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RESEARCH ARTICLE

Spectrophotometric Determination of Sulphameter with N-1-(Naphthyl) Ethylenediamine Reagent by Using Diazotization Coupling Reaction

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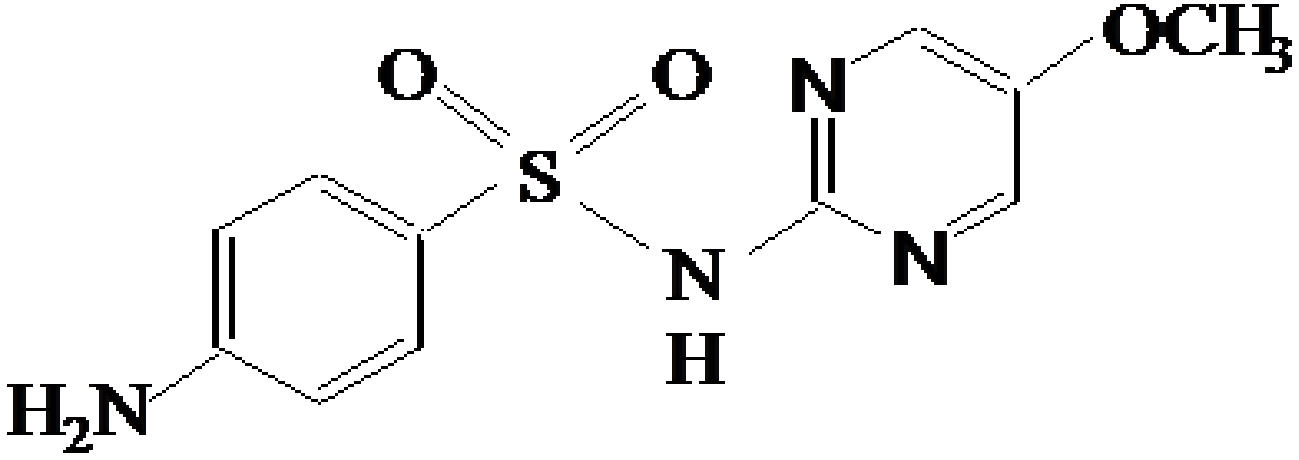
Abstract

Cutting edge method of spectrophotometric for the assay of micro amount of sulphameter The method is based on the reaction of sulphameter in acidic medium with sodium nitrite to form the diazonium ion which is coupled with N-1-(naphthyl) ethylenediamine to form a stable a pinkish- red azo dye which shows maximum absorption at 503 nm. Beer's law is obeyed over the concentration range 0.2 – 14 ppm. The molar absorptivity is (5.21×10^4) $l.mol^{-1}.cm^{-1}$, Sandal sensitivity index of (5.23×10^{-3}) $\mu g.cm^{-2}$. With a relative error of (-0.05% - + 2.3%) and relative standard deviation of (± 0.02% - ± 1.5%). The present method requires neither temperature control nor solvent extraction step.

Keywords: *Sulphameter drug, N-1-(naphthyl) ethylenediamine, Spectrophotometric Determination.*

Introduction

Sulphameter Of sulfonamides that are not dissolved in water and his scientific name 4-Amino-N-(5-methoxy-2-pyrimidinyl) benzene sulphonamide. The basic structure of the drug is shown in Scheme (1). White crystalline powder color it does not dissolve in water and dissolves in alcohol, acids and dilute bases [1] and sulphameter his half-life in the plasma is about 48 hours [2]. It exists In at least eight different forms, there are at least four crystalline crystals, a glass form, and the solubility of these forms changes as ocean temperatures change [3]. The Sulphameter is easily absorbed by the stomach but it is slowly secreted and needs a full day of liquidation [4], Literature survey indicated that few analytical methods have been reported for analysis Sulphameter. They include some spectrophotometric method [5, 6] HPLC [7, 10], the Cholometry method [11, 12]. In the present study, we succeeded in developing Spectrophotometric method of determination Sulphameter drug based on interaction Sulphameter with Sodium Nitrite In acidic surroundings to convert it into a dezionium salt After removing the non-reactive increase of nitrite, a detector is added N-1-(naphthyl) ethylenediamine to form a stable a pinkish-red azo dye Color intensity The amount of the medicine originally found in the solution is appropriate. There have been numerous attempts to find pharmaceuticals containing this drug, but have not come to fruition.



