Simultaneous Determination of Sulfanilamide and Furosemide by Using Derivative Spectrophotometry

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Received in:8/December/2015,Accepted in:1/March/2016

Abstract

A simple, precise and accurate spectrophotometric method has been developed for simultaneous estimation of sulfanilamide and furosemide in their mixture by using first and second order derivative method in the ultraviolet region. The method depends on first and second derivative spectrophotometry, with zero-crossing and peak to base line and peak area measurements. The first derivative amplitudes at 214, 238 and 266 nm were selected for the assay of sulfanilamide and 240, 260, 284, 314 and 352 nm for furosemide. Peak area at 201-222, 222-251 and 251-281 nm selected for estimation of sulfanilamide and at 229-249, 249-270, 270-294, 294-333 and 333-382 nm for furosemide. The second derivative amplitudes at 220, 252 and 274 nm for sulfanilamide and 248, 272, 292, 334, and 364 nm for furosemide. Peak area at 209-229, 239-262 and 262-285 nm for sulfanilamide and at 238-253, 262-281, 281-303, 315-353 and 353-383 nm for furosemide. The first derivative absorption at 270.5 nm (zero cross point of furosemide) was used for determination of sulfanilamide and 322.5 and 352 nm (zero cross point of sulfanilamide) for determination of furosemide. The second derivative absorption at 261 and 283.3 nm (zero cross point of furosemide) was used for determination of sulfanilamide and 266, 334 and 364 nm (zero cross point of sulfanilamide) for determination of furosemide. The linearity was established over the concentration range of 1-35 µg/ml and 1-60 µg/ml for sulfanilamide and furosemide with correlation coefficient R^2 0.9991 and 0.9995 respectively. Accuracy and precision of the determination method on the various amounts of sulfanilamide and furosemide with known concentrations were evaluated in their binary mixtures. The proposed method has been successfully applied to the estimation of sulfanilamide in its synthetic samples and furosemide in its drug tablets.

Keywords: Derivative spectroscopy, Simultaneous determination, Sulfanilamide, Furosemide.

Introduction

Sulfanilamide chemically is 4-amino benzene sulfonamide (Figure1), it is a medicinal compound used to guard against certain bacterial infections. It is frequently used in the form of a topical cream or powder to treat surface infections, as well as a pill for internal infections. It falls into the category of sulfonamide antibacterial drugs, common infections treated by sulfanilamide include urinary tract infections, vaginal infections, strep throat, and some staph infections. Depending on the type of infection, either a cream or a pill will be prescribed [1].

Furosemide chemically is 4-chloro-N-furfuryl-5-sulfamoyl-anthranillic acid, (Figure 2) an effective diuretic, has been widely used in the treatment of chronic renal failure, hypertension, congestive heart failure and cirrhosis of the liver. Furosemide is often classified as a loop diuretic due to its predominant action in the nephron, where the drug interferes with the tubular re-absorption of sodium on Henle's loop. Furosemide acts inhibiting the co-transportation of sodium, potassium and chloride, and further cause´s excretion of calcium, magnesium and bicarbonate ions. Intense and fast dieresis may also mask the ingestion of other doping agents by reducing their concentration in urine [2].

Various methods such as, spectrophotometric [3,4], HPLC [5-7], flow injection [8], ion-selective electrodes [9] have been reported in the literature for the determination of sulfanilamide in pharmaceutical preparations and water samples. On the other hand many publications described for the determination of furosemide include spectrophotometric [10,11], potentiometry [12,13], voltammetry [14], HPLC [15-17], GC [18], TLC [19], flow injection [20], fluorimetry [21,22] in pharmaceutical formulations and biological samples.

The beginning of derivative spectrophotometry is dated on 1953 when the first analogue spectrophotometer was built by Singleton and Cooler [23]. But the fast development of this technique started in 70-s of the twentieth century, when new generation of spectrophotometers controlled by computers were constructed. An apogee of its popularity was occurred in 80-s of last century. Nowadays, it is only additional technique, rarely used, though it is fully available as a build-in function in software of modern spectrophotometers [24].

Derivative spectra can be obtained by optical, electronic, or mathematical methods. Optical and electronic techniques were used in early UV-Vis spectrophotometers, but have largely been superseded by mathematical techniques. The advantages of the mathematical techniques are that derivative spectra may be easily calculated and recalculated with different parameters, and smoothing techniques may be used to improve the signal-to-noise ratio [25,26]. A narrowing of new signals is observed during the generation of consecutive derivative spectra. This feature leads to narrowing bands and as a consequence to separate the overlapped peaks [24] and allow the assay of certain analytes from complex mixtures or matrices via mathematical interpretation of the absorption signal [27].

