Spectrophotometric Determinations of Sulfacetamide Following Simple Diazotization and Coupling with Chromotropic Acid

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Received at: 27 May 2013, Accepted at 24 September 2013

Abstract

A simple, sensitive, accurate and economic spectrophotometric method has been developed for the determination of sulfacetamide (SFA) in pure form, synthetic sample and urine. The method is based on diazotization of primary amine group of sulfacetamide with sodium nitrite and hydrochloric acid followed by coupling with chromotropic acid in alkaline medium to obtain a stable orange colored chromogen which exhibit a maximum absorption (λ_{max}) at 511.5 nm. Different variables affecting the completion of reaction have been carefully optimized following the classical univariate sequence and modified simplex method (MSM). Under optimized conditions, Beer's law obeyed in the concentration range of (0.5- 20.0 $\mu g.ml^{-1}$) with molar absorptivity of $3.2186*10^4$ $L.mol^{-1}.cm^{-1}$. The limit of detection was 0.054 $\mu g.mL^{-1}$ and Sandell's sensitivity value 7.8989 $\mu g.cm^{-2}$. The proposed method was successfully applied to the determination of (SFA) in synthetic sample and urine.

Key words: Spectrophotometric determination, Sulfacetamide, Diazotization reaction, Coupling reaction, Chromotropic acid.

Introduction

Sulfacetamide (SFA) Figure (l) is a member of the sulfonamide family of antibiotics and chemically it is N-(4-Aminobenzenesulfonyl) acetamide (M.wt.=214.24). Sulfonamides are widely used in the treatment of urinary tract infection, acne vulgaris, bacterial infection of eye, superficial infection including conjnnctivitis, bacterial infection of skin, seborrhea including seborrhoeic dermatitis and seborrhoeic sicca, and trachoma; adjnnct. They are also the drugs of choice for the treatment of nocardiosis toxoplasmosis, severe diarrhea and meningococcal infections[1].

Sulfacetamide has been determined by the development of several analytical techniques such as flow injection analysis [2,3], sequential injection analysis [4], HPLC[5-7], micellar electro kinetic capillary chromatography (MEKC)[8,9], spectrophotometric method [10-14]. The aim of the present work is to provide an optimized spectrophotometric method using the univariate and multivariate simplex method. In the simplex method three-interest factors concentration of sulfamic acid, chromotropic acid, sodium hydroxide were designated as independent variables and absorbance as response.

Experimental

Instruments

The absorption spectra were recorded on a double-beam (shimadzu 1800), and all spectrophotometric measurements were carried out on (CECIL 1011)UV-Visible single beam spectrophotometer with 1cm matched quartz cells.

Materials and reagents

Pharmaceutical grade sulfacetamide received as gift sample powder in pure form (99.99%) the State Company for Drug Industries and Medical Appliances Samara-Iraq (SDI). All chemicals and reagents used were of analytical grade.

Preparation of solutions

Sodium nitrite [0.5 % (wt/v)]: was prepared by dissolving 0.5 g of $NaNO_2$ in distilled water and diluting to l00 mL in a volumetric flask.

Sulfamic acid [2 % (wt/v)]: was prepared by dissolving 2 g of sulfamic acid in 100 mL of distilled water.

Chromotropic acid (CTA) [2 % (wt/v)]: was prepared by dissolving 2 g of CTA in l00 mL of distilled water.

Sodium hydroxide [2 M]: was prepared by dissolving 8 g of NaOH in 100 mL of distilled water.

Hydrochloric acid [5 M]: 85mL of concentrated HCl was taken and diluted to 200 mL with distilled water.

Hydrochloric acid [2 M]: was prepared by taken 16.72 mL of concentrated HCl and diluted to 100 mL with distilled water.

Standard drug preparation (100 µg.mL^{-1})

The standard solution of SFA was prepared by dissolving accurate weighted 0.01 g of pure drug in 100 mL with distilled water.

Preparation of synthetic drug sample

1- To 20 mg of the bulk drug, 5 mg of interfering substances mixture (consisting of equal weights of each substance, namely, glucose, lactose, soluble starch, and vanillin) was added.

2- 12.5 mg of the resulted mixture was dissolved in 100 mL of distilled water in the same manner as used for the preparation standard drug to obtain 100 µg.mL^{-1}.

Preparation of drug solution in urine

Solution of drug in urine was prepared by dissolving 0.01 g of (SFA) in a little amount of distilled water, then the volume was complete to 100 mL urine in volumetric flask to obtain 100 $\mu g.mL^{-1}$ of spiked synthetic sample solution.

