FLOW INJECTION CHEMILUMINESCENCE DETERMINATION OF ACETYLSALICYLIC ACID IN PHARMACEUTICAL FORMULATIONS

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ABSTRACT:

A simple and sensitive flow injection chemiluminescence method was proposed for the determination of acetylsalicylic acid (aspirin) in pharmaceutical formulations. The method was based on inhibition effect of acetylsalicylic acid on the chemiluminescence reaction of luminol–hydrogen peroxide and potassium hexacyanoferrate. Different experiment parameters affecting the chemiluminescence intensity were carefully studied and incorporated into the procedure. The detection limit was 0.5 µg/mL of acetylsalicylic acid, and chemiluminescence emission intensity was correlated with the drug concentration in the range 2.5-125 µg/mL with correlation coefficient of 0.9985 and analytical frequency of 75 determinations per hour. The results obtained for the assay of pharmaceutical formulations were compared well with those obtained by the official method in British Pharmacopoeia and demonstrated good accuracy and precision.

KEYWORDS: *Flow Injection, Chemiluminescence, Acetylsalicylic Acid, Luminol-Hydrogen Peroxide, Potassium Hexacyanoferrate, Pharmaceuticals*

INTRODUCTION

Aspirin (acetylsalicylic acid, ASA, Fig. 1) is the most widely used nonsteroidal anti-inflammatory drug (NSAID) which has been prescribed for over 100 years because of its analgesic, antipyretic and anti-inflammatory properties (Singh & Triadafilopoulos, 1999). Taking a daily dose of ASA would reduce the risk of heart attack and stroke as well as many age associated diseases (Collaboration, 2002). ASA irreversibly inhibits cyclooxygenase enzyme (COX) or prostaglandin endoperoxide synthase (PGHS) by acetylating a serine residue and places a bulky substituent on serine oxygen, which inhibits binding of arachidonic acid (Marjan, Hamzeh, Rahman, & Sadeq, 2014; Roth & Majerus, 1975).

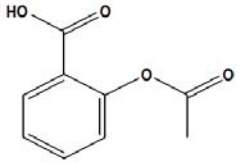

Figure (1): Chemical structure of acetylsalicylic acid

The toxicity of aspirin is multifactorial, including gastrointestinal toxicity, acid–base disturbances, and central nervous system effects. Treatment of aspirin toxicity involves aggressive supportive care, judicious use of activated charcoal, alkalinization with sodium bicarbonate, and potentially hemodialysis (Gorodetsky, 2014).

The techniques described for the determination of ASA in pharmaceuticals formulations are many and varied. These include liquid chromatography (Boixa et al., 2015; Malisetty & Rambabu, 2013), high performance liquid chromatography (HPLC) (El-Din, Eid, & Zeid, 2013; Elmasry et al., 2011), spectrofluorimetry (Alves & Poppi, 2009; Zadeh, Kohansal, & Sadeghi, 2011), potentiometry (Pasekova, Sales, Montenegro, Araujo, & Polasek, 2001), voltammetry (Sanghavi & Srivastava, 2010; Torriero, Luco, Sereno, & Raba, 2004), capillary electrophoresis (Marra et al., 2014), UV-Vis spectrophotometry (Abdelrahman, 2014; Sena & Poppi, 2004; Yamamoto, Takakuwa, Kato, & Asakaw, 2007) and flow injection analysis (FIA) with spectrophotometric detection (Lopez-Fernandez, Castro, & Valcarcel, 1990; Pereira, Aniceto, & Fatibello-Filho, 1998) and chemiluminescence (Wabaidur, Alam, Alothman, & Eldesoky, 2014).

The present study describes the development of a flow injection chemiluminescence (FI–CL) assay of ASA based on the inhibition effect of ASA on the CL reaction of luminol–hydrogen peroxide and potassium hexacyanoferrate.