The purpose of the present study was to investigate the utility of derivative spectrophotometry in the assay of sulfanilamide in its synthetic samples and furosemide in its drug tablets without the necessity of sample pre-treatment.

Experimental

Instruments

U.V.-Visible double beam spectrophotometer with 10 mm quartz cell shimadzu 1800, Windows 7 computer (DELL).

Chemicals and reagents

Pharmaceutical grade sulfanilamide and furosemide powder received in pure form (99.99%) were provided as a gift from the State Company for Drug Industries and Medical Appliances Samara-Iraq (SDI), sodium hydroxide (99.9 %) was provided by Panreac . All chemicals used were of analytical grade.

Preparation of standard sulfanilamide and furosemide stock solution (100 µg.mL^{-1})

The standard solution of sulfanilamide and furosemide were prepared by dissolving accurate weighted 10 mg of pure drug in 1-2 mL of 1 M sodium hydroxide and further diluted to 100 mL with distilled water.

Preparation of synthetic sulfanilamide sample

1- To 20 mg of the bulk drug, 5 mg of interfering substance mixture (consisting of equal weights of each substance, namely, glucose, fructose, lactose, soluble starch and sucrose) were added.
2- 12.5 mg of the resulted mixture was dissolved in in 1-2 mL of 1 M sodium hydroxide and further diluted to 100 mL with distilled water to obtain 100 µg.mL^{-1}.

Preparation of furosemide from drug tablets

The content of 10 tablets was grinded and mixed well. A 10 mg of the fine powder was accurately weighted to and dissolve in 1-2 mL of 1 M sodium hydroxide then diluted to 100 mL in a volumetric flask with distilled water to obtain 100 µg.mL^{-1}. The solution was filtered by using Whatman filter paper No.41 to avoid any suspended or un-dissolved material before use, and the first portion of the filtrate was rejected.

General procedures

Assay procedure for the determination sulfanilamide or furosemide

1.0 mL aliquots, of sulfanilamide standard solution containing 10-350 µg (or furosemide standard solution containing 10-600 µg), were transferred into a series of 10 mL volumetric flask and diluted with distilled water. The spectrum for each solution was recorded against a distilled water as blank. Zero order spectrum was then manipulated for each to get its first derivative (D1) and second derivative (D2).

Assay procedure for the determination each drug in the presence of the other

1.0 mL aliquots, of sulfanilamide standard solution containing 10-350 µg (or furosemide standard solution containing 10-600 µg), were transferred into a series of 10 mL volumetric flask containing 1.0 mL of 10 µg of furosemide solution (or 1.0 mL of 50 µg of sulfanilamide solution); the mixture was then diluted with distilled water. The spectrum for each solution was recorded against a distilled water as a blank. The recorded spectra were then manipulated to get D1 and D2.

Results and discussion

Absorption spectra

The absorption spectra of sulfanilamide, furosemide and their mixture were recorded against distilled water as a blank. The absorption spectrum of sulfanilamide which has maximum wavelength of absorption at 252 nm, and the absorption spectrum of furosemide which appears absorption maxima at 272.5 nm, in addition to the absorption spectrum of mixture of two drugs which show a maximum wavelength of absorption at 270 nm which is related to the absorption maxima of the two compounds. Figure (3) shows the absorption spectrum of sulfanilamide and furosemide and theirs mixture.

First and second derivative modes

Sulfanilamide and furosemide are shown broad and overlap spectrum when they are present in the same solution, therefore; they cannot determine using zero order absorption measurements. For this reason, the derivative spectroscopy method applied has the advantage that it locates hidden peak in the normal spectrum. First and second order are demonstrated to be adequate because they allow high amplitude and good spectra profile. The first and second order derivative spectra of sulfanilamide and furosemide and their mixture are shown in Figures (4 and 5) respectively.

Calibration curves for sulfanilamide

In order to determine the values of derivative spectra, three graphical techniques: peak to baseline (peak height), area under peak and zero crossing have been used via UV-spectrophotometric method for qualitative analyses of sulfanilamide individually and in its mixture with furosemide.

The calibration curves were constructed by plotting the graphically measured (nm) amplitudes of the first and second order derivatives spectra vs. the corresponding concentrations of the examined drugs. Figure 6 shows first order spectra for sets of solutions containing various amounts of sulfanilamide (1-35 $\mu g.mL^{-1}$) in the presence of (1 $\mu g.mL^{-1}$) of furosemide.

In the first derivative techniques, the results indicated that when the concentration of furosemide is kept constant and the concentration of sulfanilamide varied, the peak amplitudes measured at peak to baseline, zero cross, area under peak were found to be in proportion to the sulfanilamide concentration.