General standard procedures

Two procedures were recommended for the determination of SFA via the proposed methods. The first was carried out following the conditions obtained by univariate optimization, while the second base of those conditions was obtained by chemometric multivariate simplex optimization.

Univariate

To aliquots the standard solution (l00 $\mu g.mL^{-1}$) containing (5-150 µg) of SFA were transferred into a series of 10 mL volumetric flasks and cooled in an ice bath, 1.0 mL of 0.05 % (w/v) sodium nitrite solution and 1.0 mL of 0.5 M HCl were added. Each solution was shaken thoroughly, and then 1.5 mL of 0.5 % (w/v) sulfamic acid was added.The solutions were swirled and the resulting diazotized product was coupled with CTA by the addition of 1.0 mL of 1 % (w/v) of this reagent followed by 2.0 mL of 0.05 M sodium hydroxide solution. The mixtures allowed to stand for 10 min., after then were made up to the mark with distilled water. The absorbance of orange colored chomogen was measured at 511.5 nm against the reagent blank. The constructed calibration curve was used to compute the amount of SFA in given samples.

Multivariate (simplex method)

Aliquots of the standard solution (100 $\mu g.mL^{-1}$) containing (5-200 µg) of SFA were transferred into a series of 10 mL volumetric flasks. After cooling in an ice bath, to each flask, l.0 mL of 0.05% (w/v) sodium nitrite solution and l.0 mL of 0.5 M HCl were added. Each solution was shaken thoroughly and l.5 mL of 0.25 % (w/v) sulfamic acid was added. The solutions were swirled and the resulting diazotized product was coupled with CTA by the addition of l.0 mL of 0.5 % (w/v) the reagent solution followed by 2.0 mL of 0.075 M sodium hydroxide solution and allowed to stand for 10 min. The solutions were making up to the mark with distilled water. After mixing the solution well, the absorbance of orange colored chomogen was measured at 511.5 nm against the reagent blank. The amount of SFA was computed from calibration curve.

Result and discussion

Absorption spectra and reaction scheme

Primary aromatic amine upon treatment with nitrous acid in an ice-cooled solution forms diazonium salt.

$Ar\text{-}NH_2 + NaNO_2 + HCl \rightarrow Ar\text{-}N_2^+Cl^- + H_2O$

Under proper conditions, diazonium salts react with certain aromaticcompounds to yield products of general formula Ar-N=N-Ar', called as azo compounds and the reaction is called as diazo coupling reaction [15].

$Ar\text{-}N_2^+ + Ar'H \rightarrow Ar\text{-}N{=}N\text{-}Ar' + H^+$

The proposed method involves coupling reaction of the diazotized sulfacetamide with chromotropic acid in an alkaline medium to yield an azo dye according to Scheme 1. The former orange colored product shows a maximum absorption at 511.5 nm (Figure 2).

Optimization of reaction variables

Various parameters (viz sodium nitrate concentration, hydrochloric acid concentration, time of diazotization reaction, sulfamic acid concentration, chromotropic acid concentration, sodium hydroxide concentration, and time of coupling reaction) were first optimized, for the development of color dye, univariatly by systematic study of the effects of each parameters in

the development of color product. The optimization steps were carried out by varying the parameters each one at a time and controlling all others fixed.

Effect of diazotization reaction time

It was found that the diazotization reaction was taking place instantaneous when the time required for diazotization reaction was studied in the range (0-20) minute at 0 ºC. The diazotized product was found to be decomposed with time therefore, diazotization was carried out instantaneously.

Effect of sodium nitrite

Nitrite concentration for diazotization was optimized in the range of (0.025-0.500 %) (Figure 3). Maximum absorbance was observed when 1.0 mL of 0.050 % sodium nitrite was added and the absorption of dye product was measured at 511.5 nm. Below and above this concentration the absorbance was decreased.

Effect of different acidic solutions

Effect of many acids on diazotization reaction was studied such as H_2SO_4, HCl, HNO_3, CH_3COOH with 1 M of concentration. HCl was responded to the stable diazotized product thus it was used for the following experiments (Table1).

Effect of acidity

It was found that the absorbance of the formed azo dye product was enhanced as the concentration hydrochloric acid was decreased in the studied range of the acid (0.025–2.000 M), Figure 4. Maximum absorption intensity was achieved when 1.0 mL of 0.500 M HCl was used, and this amount was used for the subsequent work.

Effect of sulfamic acid concentration

According to the results obtained upon studying the effect of the addition of 1.5 mL of (0.25–2.00 %) of sulfamic acid on the reaction product, the optimum amount was found to be 1.5 mL of 0.50 % solution (Figure5).