Scheme 1: the chemical structure of Sulphameter

Materials and Methods

Apparatus

A UV / Visible Spectrophotometer (160 Shimadzu) with 1 cm matched quartz cells was used for absorption measurements, CECIL - CE - 1021 Single - beam Spectrometer with Plastic Cells with Optical Path of 1 cm, Philips PW 9420 pH meter

Reagents

Sulphameter Solution (100 ppm)

Prepared by dissolving (0.01gm) of sulphameter in (15ml) absolute ethanol and then diluted with distilled water to 100 ml, this solution is kept in a dark-colored bottle. This solution is stable for at least 10 days.

Hydrochloric Acid Solution (1N)

This solution is prepared by a suitable dilution of the concentrated acid.

Sodium Nitrite Solution (% 1)

Prepared by dissolving (1) gm. of sodium nitrite in distilled water this solution is kept in a dark-colored bottle. This solution is stable for at least 20 days.

Salafamic Acid Solution (3%)

Prepared by dissolving (3) gm. of salafamic acid in distilled water and complete size In distilled water to 100 ml, this solution is kept in a dark-colored vial that is stable for at least 20 days.

N-1-(naphthyl) Ethylenediamine Solution (0.1%)

Prepared by dissolving (0.1gm) of N-1-(naphthyl) ethylenediamine in distilled water and complete size in distilled water to 100 ml, this solution is kept in a dark-colored bottle. This solution is stable for at least 5 days.

Results and Discussion

Sulphameter drug react with N-1-(naphthyl) ethylenediamine Where its dye is made up stable a pinkish- red azo dye in in acidic medium that can be measured spectrophotometrically at 503 nm Figure (1).

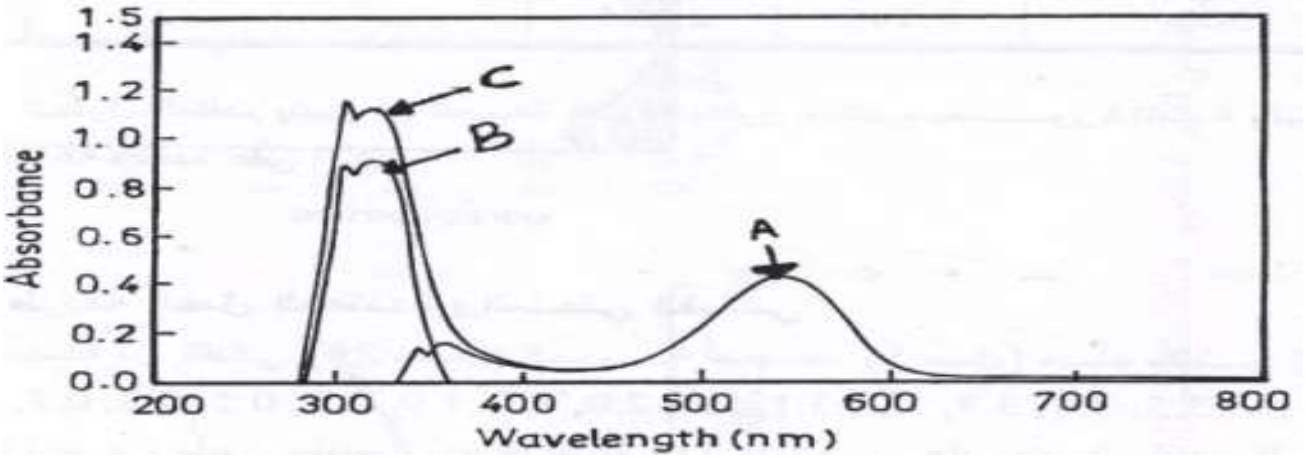


Figure 1: (A) Absorption spectrum of Sulphameter vs. Blank, (B) Blank vs. distilled water, (C) Sulphameter vs. distilled water

Optimization of the reaction conditions

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 Sulphameter.