For the second derivative technique, the spectra recorded for the previous mixtures of sulfanilamide and furosemide and the results of peak to baseline (peak height), zero cross and peak area at the specified wavelength are used to determine the exact concentration of sulfanilamide in the presence of furosemide. Figure (7) shows the measured second derivative spectra of mixture of the examined drugs. Table 1 summarizes all the results for sulfanilamide analysis by using first and second derivative technique.

Calibration curves for furosemide

Under the experimental conditions described, the graph obtained for UV, first and second derivative spectra showed linear relationship when calibration curves of furosemide were plotted. Peak to baseline, zero cross and area under peak techniques are also used for quantitative determination.

Figure 8 shows the recorded spectra using first derivative UV spectrophotometry for solutions containing (1-60 $\mu g.mL^{-1}$) furosemide with (5 $\mu g.mL^{-1}$) of sulfanilamide. Mixture of furosemide (1-60 $\mu g.mL^{-1}$) and (5 $\mu g.mL^{-1}$) sulfanilamide of UV second derivetive spectra are obtained and presented in Figure(9). The results calculated for the determination of furosemide by proposed methods are listed in Table (2).

Accuracy and precision

To study the accuracy of the proposed method, the relative error percentage was carried out for five replicate analyses of two different amounts of each of the examined drug (with Beer's law). The techniques of derivative (peak to baseline, zero cross and area under the peak) were selected to deal with the recorded spectra. To determine the precision of the method, two drug solutions at studied concentration levels were analyzed each five times for both first and second order derivative spectrophotometric method, and percent coefficient of variation was calculated, Table (3) shows all results.

Interferences study

To check the interference from excipients may be used in the dosage forms, percentage recovery were calculated. This study was performed by addition of known amounts of excipients to mixture solutions of two examined drugs. First and second derivative techniques are used at the selected wavelength for the concentrations of drugs measurement. High recovery showed that no interferences were found using first and second derivative mode for the determination of sulfanilamide and furosemide in their mixture even in the presence of the excipients added, these results are listed in Table (4).

Application in synthetic sample and in tablet

In order to evaluate the efficiency of the derivative technique in the determination of sulfanilamide and furosemide drug, D1 and D2 procedures used for the applications of sulfanilamide in synthetic sample and furosemide in its Tablet. Good recovery % and C.V% values indicated the suitability of these methods for routine analysis of sulfanilamide and furosemide. The summary of the results is depicted in Table (5).

Conclusion

Derivative Spectrophotometric method was found to be rapid, simple, economical, and sensitive. It can be used in routine analysis of sulfanilamide and furosemide in their pure forms, synthetic samples and drug tablets without prior separation or treatment.

Reference

1. Kent, M. (2000) “Advanced Biology”, Oxford University Press, 46.
2. Espinosa Bosch, M.; Ruiz Sánchez, A.J.; Sánchez Rojas, F. and Bosch Ojeda, C. (2013) “Analytical Determination of Furosemide: The Last Reserches” IJPBS, 3:168-181.
3. Betageri, S.; Kulkarni, M.; Shivaprasad, K. H. and Shivshankar, M. (2011) “Kinetic spectrophotometric determination of Sulfa drugs in Pharmaceutical formulations”, Der. Pharma. Chemica, 3(2):227-235.
4. Klokova, E. V. and Dmitrienko, S. G. (2008) “Spectrophotometric determination of sulfanilamides by a condensation reaction with p-dimethyl amino cinnamaldehyde” Moscow University Chemistry Bulletin 63:284-287.
5. Herrera-Herrera, A. V.; Hernandez-Borges, J.; Afonso, M. M.; Palenzuela, J. A. and Rodriguez-Delgado, M. A. (2013) “Comparison between magnetic and nonmagnetic multi-walled carbon nanotubes-dispersive solid-phase extraction combined with ultra-high performance liquid chromatography for the determination of sulfonamide antibiotics in water samples” Talanta 116:695-703.
6. Waleed, M. M.; Khaleel, N.D.H.; Hadad, G.M.; Abdel-Salam, R.A.; Haiss, A. and Kummerer, K. (2013) “Simultaneous Determination of 11 Sulfonamides by HPLC-UV and Application for Fast Screening of Their Aerobic Elimination and Bio-degradation in a Simple Test” Clean-Soil Air Water 41(9):(907-916).
7. Shaaban, H. and Go´recki, T. (2011) "Optimization and validation of a fast Ultrahigh pressure liquid chromatographic method for simultaneous determination of selected sulfonamides in water samples using a fully porous sub-2 µm column at elevated temperature " J. Sep. Sci. 35:216-224.