Effect of chromotropic acid concentration

The effect of coupling reagent concentration on the intensity of the color dye development was tested by using different concentrations of CTA in the range (0.1–2.0 %), Figure 6. The result shows that 1.0 mL of 0.7 % of CTA solution was optimum for this method.

Effect of different bases

In order to develop intensity and color of dye product, different bases viz sodium hydroxide, sodium carbonate, potassium hydroxide and ammonium hydroxide were studied. The results indicate that the sodium hydroxide was suitable alkaline medium to give a maximum absorption as shown in (Table 2).

Effect of alkalinity

The stability and formed azo dye product depends upon the nature of reaction medium. The formed azo dye was found to have a reasonable stability when the reaction medium render alkaline via addition of 2.0 mL of 0.1 M sodium hydroxide. Figure 7 illustrates the results of the study.

Effect of coupling reaction time

The maximum time required for coupling reaction to be completed was found to be 10 min. at room temperature. After that period, the absorbance remained constant for at least 3.5 hours as presented in (Table 3).

On the other hand, simplex program was employed to find the optimum experimental conditions of three important parameters that considerably affect the colored product formation (viz the concentration of sulfamic acid, the concentration of CTA, and the concentration of sodium hydroxide) for the determination of (SFA). The boundary conditions for the studied parameters delineated above, were first set as depicted in (Table 4) together with the step size values.

Four (numbers of studied parameters +1) arbitrary experimental conditions were chosen, by selecting the values of these parameters within specified boundaries for each, at which they affected the measured absorptions signal of the colored products (experiment 1-4 in Table 5), the absorbencies of these four experiments together with the applied experimental conditions were fed into a multi simplex computer program. The program run the simplex system to produce a new set of experimental conditions. An experiment is carried out under the new generated conditions and the measured absorption signal was fed again to the program and so on. Only 22 experiments were enough to evaluate the proper conditions that yield the maximum response. Results given in Table 5, and Figure 8 show the progress of the simplex, at step 20 (i.e. 0.25 % sulfamic acid, 0.5 % CTA and 0.075 M sodium hydroxide) the highest response function value was obtained, which was taken to be the optimum conditions for the determination of (SFA).

Calibration curves and analytical data

I- For univariate

According to the optimum experimental conditions, obtained via univariable method, linear calibration graph for sulfacetamide was obtained, Figure 9, which shows that Beer's law is obeyed in the concentration range of (0.5-15.0 $\mu g.mL^{-1}$). The regression equation, correlation coefficient, molar absorptivity, Sandell's sensitivity and detection limit are given in Table 6.

II- For simplex

Table 7 and Figure10 show the results for the calibration curve and statistical data. Better optical characteristics for calibration curve and statistical data were obtained under optimum conditions obtained by multi simplex optimization, in comparison with those obtained via univariate method.

Precision and accuracy

The accuracy (in term of relative error percent) and the precision (in term of coefficient of variation) of the proposed method under univariate and multivariate conditions were evaluated by doing five replicate analyses of SFA at three different levels of concentration within Beer's law range (Table 8). The results indicate good accuracy and precision of the proposed method at the studied concentration levels.

Interference studies

The effect of various foreign species, which may be present in pharmaceutical products and affecting the reaction between the sulfacetamide and CTA, were studied. Optimum experimental conditions, for simplex optimization, were employed to determine 10 $\mu g.ml^{-1}$ concentration of SFA. Table 9 shows that the presence of 1000 $\mu g.mL^{-1}$ of the studied interfering excipient cause errors less than ± 3 %.

Application on synthetic sample

The developed method was applied for the determination of the amount of SFA in its synthetic sample. The results of the application of the proposed method given in Table 10 were satisfactory. The recovery was ranged from (98.9-101.2 %) and the coefficient of variation range was (0.4326-2.0458 %).

Application on spiked urine sample

The proposed method was also applied for the determination of SFA in spiked human urine samples.The results, listed in Table 11; indicate that the proposed method could successfully be applied. The recovery values were ranged (98.4-104.0 %) with the coefficient of variation of (0.4157-1.5678 %).

Application on spiked urine by standard addition method

To increase the insurance, the proposed spectrophotometric method was applied for the determination of SFA in spiked urine sample following the standard addition technique. Good recoveries of the drug present in studied sample indicate that non-interference from urine matrix. Figure 11 shows the standard addition plot and Table 12 shows the result of recovery and coefficient of variation for the method.