Effect of Acid Concentration

The effect of different quantities of different types of acids on dye intensity and results was discussed in Table 1, where it was shown that the substance could be oxidized with strong and weak acids. Therefore, 3 mL of (1N) hydrochloric acid solution was used in subsequent experiments.

Table 1: Effect of the amount of different acids on absorption

Acid soln. (1M) used	Absorbance / ml of acid used				
	1	2	3	4	5
H_2SO_4	0.205	0.210	0.233	0.223	0.231
HNO_3	0.311	0.322	0.292	0.331	0.312
CH_3COOH	0.301	0.286	0.296	0.291	0.331
H_3PO_4	0.326	0.300	0.306	0.304	0.329
HCl	0.330	0.311	0.325	0.317	0.332

Effect of Sodium Nitrite

The optimum concentration of sodium nitrite solution was found to be 0.3 ml of 1% solution of sodium nitrite for Sulphameter; the results are shown in the Table (2).

Table 2: Effect of nitrite and time on absorption

ml of 1% $NaNO_2$ solution	Absorbance / min. standing time						
	0	1	2	3	5	10	15
0.1	0.311	0.321	0.331	0.325	0.319	0.320	0.340
0.3	0.309	0.338	0.328	0.312	0.451	0.440	0.430
0.5	0.357	0.345	0.321	0.366	0.340	0.331	0.357
0.7	0.311	0.363	0.397	0.340	0.334	0.329	0.349
1.0	0.324	0.322	0.315	0.325	0.316	0.329	0.324
1.5	0.335	0.319	0.305	0.338	0.329	0.316	0.307

Effect of Temperature

The impacts of different temperature on diazotization and coupling studied for Sulphameter it was found that diazotization at 0 ^{0}C and at room temperature produced the same colour intensity. Therefore working at room temperature (25 ^{0}c) was preferred it was found at higher temperatures the absorbance value decrease indicating the dissociation of the product on prolonged heating.

Effect of Coupling Agent

N-1 (naphthyl) ethylene diamine reagent was selected for coupling with sulphameter and results are shown in Table (3). Where 3 mL of N-1 ethylene 0.1% d. solution was adopted for use in subsequent experiments, since this quantity of coupling reagent gave excellent value for r, good sensitivity.

Table 3: Effect of N-1 (naphthyl) ethylene diamine

ml of 0.1% N-1 (naphthyl) ethylene diamine solution	Absorbance / µg of sulphameter						R
	10	20	30	50	70	100	
1	0.084	0.143	0.208	0.363	0.487	0.696	0.999596
2	0.085	0.145	0.216	0.373	0.498	0.712	0.999598
3	0.088	0.142	0.211	0.362	0.500	0.691	0.999425
4	0.089	0.133	0.193	0.335	0.493	0.677	0.998401
5	0.087	0.148	0.216	0.361	0.516	0.707	0.999475

Effect of Organic Solvents

Such as ethanol, acetone, Acetic acid and distilled water by using in the dilute and measure, the absorbance was finding 0.176, 0.156, 0.144 and 0. 781 respectively distilled water found to be the best.

Effect of Time on Stable Interaction

The stability of the reaction was tracked by measuring the absorption of the colored solution after different time periods and the results are shown in the following Table (4). Where it turns out that the colored pigments is directly composed directly and remains stable for at least an hour.

Table 4: Effect of time on absorption

µg of sulphameter	Absorbance / min. standing time				
	0	10	20	40	60
5	0.057	0.061	0.062	0.064	0.066
50	0.350	0.360	0.373	0.362	0.365
100	0.774	0.785	0.796	0.786	0.795
200	1.420	1.439	1.428	1.428	1.428
300	2.140	2.125	2.132	2.122	2.125

Effect of Interference

The method was examined by estimating sulphameter according to the proposed modus operandi and with different quantities of exotic compounds. The results are displayed in the Table (5). Where we observed that there was no overlap by the compounds expected to be present with the drug in pharmaceuticals.