8. Icardo, M. C.; Mateo, J. V. G.; Lozano, M. F. and Calatayud, J. M. (2003) "Enhanced flow injection chemiluminometric determination of sulfonamides by on-line photochemical reaction" Analytica Chimica Acta 499:57-69.
9. Kharitonov, S. V. and Gorelov, I. P. (2000) "Ion-Selective Electrodes for Determination of Some Sulfanilamide Drugs" Pharmaceutical Chemistry Journal 34:673-676.
10. Semaana, F. S. and Cavalheiro, É. T. G. (2006) "Spectrophotometric Determination of Furosemide Based on Its Complexation with Fe(III) in Ethanolic Medium Using a Flow Injection Procedure" Analytical Letters 39:2557-2567.
11. Naveed, S.; Qamar, F. and Zainab, S. (2014) "Simple UV spectrophotometric assay of Furosemide" JIPBS 1 (3):97-101.
12. Semaan, F. S.; Pinto, E. M.; Cavalheiro, E´. T. G.; and Brettb, C. M. A. (2008) "A Graphite-Polyurethane Composite Electrode for the Analysis of Furosemide" Electroanalysis 20(21):2287-2293.
13. Santini, A. O.; Pezza, H. R.; Sequinel, R.; Rufino, J. L. and Pezza, L. (2009) "Potentiometric Sensor for Furosemide Determination in Pharmaceuticals, Urine, Blood Serum and Bovine Milk" J. Braz. Chem. Soc. 20(1):64-73.
14. Shetti, N. P.; Sampangi, L. V.; Hegde, R. N. and Nandibewoor, S. T. (2009) "Electrochemical Oxidation of Loop Diuretic Furosemide at Gold Electrode and its Analytical Applications" Int. J. Electrochem. Sci., 4:104-121.
15. Kher, G.; Ram, V.; Kher, M. and Joshi, H. (2013) "Development and Validation of a HPTLC Method for Simultaneous Determination of Furosemide and Spironolactone in Its Tablet Formulation" RJPBCS 4:365-377.
16. Rao, B. U. and Nikalje, A. P. (2012) "Determination of Furosemide and Zonisamide as a Drug Substance and in Dosage Form by Ion Pair –Reversed Phase Liquid Chromatographic Technique" Journal of Applied Pharmaceutical Science 2(5):94-99.
17. Patel, H. and Solanki, S. (2012) "Simultaneous Estemation of Furosemide and Spironolactone in Combined Pharmaceutical Dosage Form by RP-HPLC" Asian J Pharm Clin Res 5:195-198.
18. Zaporozhets, O.; Tsyrulneva, I. and Ischenko, M. (2012) "Determination of 8 Diuretics and Probenecid in Human Urine by Gas Chromatography-Mass Spectrometry: Confirmation Procedure" American Journal of Analytical Chemistry 3:320-327.
19. Wesley-Hadzija, B. and Mattocks A.M. (1982) "Thin-layer chromatographic determination of furosemide and 4-chloro-5-sulfamoyl anthranilic acid in plasma and urine" Journal of Chromatography B 229:425-432.
20. Semaan, F. S.; de Sousa, R. A. and Cavalheiro, É. T. G. (2005) "Flow Injection Spectrophotometric Determination of Furosemide in Pharmaceuticals by the Bleaching of a Permanganate Carrier Solution" J. Flow Injection Anal. 22(1):34-37.
21. Liu, Y.; Wang, H.; Wang, J. and Li, Y. (2013) "A simple and sensitive spectro-fluorimetric method for the determination of furosemide using zinc(II)- 1,4-bis(imidazol-1-ylmethyl)benzene complexes" Luminescence 28(6):882-887.
22. Semaana, F. S.; Nogueiraa, P. A. and Cavalheiro, É. T. G. (2008) "Flow Based Fluorimetric Determination of Furosemide in Pharmaceutical Formulations and Biological Samples: Use of Micelar Media to Improve Sensitivity" Analytical Letters 41:66-79.
23. Talsky, G. (1994) "Derivative Spectrophotometry" 1st edition., VCH, Weinheim.

24. Karpinska, J. (2012), "Basic Principles and Analytical Application of Derivative Spectrophotometry" Macro to nano spectroscopy, book edited by Jamal Uddin 253-256.
25. Owen, A. J. (1995) "Uses of Derivative Spectroscopy" UV-Visible Spectroscopy, Agilent Technologies.
26. Owen, T. (2000) "Obtaining derivative spectra" Fundamentals of modern UV-visible spectroscopy, Agilent Technologies 8.
27. Lakiss, H.; Ilie, M.; Baconi, D. L. and Bălălău, D. (2012) "Derivative UV Spectrophotometry Used for The Assay of Diazepam from Human Blood Plasma" FARMACIA 60(4):565-570.