Conclusions

Diazotization reaction of primary amine group followed by coupling with chromotropic acid in alkaline medium was found to be a simple, sensitive, accurate and economic spectrophotometric method for quantitative determination of (SFA) in pure form and synthetic samples. The classical univariate and modified simplex method have been used for optimizing the different variable affecting the completion of the reaction. The proposed method offers good linearity and precision.

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Table (1): Effect of different acids on diazotization of 10 µg.mL^{-1} of SFA.

Acidic solution (1M)	Absorbance
H_2SO_4	0.200
HCl	0.253
HNO_3	0.178
CH_3COOH	0.081

Table (2): Effect of different bases.

Base (4M)	Absorbance
sodium hydroxide	0.854
sodium carbonate	0.687
potassium hydroxide	0.609
ammonium hydroxide	0.268

Table (3): Effect of coupling reaction time.

Time (min)	Absorption
0	0.982
5	0.985
10	1.047
15	1.029
20	1.012
210	1.012

Table (4): Boundary of simplex for the studied variables.

Variable	Minimum boundary	maximum boundary	Step size
Conc. of sulfamic acid (%)	0.25	2.0	0.25
Conc. of CTA (%)	0.1	2.0	0.1
Conc. of NaOH (M)	0.025	2	0.025

Table (5): Multivariate experiments (Simplex) for the determination of (10 µg.mL^{-1}) SFA.

Exp. No.	Conc. of sulfamic acid (%)	Conc. of chromotropic acid (%)	Conc. of sodium hydroxide (M)	Abs.
1	0.25	0.2	0.125	1.019
2	0.5	0.7	0.05	1.024
3	1.5	1.2	0.025	0.681
4	2.0	0.1	2.0	0.096
5	0.25	1.3	0.025	1.234
6	0.25	0.6	0.075	1.244
7	0.25	1.4	0.025	1.212
8	0.25	1.2	0.05	1.223
9	0.25	0.4	0.1	1.263
10	0.25	0.1	0.125	0.830
11	0.25	0.1	0.1	0.893
12	0.25	0.9	0.075	1.242
13	0.25	1.0	0.05	1.238
14	0.25	0.1	0.15	0.813
15	0.25	0.9	0.05	1.233
16	0.25	0.4	0.075	1.250
17	0.25	0.1	0.1	0.893
18	0.25	0.7	0.075	1.243
19	0.25	0.3	0.1	1.186
20	0.25	0.5	0.075	1.273
21	0.25	0.3	0.075	1.195
22	0.25	0.6	0.05	1.237

Table (6): Optical characteristics and statistical data for the determination of SFA by univariable method.

Parameter	Value
λ_{max} (nm)	511.5
Color	Orange
Linearity range (µg.mL^{-1})	0.5 – 15.0
Regression equation	A= 0.1255 [SFA. µg.mL^{-1}] - 0.0198
Calibration sensitivity	0.1255
Correlation coefficient (R)	0.9997
Correlation of linearity (R^2)	0.9994
Molar absorptivity (L.mol^{-1}.cm $^{-1}$)	3.1907*10^4
Sandell's sensitivity (µg.cm^{-2})	7.9681
Detection limit (µg.mL^{-1})	0.071

Table (7): Optical characteristics and statistical data for the determination of SFA by simplex method.

Parameter	Value
λ_{max} (nm)	511.5
Color	Orange
Linearity range (µg.mL^{-1})	0.5-20.0
Regression equation	A= 0.1266 [SMX. µg.mL^{-1}] - 0.0282
Calibration sensitivity	0.1266
Correlation coefficient (R)	0.9998
Correlation of linearity (R^2)	0.9996
Molar absorptivity (L.mol^{-1}.cm $^{-1}$)	3.2186*10^4
Sandell's sensitivity (µg.cm^{-2})	7.8989
Detection limit (µg.mL^{-1})	0.054

Table (8): Accuracy and precision of the proposed method.

	Conc. of SFA (µg.mL^{-1})		Relative Error %	C.V
	Taken	Found*		
For univariate conditions	2.00	2.05	2.500	1.395
	7.00	6.97	-0.428	0.536
	15.00	15.06	0.400	0.208
For simplex conditions	2.00	1.95	-2.500	0.834
	7.00	7.04	0.571	0.391
	15.00	14.99	-0.066	0.228

*mean of five replicate.

Table (9): Percent recovery for 10 µg.mL^{-1} of sulfacetamide in the presence of 1000 µg.mL^{-1} of excipients.

Excipients	Concentration of Excipient µg.mL^{-1}	Sulfacetamide Conc. Taken 10 µg.mL^{-1}	
		Conc. Found µg.mL^{-1}	% Recovery
Vanillin	1000	9.70	97.00
Glucose		10.14	101.44
Sucrose		10.11	101.12
Starch		10.22	102.20

Table (10): Application of the proposed method to the SFA concentration measurements in synthetic sample.