Table 5: Effect of interferences on sulphameter estimation

Interferent	Recovery % µg of compound added				
	1000	2000	3000	4000	5000
Trimethoprim	96.5	100	100.56	101.69	101
Starch	98.4	100	100.28	100	100.8
Glucose	97.3	98.87	99.7	100.28	100
Sucrose	101.9	101.4	101.6	100.8	100.28
Lactose	98.8	99	100.28	100	99.7
Acacia	97.7	98.3	99.1	99.4	100.2

Calibration Graph

To a series of 25 ml bottles, add 5 mL of distilled water to 3.5, 3.4, 3.3, 3.2, 3.1, 3.0, 2.0, 1.5, 1.0, 0.7, 0.5, 0.3, 0.2, and (3) ml of hydrochloric acid and 0.3 ml of solution (1%) sodium nitrite and leave the solution for 2 minutes to complete the reaction and add (1 ml) of solution (% 3) Sulfamic acid and leave the solution for 3 minutes to complete the destruction of the increase of nitrite then add 3 ml of the N-1 (naphthyl) ethylenediamine solution. Then complete the size of the label with distilled water the absorption is measured directly after dilution at wavelength 503 nm, Figure (2) represents the standard curve that is in accordance with the Beers law in the concentration range (5 - 350 µg / 25 ml). It was found that the molar absorption coefficient of the resulting dye is (5.21×10^4) $l.mol^{-1}.cm^{-1}$ Sandal sensitivity index of (5.23×10^{-3}) $\mu g.cm^{-2}$

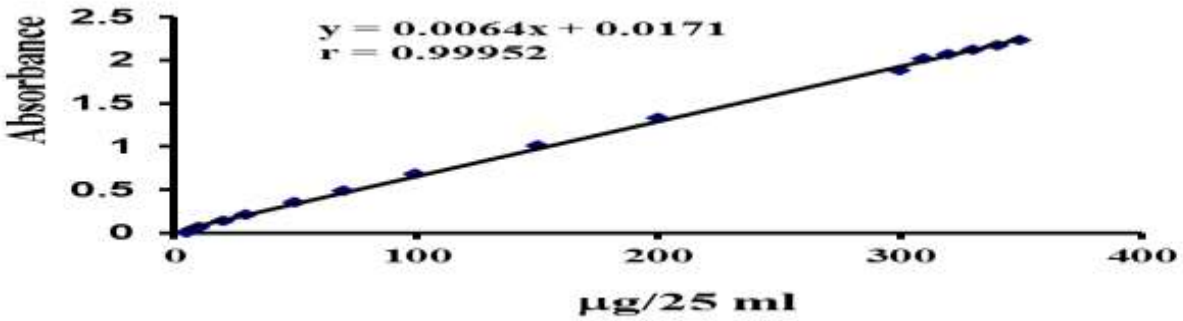
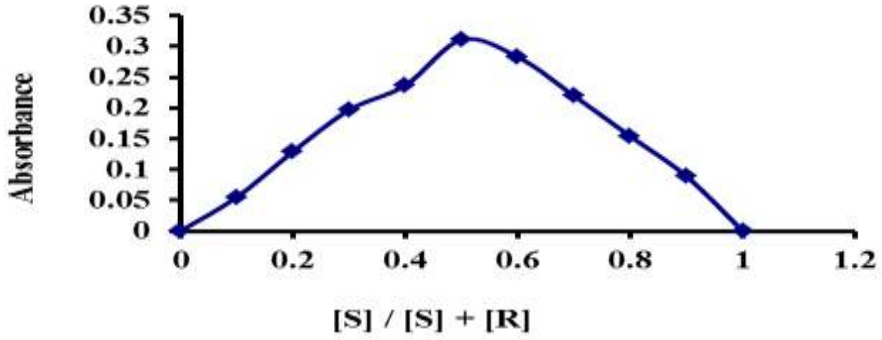


Figure 2: Calibration Curve for the determination of sulphameter

Reaction Mechanism of the Dye

The stoichiometry of the reaction between sulphameter with N-1-(naphthyl) ethylenediamine by Job's method [10, 13, 14, 18].And mole ratio method .The results obtained in Figs (3) show that 1:1 drug to reagent was formed at 503 nm for sulphameter [15, 16, 17]. The product formed was water soluble, the stability constant wascalculated by comparing the absorbance of a solution containing stoichiometric amount of 3.56×10^{-4} M both sulphameter and N-1-(naphthyl) ethylenediamine with that of solution containing the optimum amount of N-1-(naphthyl) ethylenediamine 3.56×10^{-4} M. The proposed mechanism of reaction between N-1-(naphthyl) ethylenediamine and the sulphameter drug is illustrated in Scheme (2).