Table (1): Statistical analysis for the determination of sulfanilamide using first and second derivative spectrophotometric technique.

Order of derivative	Mode of calculation	λ (nm)	Regression equation	R^2
First	Peak to base line	214	Y= 0.0082x+0.0012	0.9954
	Peak to base line	238	Y=0.0042x+0.0109	0.9991
	Peak to base line	266	Y= 0.0042x+0.0059	0.9993
	Zero cross	270.5	Y= 0.0039x-0.0055	0.9999
	Peak area	201-222	Y= -0.0935x-0.4170	0.9541
	Peak area	222-251	Y= 0.0771x-0.1971	0.9992
	Peak area	251-281	Y= -0.0680x+0.0336	0.9990
Order of derivative	**Mode of calculation**	**λ (nm)**	**Regression equation**	**R^2**
Second	Peak to base line	220	Y= 0.0011x-0.0006	0.9995
	Peak to base line	252	Y= 0.0005x+0.0001	0.9960
	Peak to base line	274	Y=0.0003x-0.0011	0.9991
	Zero cross	261	Y= 0.0002x+0.0003	0.9963
	Zero cross	283.3	Y=9E-05x-1E-05	0.9988
	Peak area	209-229	Y= 0.021x-0.0786	0.9803
	Peak area	239-262	Y= -0.0085x+0.0111	0.9978
	Peak area	262-285	Y= 0.0038x-0.0129	0.9951

Table (2): Statistical analysis for the determination of furosemide using first and second derivative spectrophotometric technique.

Order of derivative	Mode of calculation	nm (λ)	Regression equation	R^2
First	Peak to base line	240	Y=0.0046x+0.023	0.9824
	Peak to base line	260	Y=0.002x-0.0075	0.9997
	Peak to base line	284	Y=0.0035x+0.0167	0.9995
	Peak to base line	314	Y=0.0003x+0.0014	0.9991
	Peak to base line	352	Y=0.0004x-0.0001	0.9993
	Zero cross	322.5	Y=0.0003x+0.0007	0.9992
	Zero cross	352	Y=0.0004x-0.0001	0.9993
	Peak area	229-249	Y=-0.0565x-0.287	0.9866
	Peak area	249-270	Y=0.0249x-0.0872	0.9993
	Peak area	270-294	Y=-0.0461x-0.1651	0.9991
	Peak area	294-333	Y=0.0091x+0.1612	0.9992
	Peak area	333-382	Y=-0.0119x-0.0383	0.9999
Order of derivative	Mode of calculation	nm (λ)	Regression equation	R^2
Second	Peak to base line	248	Y=0.0006x+0.004	0.9982
	Peak to base line	272	Y=0.0005x+0.0009	0.9994
	Peak to base line	292	Y=0.0004x+0.0008	0.9997
	Peak to base line	334	Y=4E-05x+0.0002	0.9998
	Peak to base line	364	Y=3E-05x+0.0001	0.9994
	Zero cross	266	Y=3E-05x+8E-05	0.9991
	Zero cross	334	Y=4E-05x+0.0002	0.9998
	Zero cross	364	Y=3E-05x+0.0001	0.9994
	Peak area	238-253	Y=0.0066x+0.0126	0.9842
	Peak area	262-281	Y=-0.0074x-0.002	0.9981
	Peak area	281-303	Y=0.0011x+0.0293	0.9995
	Peak area	315-353	Y=-0.0006x-0.0059	0.9993
	Peak area	353-383	Y=0.0004x+0.0004	0.9992

Table (3): Evaluation of accuracy and precision for the determination of sulfanilamide and furosemide by derivative technique.