Weight of SFA in 25 mg of sample	*Weight (mg) of SFA found in 25 mg of sample	Conc. taken µg.mL^{-1}	*Conc. found µg/mL	Recovery %	C.V*
20	20.24	2	2.024	101.2	2.0458
	20.20	5	5.050	101.0	0.4992
	19.78	10	9.890	98.9	0.4326

*****Average of three determinations.

Table (11): Application of the proposed method to the SFA concentration measurements in spiked urine.

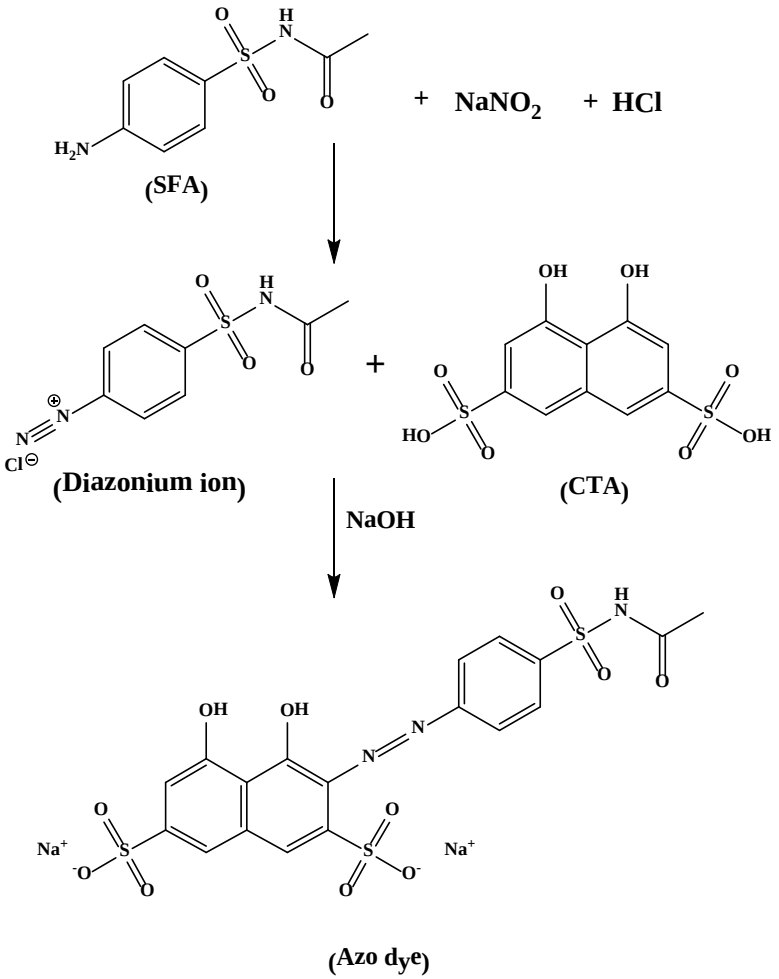
Sample	Conc. taken µg.mL^{-1}	Conc.* found µg.mL^{-1}	Recovery %	C.V*
SFA in urine	2.00	2.08	104.0	1.5678
	5.00	4.92	98.4	0.5891
	10.00	9.87	98.7	0.4157

*Average of three determinations.

Table (12):Application of the proposed method to the SFA concentration measurements in spiked urine by standard addition method.

Sample	Conc. taken µg.mL^{-1}	Conc.* found µg.mL^{-1}	Recovery %	C.V*
SFA in urine	200.00	202.52	101.26	0.8266

*****Average of three determinations.



Scheme (1): The proposed reaction mechanism for diazotization and reaction between SFA and CTA.

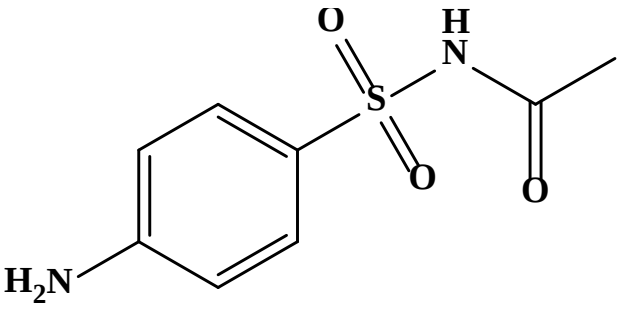


Figure (1): The chemical structure of sulfacetamide.