(A)



(B)

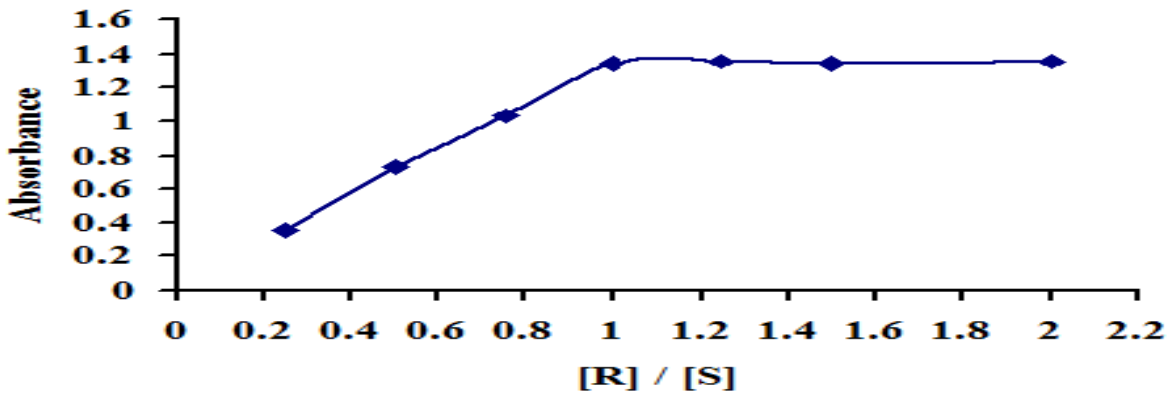
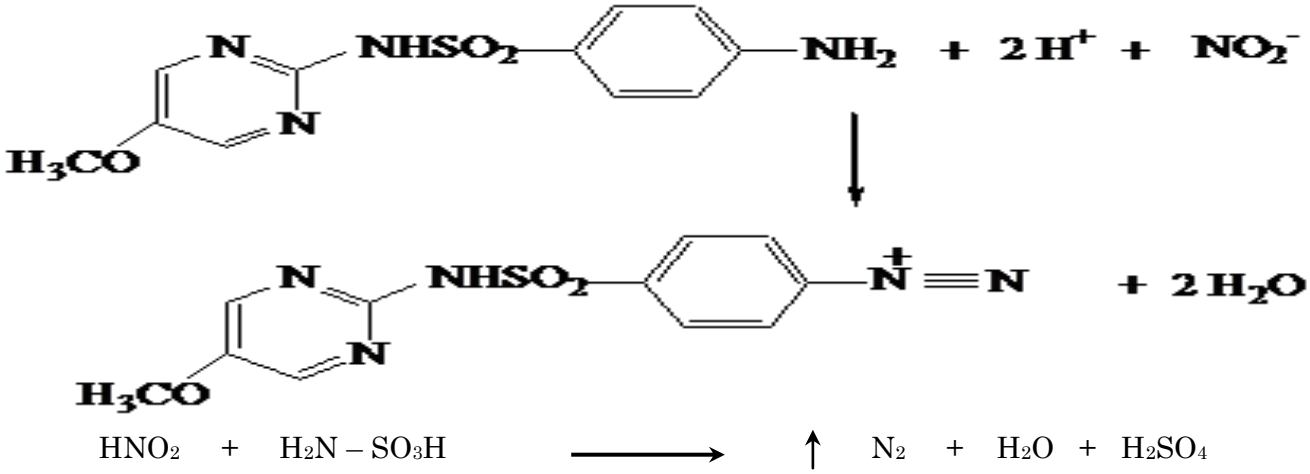