Drug	Order of derivative	Mode of analysis	λ(nm)	Drug conc. (μg.mL^{-1})		RE %	C.V%
				Taken	*Found		
SFM	First	Peak to baseline	238.0	10.000	10.087	0.870	1.190
				25.000	25.068	0.272	0.182
		Zero cross	270.5	10.000	10.100	1.000	0.629
				25.000	24.790	-0.840	0.344
		Peak area	222.0-251.0	10.000	9.883	-1.170	0.486
				25.000	24.873	-0.508	0.307
	Second	Peak to baseline	252,0	10.000	9.714	-2.860	0.559
				25.000	24.969	-0.124	0.258
		Zero cross	283.3	10.000	10.072	0.720	0.836
				25.000	24.832	-0.672	0.475
		Peak area	239.0-262.0	10.000	9.800	-2.000	0.789
				25.000	25.075	0.300	0.348
Drug	**Order of derivative**	**Mode of analysis**	**λ(nm)**	**Drug conc. (μg.mL^{-1}) Taken**	***Found**	**RE %**	**C.V%**
FUM	First	Peak to baseline	260.0	20.000	20.072	0.360	0.201
				40.000	40.060	0.150	0.078
		Zero cross	352.0	20.000	19.830	-0.850	0.506
				40.000	39.855	-0.363	0.127
		Peak area	249.0-270.0	20.000	20.081	0.400	0.387
				40.000	40.100	0.250	0.095
	Second	Peak to baseline	292.0	20.000	19.947	-0.260	0.603
				40.000	40.084	0.210	0.219
		Zero cross	334.0	20.000	20.095	0.475	0.294
				40.000	40.125	0.313	0.208
		Peak area	281.0-303.0	20.000	19.975	-0.120	0.437
				40.000	39.920	-0.200	0.109

Table (4): Percent recovery for the mixtures of sulfanilamide and furosemide in the presence of 1000 µg.mL^{-1} of excipients.

Excipients	Mixture of 20 µg.mL^{-1} of SFM with 5 µg.mL^{-1} of FUM		Mixture of 30 µg.mL^{-1} of FUM with 7 µg.mL^{-1} of SFM	
	Conc. found* of SFM (µg.mL^{-1})	Recovery %	Conc. found* of FUM (µg.mL^{-1})	Recovery %
Glucose	20.074	100.37	30.200	100.67
Fructose	19.980	99.90	30.098	100.33
Lactose	19.790	98.95	29.580	98.60
Starch	20.092	100.46	30.000	100.00
Sucrose	20.150	100.75	30.342	101.14

*Average of three determinations.
* D1 and D2 for sulfanilamide peak to baseline at 238nm and 252nm respectively.
* D1 and D2 for furosemide peak to baseline at 260nm and 292nm respectively.

Table (5): Application of D1 and D2 spectrophotometric techniques for the determination of sulfanilamide (taken 10 and 25 µg.mL^{-1}) and furosemide (taken 20 and 40 µg.mL^{-1}) in synthetic sample and in tablet.

Sample	Order	Mode of analysis	λ(nm)	SFM amount (mg)		Rec.%	*C.V%
				Taken	Found		
Synthetic sample of (SFM)	First	Zero cross	270.5	20.00	20.14	100.70	0.510
	Second	Zero cross	261.0	20.00	19.60	98.00	0.442
		Zero cross	283.3	20.00	20.32	101.60	0.675
Sample	**Order**	**Mode of analysis**	**λ(nm)**	**FUM amount (mg)**		**Rec.%**	***C.V%**
				Taken	**Found**		
(FUM) in Tablet	First	Zero cross	322.5	40.00	39.60	99.00	0.259
		Zero cross	352.0	40.00	39.75	99.83	0.319
	Second	Zero cross	266.0	40.00	40.50	101.25	0.504
		Zero cross	334.0	40.00	39.41	98.53	0.427
		Zero cross	364.0	40.00	40.63	101.58	0.362

*Average of three determinations.

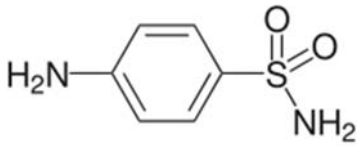

Figure (1): The chemical structure of sulfanilamide.

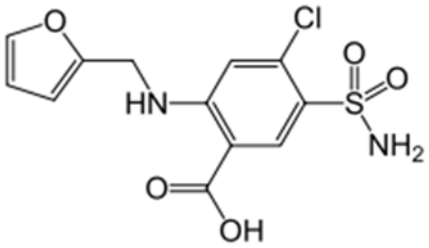

Figure (2):The chemical structure of furosemide.

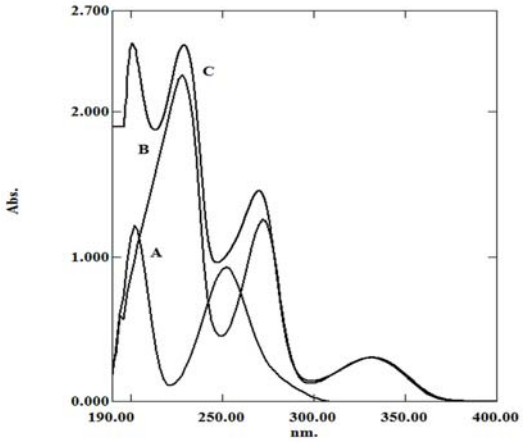

Figure (3): Absorption spectra of: (A) 10 µg.mL^{-1} sulfanilamide, (B) 20 µg.mL^{-1} furosemide and (C) a mixture of 10 µg.mL^{-1} sulfanilamide and 20 µg.mL^{-1} furosemide.