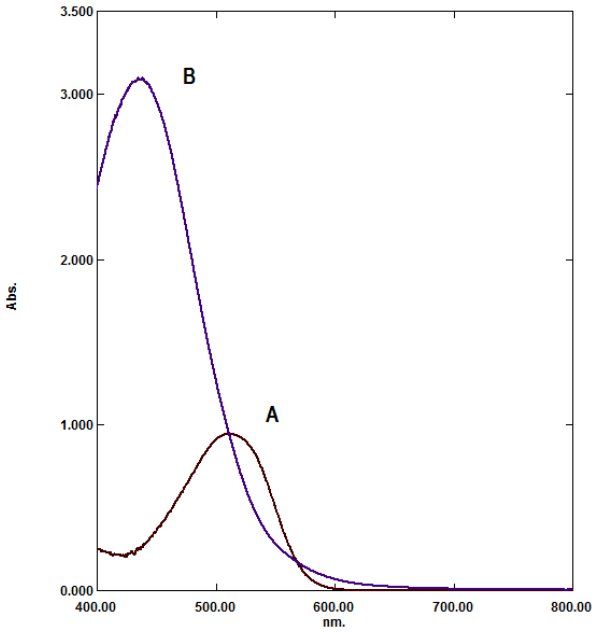


Figure (2): Absorption spectrum of (A) chromogen against (B) reagent blank solution.

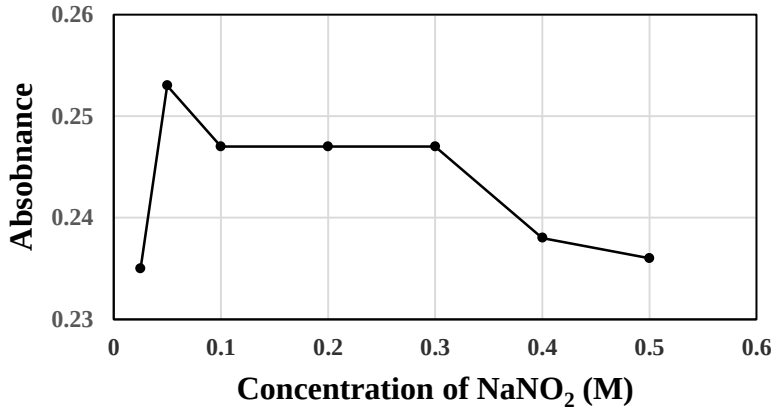


Figure (3): Effect of sodium nitrite on the color development of dye in the determination of chromogen from 10 µg.mL^{-1} of SFA.

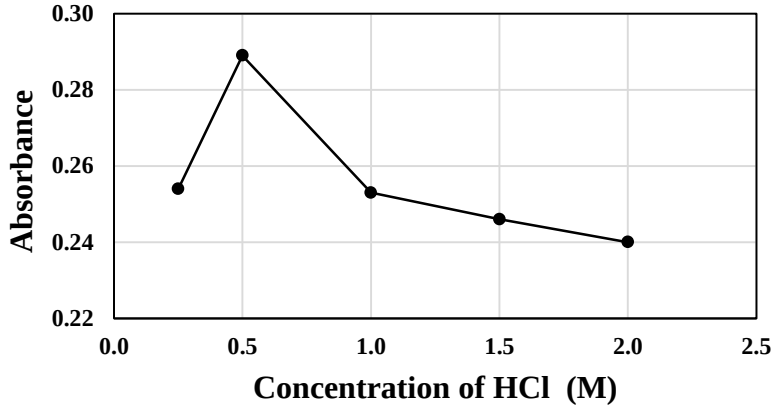


Figure (4): Effect of HCl concentration on the color development of dye in the determination of chromogen from 10 µg.mL^{-1} of SFA.

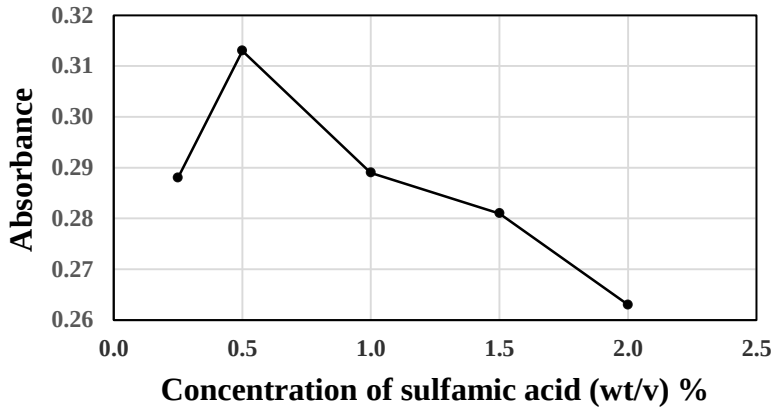


Figure (5): Effect of sulfamic acid concentration on the determination of chromogen from 10 µg.mL^{-1} of SFA.

