

Spectrophotometric Determination of Sulfacetamide

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Abstract: Sulfacetamide is a sulfonamide antibiotic that is used as a cream to treat skin infections and as eye drops infections. On the skin it is used to treat acne and seborrheic dermatitis. In Sulfacetamide Sodium is the sodium salt of sulfacetamide, a synthetic sulfanyl acetamide derivative with bacteriostatic activity. Sulfacetamide [N (4 – aminophenyl) sulphonyl] acetamide is a N – substituted derivatives of sulfanilamide which is widely used in the treatment of urinary tract infection and burn therapy. A spectrophotometric method for the determination of trace amounts of sulfacetamide, based on coupling of diazotized sulfacetamide with m-aminophenol to form an intense orange coloured, water soluble monoazo dye which exhibits maximum absorption at 436 nm. Adherence to Beer's law is observed over the concentration range (0.4 – 8) ppm with a molar absorptivity of (1.68×10^4) $\text{mol}^{-1} \cdot \text{cm}^{-1}$, Sandell sensitivity index of (1.4×10^4) $\text{mg} \cdot \text{cm}^{-2}$, precision of ($\pm 0.28 - \pm 0.8$) and accuracy of (0 – 0.3) indicating that the method is accurate and precise. the proposed method has been successfully applied to the assay of sulfacetamide eye drops.

Keywords: Sulfacetamide, N (4 – aminophenyl) sulphonyl] acetamide, Diazotized, Eye drops

Introduction

Sulfacetamide [1] is a white crystalline powder that dissolves in water and alcohol and its water solution is evenly acidic. Sulfacetamide has a half age in plasma of about 7 hours [2]. Sulfacetamide is an early discovered sulfa drug [3], but at present, sulfacetamide is an important bacterial antibiotic used in ophthalmic solutions and ophthalmic. Sulfacetamide is a well_water soluble sulfonamide and has several scientific names, including : N_sulfanilyl acetamide or N_acetyl sulfanilamide or N-[(4- aminophenyl) sulfonyl] _acetamide] [2]. Including the following composition [4].

Methodology

Apparatus

Spectral and absorbance measurements are carried out using Shimadzu UV-160 UVVisible computerized double-beam spectrophotometer. In all measurements, 1-cm matched cells are used. The pH measurements are carried out using HANA pH meter.

Chemicals

All chemicals used are of analytical reagent grade

Sulfacetamide sodium solution (100 ppm): 0.011 g of Sulfacetamide was dissolved in distilled water then the volume was completed to 100 ml with distilled water in a dry and clean volumetric flask. Then, the solution was transferred to a dark bottle to be stable for at least 10 days.

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Sulphuric acid 1N: 98% concentration sulphuric acid solution has been used for preparing 1N of sulphuric acid solution in 100 ml volumetric flask.

Sodium nitrite solution (1%): Prepare this solution by dissolving (1) g of sodium nitrite and complete the volume to (100) in a volumetric bottle with distilled water.

Sulfamic acid solution (3%): Prepare this solution by dissolving (3) g of sulfamic acid with distilled water and complete the volume to (100) ml in a volumetric bottle with distilled water. This solution is stored in a dark volume volumetric vial and is stable for at least 20 days.

Meta-amin-phenol solution (0.5%): Prepare this solution by dissolving (0.5) grams of meta-amino-phenol in distilled water and complete the volume to (100) ml in a volumetric bottle with distilled water. This solution is stored in a dark volume volumetric vial and is stable for 10 days after which the solution becomes yellowish brown.

Sodium acetate solution (1N): Prepare a sodium acetate solution at a concentration of (1N) by dissolving (8.2) grams of the substance with distilled water and complete the volume to (100) ml in a volumetric bottle with distilled water. This solution is stored in a dark plastic bottle

Drug solutions:

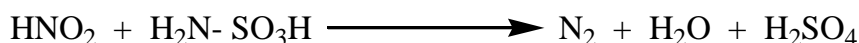
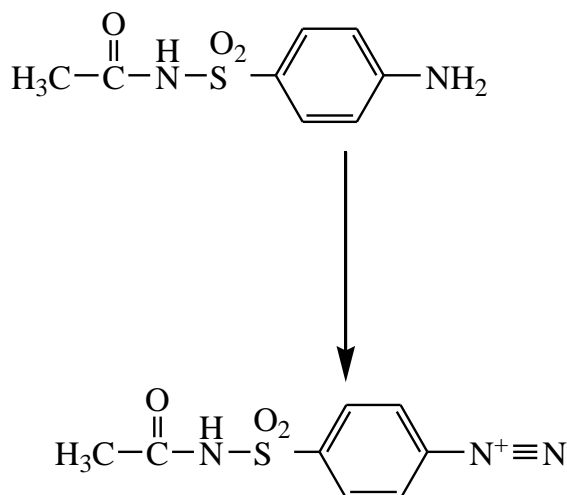
First: Samacetamide Drops (SDI): The solution is prepared by taking (0.1) ml of the drop after shaking and diluted to (100) ml using distilled water.