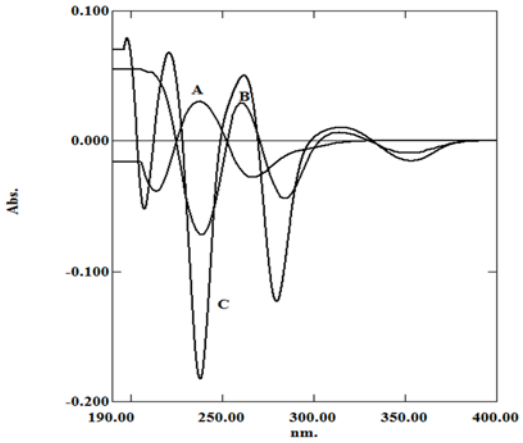

Figure (4): First derivative spectra of (A) 10 µg.mL^{-1} sulfanilamide, (B) 20 µg.mL^{-1} furosemide and (C) a mixture of 10 µg.mL^{-1} sulfanilamide and 20 µg.mL^{-1} furosemide.

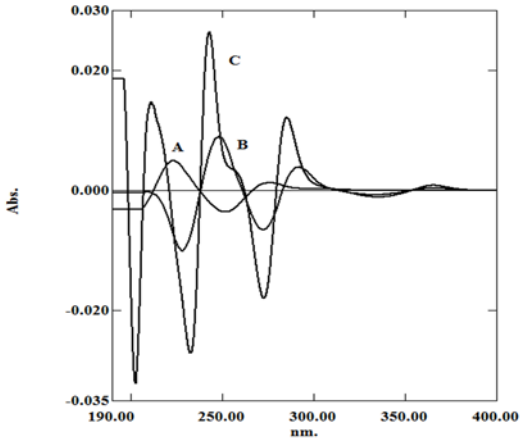

Figure (5): Second derivative spectra of (A) 10 µg.mL^{-1} sulfanilamide, (B) 20 µg.mL^{-1} furosemide and (C) a mixture of 10 µg.mL^{-1} sulfanilamide and 20 µg.mL^{-1} furosemide.

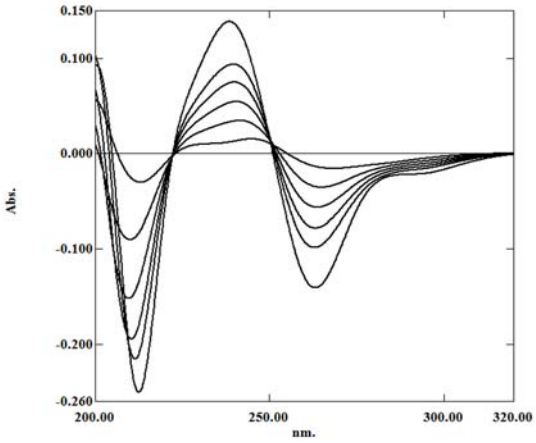

Figure (6): First derivative spectra of mixture contain (1-35 µg.mL^{-1}) sulfanilamide in the presence of (1 µg.mL^{-1}) furosemide.

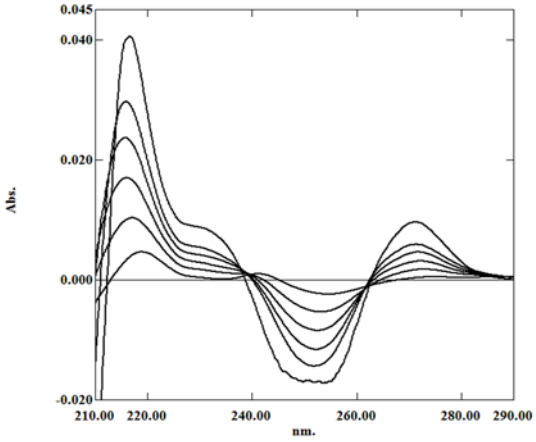

Figure (7): Second derivative spectra of mixture contain (1-35 µg.mL^{-1}) sulfanilamide in the presence of (1 µg.mL^{-1}) furosemide.

