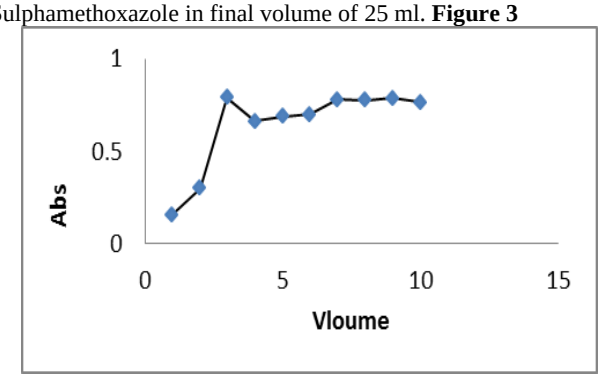


Figure 3: Effect of coupling agent

Effect of temperature:

The effect of different temperature on oxidative coupling was studied for Sulphamethoxazole and found the best temperature in room temperature (25 °C). **Figure 4**

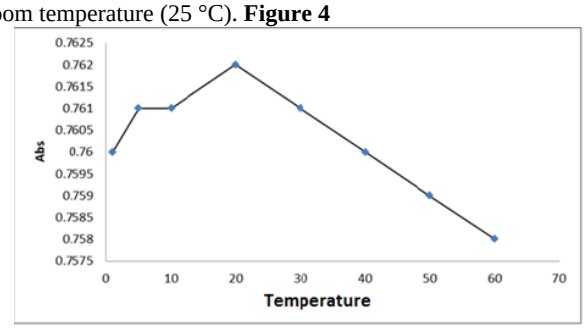


Figure 4: Effect of temperature

Sulfuric acid volume:

Optimum concentration of sulfuric acid was found to be 0.4 ml of sulfuric acid (0.5 M).

Effect of degree of addition:

Effect of degree reagents addition on the absorption of orang product dye was investigated look **Table (1)**.

Table 1: Effect of order of addition

Degree of addition	Abs.495 nm
Drug: cerium ammonium nitrate: Arsenazo III: sulfuric acid	0.385
Drug: sulfuric acid :cerium ammonium nitrate:ArsenazoIII	0.332
Drug :Arsenazo III: cerium ammonium nitrate: sulfuric acid	0.341
Drug : cerium ammonium nitrate: sulfuric acid: Arsenazo III	0.362
Sulfuric acid: Drug: cerium ammonium nitrate :Arsenazo III	0.371

Calibration Curve:

Using optimum condition, a linear calibration curve to determine of Sulphamethoxazole was determined top conc. range of (1 – 7) µg.ml^{-1}. The linear regression equation for determination Sulphamethoxazole is (Y= 0.0827x + 0.0147) and correlation coefficient of 0.9953, the linear calibration curve look **Figure (5)**. The statistical treatments of the analytical data are shown in **Table (2).**

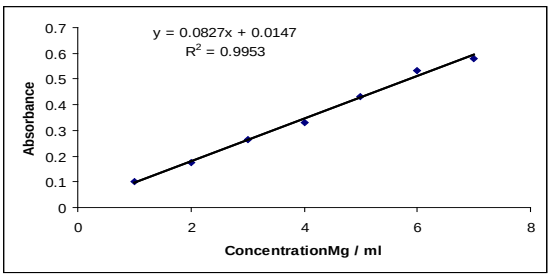


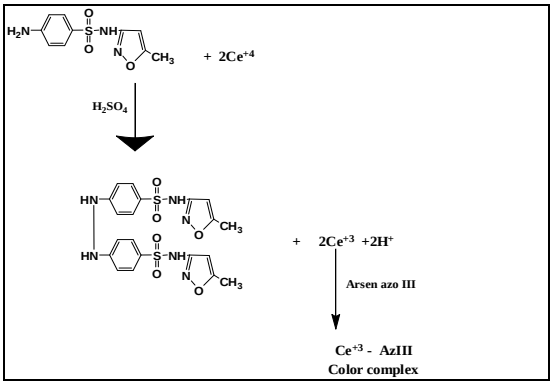
Figure 5: Calibration Curve for the determination of Sulphamethoxazole

Table 2: Statistical data for the determination Sulphamethoxazole.