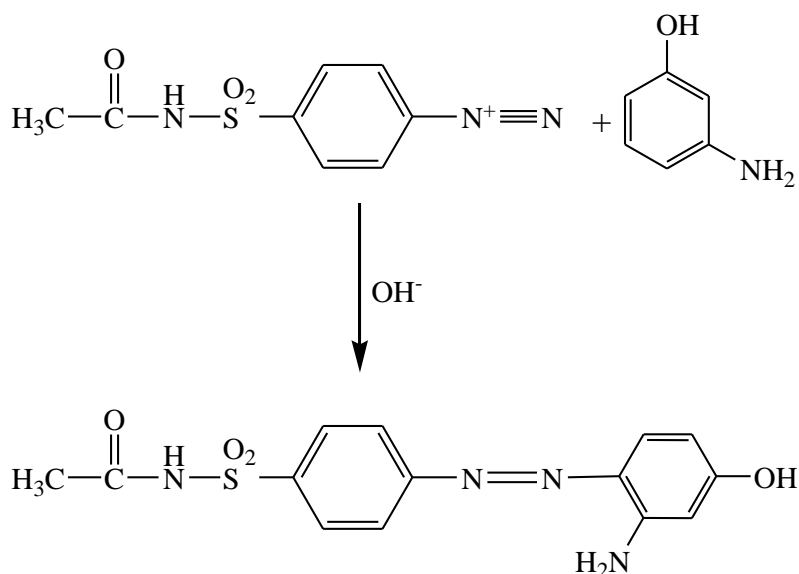
Second: Samacetamide Drops (Shifa Drug Factory - Iraq): The solution is prepared by taking (0.1) ml of the drop after shaking and diluted to (100) ml using distilled water.

Results and Discussion

(30) µg of sulfamethoxazole in a final volume (25) ml was used for subsequent experiments at wavelength (436) nm.

Principle of the Suggested Method





Study the optimal conditions for the reaction

The effect of different conditions on the optical properties of the dye produced by the reaction of sulfacetamide with an increase of nitrite in acidic medium was studied to be diazonium salt.

Effect of acid amount

The effect of different acids on the dye absorption intensity was studied (HCl, H₃PO₄, HCOOH, CH₃COOH, HNO₃, H₂SO₄). The results showed that the use of 3.5 ml of a solution of sulfuric acid at a concentration of 1 Ml gave the best results and was adopted in subsequent experiments.

Effect of nitrite quantity and time

The use of 0.7 ml of sodium nitrite solution at a concentration of 1% and the reaction time of 3 minutes gave the best results and was adopted in subsequent experiments. (Table 1).

Table 1. Effect of nitrite quantity and time

| ml of NaNO ₂ solution | Absorbance / min. standing time | | | | | | | |
|----------------------------------|---------------------------------|-------|-------|-------|-------|-------|-------|-------|
| | 0 | 1 | 2 | 3 | 5 | 10 | 15 | 20 |
| 0.1 | 0.092 | 0.113 | 0.086 | 0.113 | 0.098 | 0.102 | 0.113 | 0.062 |
| 0.3 | 0.097 | 0.108 | 0.109 | 0.096 | 0.070 | 0.076 | 0.074 | 0.074 |
| 0.5 | 0.098 | 0.105 | 0.105 | 0.113 | 0.096 | 0.104 | 0.110 | 0.100 |
| 0.7 | 0.267 | 0.239 | 0.211 | 0.211 | 0.203 | 0.201 | 0.200 | 0.212 |
| 1.0 | 0.164 | 0.140 | 0.128 | 0.118 | 0.120 | 0.122 | 0.124 | 0.122 |
| 1.5 | 0.145 | 0.134 | 0.177 | 0.186 | 0.203 | 0.190 | 0.218 | 0.270 |
| 2.0 | 0.190 | 0.075 | 0.263 | 0.297 | 0.113 | 0.221 | 0.175 | 0.252 |

Effect of meta-amino phenol intake

The results showed that using different volumes of the meta-amino-phenol reagent solution, 1.5 ml of the reagent at 0.5% concentration gave the highest value of r at 0.999041 and was therefore used in subsequent experiments.