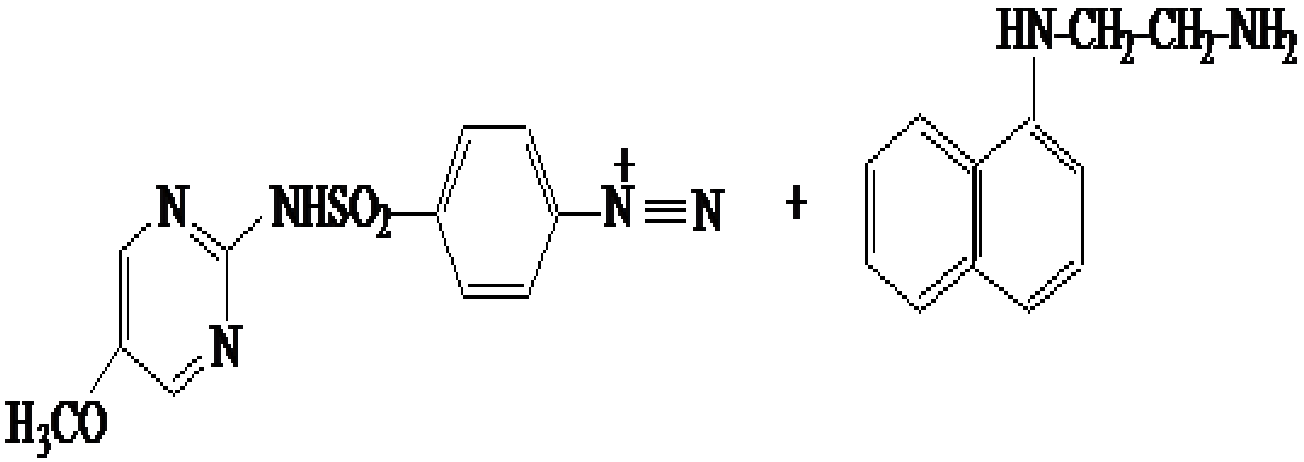
Fig 3: (A) Job's method and (B) molar ratio method of sulphameter

The sulphameter reacts with an increase of nitrite in an acidic medium to be a dezonium salt:

:The increase of nitrite is removed using sulfamic acid



The dizonium salt is combined with the N-NED reagent, the azo dye is formed:
Stable a pinkish- red azo dye



Scheme 2: scheme of the proposed reaction mechanism

Precision and Accuracy

It was possible to determine the accuracy of the sulphameter method by applying the method of work adopted in finding the standard curve for four different concentrations (350, 200, 100 and 5) micrograms of sulphameter (each with 5 readings) as shown in the following Table (6).

Table 6: Accuracy of the new method

µg of sulphameter	E_r, %*	Relative std. dev., %*
5	+ 2.40	± 1.50
100	- 1.40	± 0.13
200	- 0.05	± 0.02
300	- 0.02	± 0.02

Average of five determinations *

Conclusion

This cutting edge theories were found to be simple economical, selective and sensitive the statistical parameters and recovery study data clearly indicate the reproducibility and accuracy of the methods. Analysis of the samples containing sulphameter showed no interference from the common excipients. Hence, these methods could be considered for the determination of sulphameter in the quality control laboratories.

Table 7: Comparison of sulphameter determination in the proposed method and other literature methods

Parameters	Resorcinol method[62]	Present method
Ph.	…	1.15
λ_{max}, nm	490	503
ε, $l.mol^{-1}.cm^{-1}$	3.08×10^4	5.21×10^4
Beer's law range, ppm	0.2 – 9	0.2 – 14
Time of colour development	5	Immediately
E_r, %	-1.8 - -0.1	- 0.05 – 2.4
RSD, %	±0.1 - ±1.3	±0.02 - ±1.5
Analysis time, min	10 min	Immediately
Cost of reagent	Cheap	Cheap
Toxicity of reagent	Irritant	Irritant
Colour of dye	purple	pinkish- red
K, M^{-1}	…	7.92×10^6 to 13.31×10^6

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