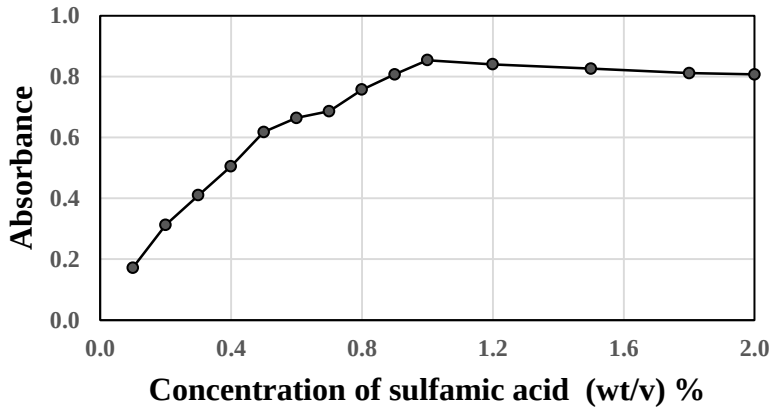


Figure (6): Effect of CTA concentration on the determination of chromogen from 10 µg.mL^{-1} of SFA.

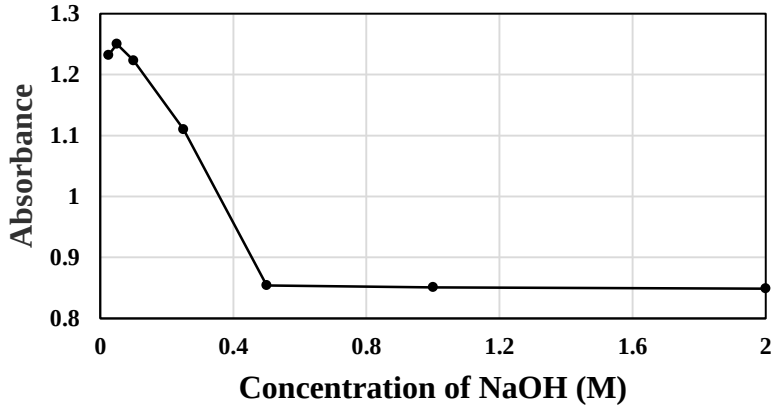


Figure (7): Effect of NaOH concentration on the determination of chromogen from 10 µg.mL^{-1} of SFA.

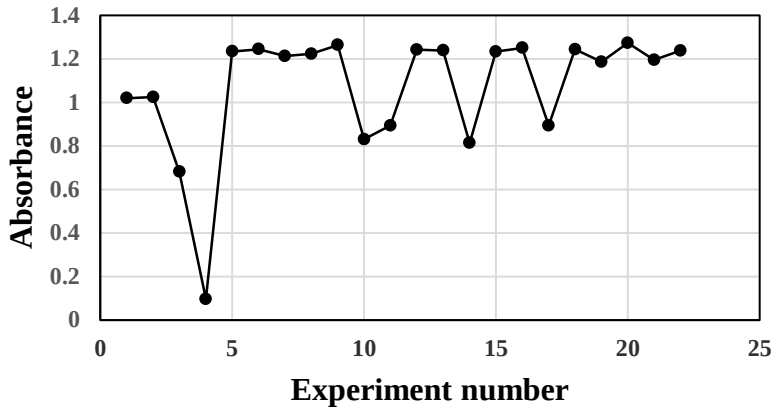


Figure (8): Response function progress for simplex.

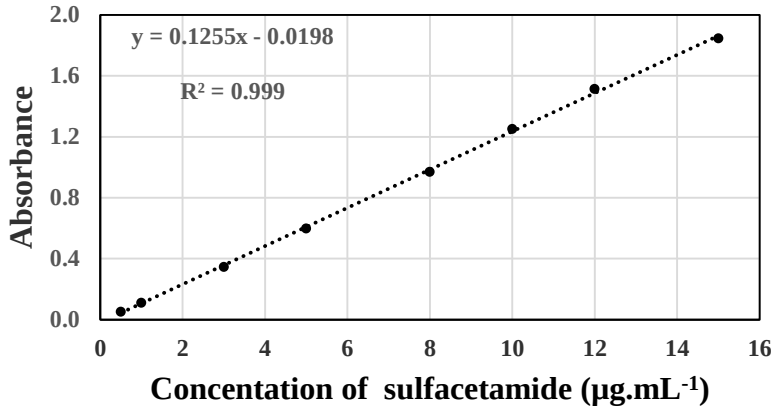


Figure (9): Calibration curve for the determination of SFA under univariate optimal conditions.