Base effect

By studying several types of weak and strong bases such as (KOH, NaOH, NH₄OH, Na₂CO₃, NaHCO₃, CH₃COONa, HCOONa) and at a concentration of 1 molar it was found that the use of a solution of 7 ml CH₃COONa gives the best results for this use in subsequent experiments.

Effect of surfactants

The purpose of adding surface tensile agents is to increase the sensitivity of the method and increase the speed [5]. In addition, colloidal systems are stable and non-toxic. (SDS), Cetyl pyridinium chloride (CPC) and Alkyl phenol ethoxylate (Triton X-100) and practical results showed that the addition of surface tensile agents does not improve the properties of the resulting dye.

Final absorption spectrum

When the meta-amino-phenol reagent is added to the solution containing sulfacetamide, which is obtained under experimentally optimal conditions, an orange colored pigment is formed and gives the highest absorption at the wavelength of 436 nm compared to the image solution, which gives weak absorption at the same wavelength. Figure 1 shows the absorption spectrum. The resulting dye vs. solution solution and the absorption spectrum of the solution solution versus distilled water and the wavelength of the highest absorption of 436 nm were used for subsequent experiments.

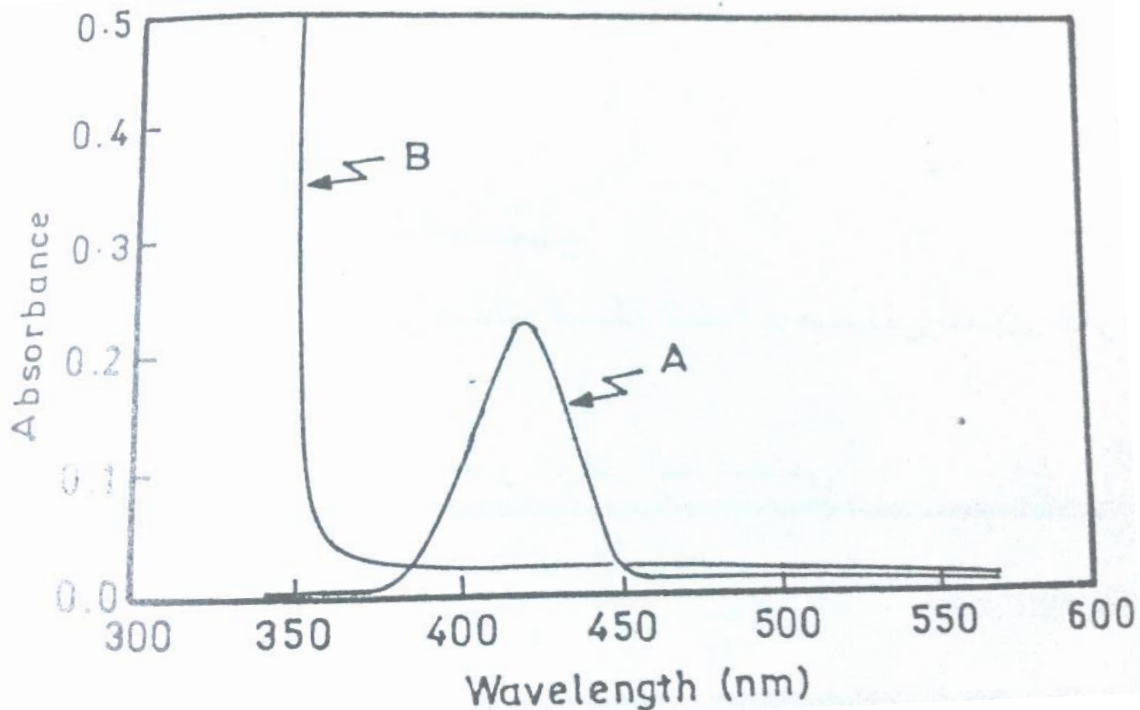


Fig. 1: Final absorption spectrum of 30 µg /25 ml of sulfacetamide measured against (A): reagent blank, (B): reagent blank against distilled water

Standard working method and standard curve

To a series of volumetric bottles (25) ml add (5) ml distilled water to (0.1, 0.3, 0.5, 0.7, 1.0, 1.5, 2.0) ml solution (100) ppm sulfacetamide and then add (4) ml of sulphuric acid (1N)) And (0.7) ml solution (1%) sodium nitrite and leave the solution for (3) minutes to complete the reaction and then add (1.5) ml solution (3%) sulfamic acid and leave the solution for (2) minutes to complete the destruction of the increase Nitrite was then added (1.5) ml of the reagent solution (0.5%) m-aminopheol and then added (7) ml of the solution (1M) sodium acetate then the volume was completed to the extent of the mark with distilled water and then measured absorption immediately

after dilution at the wavelength (436) nm . Figure (2) represents the standard curve, which is in accordance with Beer's law, in the range of concentrations (10-200 µg / 25 ml). the value of molar absorptivity and sandell sensitivity are $1.68 \times 10^4 \text{ l.mol}^{-1}\text{cm}^{-1}$, $1.4 \times 10^{-4} \mu\text{g} / \text{cm}^{-2}$.