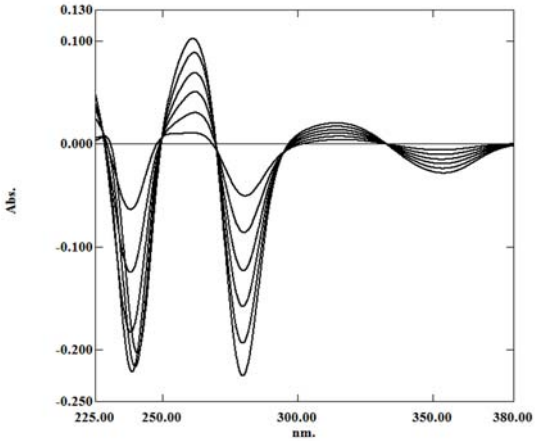

Figure (8): First derivative spectra of mixture contain (1-60 µg.mL^{-1}) furosemide in the presence of (5 µg.mL^{-1}) sulfanilamide.

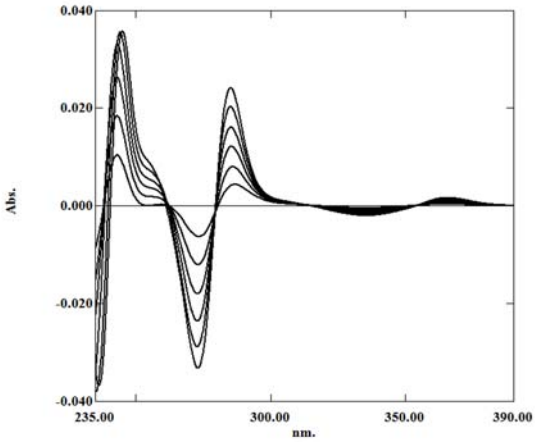

Figure (9): Second derivative spectra of mixture contain (1-60 µg.mL^{-1}) furosemide in the presence of (5 µg.mL^{-1}) sulfanilamide.

التقدير الأني للسلفانيل أمايد والفروسيمايد بأستعمال مطيافية المشتقة

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استلم في:8/كانون الأول/2015،قبل في:1/اذار/2016

الخلاصة

طورت طريقة طيفية بسيطة, متوافقة ودقيقة للتقدير الأني لعقاري السلفانيل أمايد والفروسيمايد في مزيجهما باستعمال طريقة المشتقة الاولى والثانية في المنطقة فوق البنفسجية. أعتمدت الطريقة على المشتقة الاولى والثانية بقياسات التقاطع الصفري وارتفاع القمة ومساحة القمة. قيم ارتفاع القمة للمشتقة الاولى عند 214 و 238 و266 نانومتراختيرت لتقدير السلفانيل أمايد و 240 و 260 و 284 و 314 و 352 نانومتر للفروسيمايد. مساحة القمة عند 201-222 و 222-251 و 251-281 نانومتراختيرت لتقدير السلفانيل أمايد وعند 229-249 و 249-270 و 270-294 و294-333 و 333-382 نانومترللفروسيمايد. قيم ارتفاع القمة للمشتقة الثانية عند 220 و252 و274 نانومترللسلفانيل أمايد و 248 و272 و292 و 334 و364 نانومترللفروسيمايد. مساحة القمة 209-229 و 239-262 و 262-285 نانومترللسلفانيل أمايد وعند 238-253 و262-281 و 281-303 و 315-353 و 353-383 نانومترللفروسيمايد. امتصاص المشتقة الاولى عند 270.5 نانومتر (نقطة تقاطع الصفر للفروسيمايد) استعملت لتقدير السلفانيل أمايد وعند 322.5 و 352 نانومتر (نقطة تقاطع الصفر للسلفانيل أمايد) استعملت لتقدير الفروسيمايد. امتصاص المشتقة الثانية عند 261 و383.3 خنانومتر (نقطة تقاطع الصفر للفروسيمايد) استعملت لتقدير السلفانيل أمايد وعند 266 و 334 و 364 نانومتر (نقطة تقاطع الصفر للسلفانيل أمايد) استعملت لتقديرالفروسيمايد. الخطية المطبقة ضمن المدى 1-35 مايكروغرام/مللترو1-60 مايكروغرام/ مللتر للسلفانيل أمايد والفروسيمايد بمعامل أرتباط 0.9991 و0.9995 على التوالي. قدرت الدقة والتوافق لطريقة التقديرعلى كميات مختلفة من السلفانيل أمايد والفروسيمايد بتراكيز معلومة في المزائج الثنائية. طبقت الطريقة المقترحة بنجاح لتقدير السلفانيل أمايد في نماذجه المصنعة والفروسيمايد في اقراصه الدوائية.

الكلمات المفتاحية: مطيافية المشتقة، التقدير الأني، سلفانيل أمايد، فروسيمايد.