Parameter	Value
λmax(nm)	495
Color	Orange
Linearity range µg.ml^{-1}	1 – 7
Regression equation	Y= 0.0827x + 0.0147
Calibration sensitivity ml .µg $^{-1}$	0.0827
Correlation of linearity	0.9953
Molar absorptivity L.mol^{-1}.cm^{-1}	2.1 * 10^{4}
Sandals sensitivity µg.cm^{-2}	0.012
LOD and LOQ µg.ml^{-1}	0.0201 and 0.0673

Interaction of the composition of the dye product

Equation of chemical reaction between Sulphamethoxazole , cerium nitrate Alamouniakih , Arsenazo III and sulfuric acid was using the mole ratio using the optimized conditions , The results in **Figure (6)** and the formation of the product as follows :



Scheme 2: The mechanism of oxidation of sulphamethoxazole.

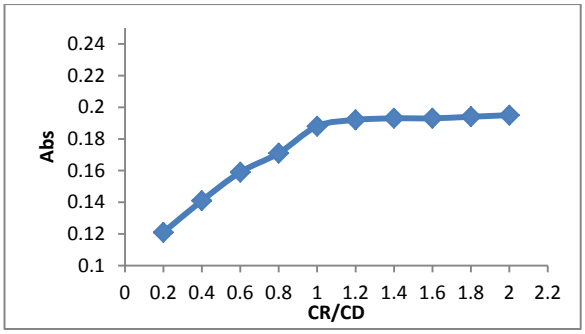


Figure 6: Molar ratio of drug to reagent

Precision and accuracy

Sulphamethoxazole were determined at three different concentrations look **Table (3)** a satisfactory precision and accuracy could be obtained with the proposed method.

Medical application

The method of work has been for the designation of Sulphamethoxazole drug in pharmaceutical preparations with good accuracy and precision for the drugs studied. The results obtained were given in Table (3) which confirms finally, the proposed procedure was compared successfully with the standard procedure **Table (2)**

Table 3: Accuracy and precision of the proposed method

Pure drugs	Taken Mg / ml)(	Found Mg / ml)(	Recovery %)(	Average * Recovery (%)	%) (RSD *
SFMX	5	5.1	102	100.72	0.434
	6	6.1	101.6		0.487
	7	6.9	98.57		0.555

***Average of five determination. SFMX= Sulphamethoxazole.**

Table 4: Result application of pharmaceutical for tablet Trimethoprim

Amount Sulphamethoxazole-{Mg / ml}	-*Found	Recovery %	Average Recovery	Er %	Average Er %	RSD%
5	5.1	102	100.56	2	1.6	0.758
6	5.9	98.3		1.6		0.785
7	7.1	101.4		1.4		0.956

***Average of five determinations**

Table 5: Result application of pharmaceutical for Oral solution

Amount of Sulphamethoxazole-{Mg / ml}	-*Found	Recovery %	Average Recovery.	Er %	Average Er %	RSD%
5	5.2	104	100.66	4	2.8	0.875
6	5.8	96.6		3		0.603
7	7.1	101.4		1.4		1.375

*Average of five determinations

Table 6: (Comparison of sulphamethoxazole determination in the proposed method and other literature methods)

Drug	Reagent	λmax (nm)	Molar absorptivity $L.mol^{-1}.cm^{-1}$	Bees law range(ppm)	Colour of dye	RSD (%)	LOD	Ref.
SFMX	8-Hydroxyquinoline	500	-	0.1 -7.0	Red	0.1-0.5	0.03-0.05	12
	Salicylaldehyde	445	-	5-40	Yellow	-	0.06	1
	2-Naphthol	482	$1.34 * 10^4$	0.21-0.66	-	-	-	5
	Orcinol	390	-	2-10	-	-	-	11
	o-Phthalaldehyde	340	-	0.01-0.24	-	1.95-2.08	-	3
	Present method	495	$2.1* 10^4$	1 -7	Orange	0.492	0.0084	

SFMX=sulphamethoxazole

CONCLUSION

A simple, accurate and excellent spectrophotometric method was investigated for the determination of Sulphamethoxazole in pure and in pharmaceutical preparations. The proposed method can be carried out with no need for further steps such as solvent extraction step. PH. or temperature control.

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