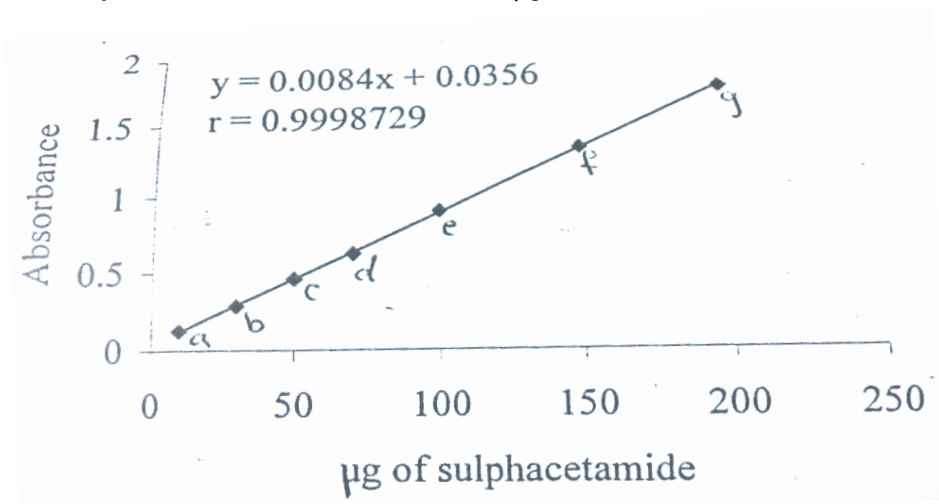


Fig.2. Standard curve for sulfacetamide assay

Accuracy Method

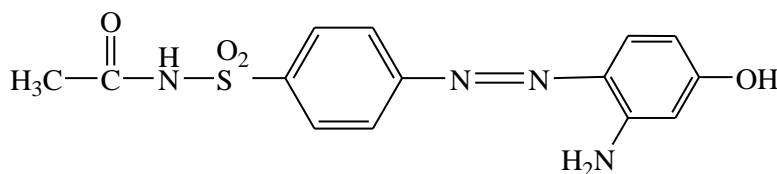
The accuracy of the method of estimating sulfacetamide has been determined by applying the method of work used to find the standard curve for three different concentrations (10, 30, 50, 100) micrograms of sulfacetamide (each of 5 readings) as shown in the following table:

Table 2. Compatibility and accuracy of the proposed method

| µg of sulphacetamide | Er, % | R.S.D% |
|----------------------|-------|--------|
| 10 | 0 | ± 0.8 |
| 30 | 0.3 | ± 0.39 |
| 50 | 0.040 | ± 0.43 |
| 100 | 0.070 | ± 0.28 |

Nature of dye formed

The continuous change method (Job's method) was used to determine the reaction rate of sulfacetamide with the reagent. It was noted that the coupling ratio between sulfacetamide and meta-aminophenol reagent is 1: 1. Accordingly, the proposed wording would be as follows:



Application Method

For the purpose of checking the validity of the method was applied to different samples , the recovery of Samacetamide Drops (SDI) was (93%), while the recovery of Samacetamide Drops (Shifa Drug Factory - Iraq) was 97.

Comparison of method

Table 4 shows the comparison between the analytical variables of the present method with those of other method for sulfacetamide determination

Table 7. Comparison of method

| Analytical parameters | Present method | Literature method[6] |
|--|-----------------------------|-----------------------------|
| Reagent | m-aminophenol | chromotropic acid |
| Medium | Basic | Basic |
| λ_{max} (nm) | 436 | 511.5 |
| Beer's law range ($\mu\text{g} \cdot \text{ml}^{-1}$) | 10-200 | 0.5-20.0 |
| Molar absorptivity ($\text{l} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$) | 1.68×10^4 | 3.2186×10^4 |
| Color of the product | orange | orange |
| Application of the method | Pharmaceutical preparations | Pharmaceutical preparations |

Conclusions

The method is based on the coupling of sulfacetamide with the meta-aminophenol reagent and the formation of a water-soluble colored dye. The proposed method does not require temperature control, use of organic solvents or solvent extraction and has been successfully applied in the determination of sulfacetamide in pharmaceutical preparations.

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