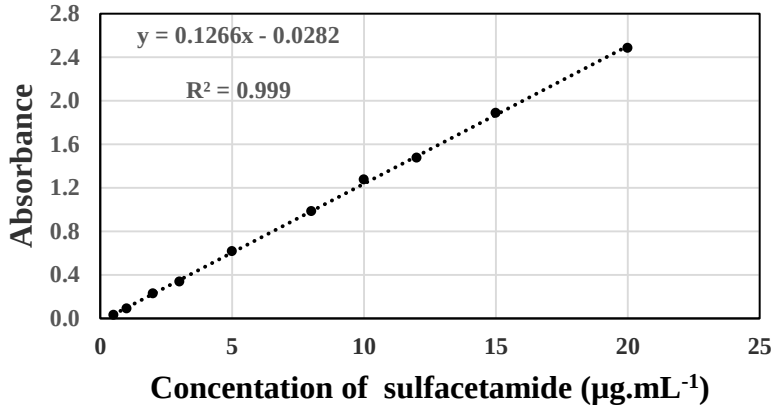


Figure (10): Calibration curve for the determination of SFA under simplex optimal conditions.

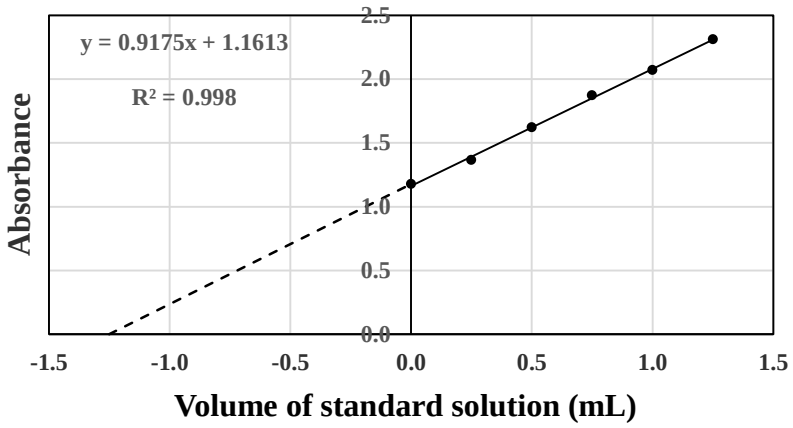


Figure (11): Determination of SFA in spiked urine sample by standard addition method.

التقديرات الطيفية للسلفاسيتمايد المتضمنة الأزوتة البسيطة والأزدواج مع حامض الكروموتروبيك

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أستلم في: 27 آيار 2013، قبل في: 24 أيلول 2013.

الخلاصة

طورت طريقة طيفية بسيطة وحساسة ودقيقة وغير مكلفة للتقدير الكمي للسلفاسيتمايد (SFA) بشكله النقي وفي نماذج مصنعة وفي الأدرار. تعتمد الطريقة على أزوتة مجموعة الأمين الأولي في السلفاسيتمايد بمعاملته مع نتريت الصوديوم وحامض الهيدروكلوريك ثم اجراء تفاعل الازدواج مع حامض الكروموتروبيك في وسط قلوي للحصول على كروموجين مستقر ذي لون برتقالي يظهر أعظم امتصاص (λ_{max}) عند 511,5 نانومتر. وقد تمت دراسة العوامل التي تؤثرفي إتمام التفاعل بعناية للحصول على الظروف الفضلى وذلك باتباع نمط المتغير الواحد وبالاعتماد على طريقة السمبلكس المحورة. عند الظروف الفضلى، وجد أن قانون بير ينطبق على مدى من التراكيز يتراوح بين($0.5– 20.0\ \mu g.mL^{-1}$) مع قيمة الامتصاصية المولارية مساوية لـ $3.2186*10^4\ L.mol^{-1}.cm^{-1}$ وكان حد الكشف يساوي $0.054\ \mu g.mL^{-1}$ ودلالة ساندل يساوي $7.8989\ \mu g.cm^2$. لقد أمكن تطبيق الطريقة المقترحة بنجاح لتقدير السلفاسيتمايد في نماذج مصنعة وكذلك فى الادرار.

الكلمات المفتاحية: التقدير الطيفي، سلفاسيتمايد، تفاعل الأزوتة، تفاعل الأزدواج، حامض الكروموتروبيك.