EXPERIMENTAL

Apparatus

The schematic diagram of the FI-CL system used in this work is shown in Fig. 2. It consists of a peristaltic pump (DESAGA Heidelberg, with 6 channels and variable speed up to 10 mL/min) to deliver flow streams. A rotary valve

(Rheodyne U.S.A.) with variable sample volume was used to inject the reagent into flowing carrier streams. The flow cell that used for the present work was made by winding the length of glass tubing (0.8 mm i.d) to form coil of 100 µL volume. At the entrance of the cell, the reagent and luminol are mixed to produce CL. The mixing position of the flow cell was considered on the detector inside the spectrophotometer (Type CECIL CE303) the light source of which was blocked. D.C-microvoltmeter type (PHILIPS PM 2434) was used as associated electronics. The CL out-put was recorded by mean of x-t recorder (Type PM 825A PHILIPS – one line recorder).

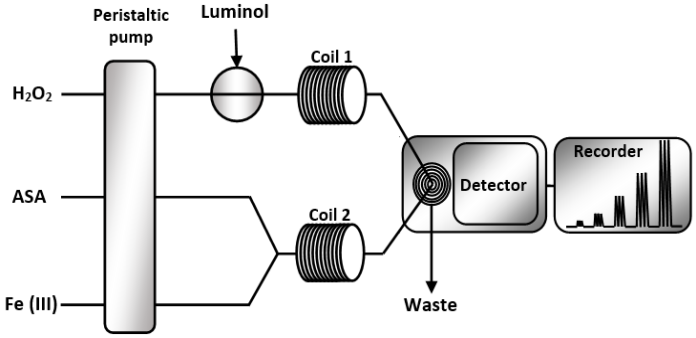

Figure (2): Schematic diagram of the FIA-CL manifold used for the determination of ASA.

Reagents

All chemicals and reagents used were of analytical or pharmaceutical grade. Distilled water (DW) was used for the preparation of all solutions.

Sodium carbonate solution

0.1 M sodium carbonate solution was prepared by dissolving 10.599 gm of Na_2CO_3 (Fluka) in a little amount of distilled water, transfer to 1.0 L volumetric flask quantitatively and the volume completed with distilled water.

Luminol solution

0.001 M luminol solution was prepared by dissolving 0.1771 gm of the solid (Surechem-LTD) in a little of 0.1 M sodium carbonate solution and completed the volume to 1.0 L in a volumetric flask with the same solution.

Hydrogen peroxide

A 1.0 M hydrogen peroxide solution was prepared daily by diluting 6.69 mL of H_2O_2 (GCC) (45% (v/v), d= 1.13 g/mL) in a 100 mL volumetric flask with distilled water. The peroxide solution was standardized against standard 0.1 M $KMnO_4$ (Jabbar & Faizullah, 2013). This solution was protected from light and kept in a brown bottle.

Potassium hexacyanoferrate solution

0.01 M was prepared by dissolving 3.472 gm of $K_3[Fe(CN)_6].H_2O$ (Fluka) in a little of DW (after addition of a small portion of acid), the volume was completed to 1.0 L in a volumetric flask.

Stock solution of acetylsalicylic acid

A stock standard solution of 200 µg/mL acetylsalicylic acid was prepared by dissolving 0.2 gm of ASA (Sammara-Iraq) in a small portion of DW and then completed to 1.0 L. Working solutions were prepared by dilution of stock standard solution.

Sample preparation

The average tablet weigh was calculated from the weight of 20 tablets which were ground into a fine powder and mixed. Equivalent to about 100 mg of acetylsalicylic acid was accurately weighted and put into a small beaker and dissolved in a small portion of DW. The mixture was shaken mechanically for 15 minute and then filtrated. The solution quantitatively was transferred to a 1.0 L volumetric flask and diluted with distilled water to the 1.0 L mark. Three different volumes of this solution were diluted in such a way that the concentration of the ASA in each case is in the range of the plotted calibration graph and analyzed according to the proposed procedure.

RESULTS AND DISCUSSION

In the absence of ASA, the chemiluminescence reaction of the luminol–hydrogen peroxide and potassium hexacyanoferrate (III) as catalyst system is strong. However, trace amounts of ASA inhibited the CL of the system.

At the same time, it was found that the reversed flow injection system can reduce the

waste of the reagents, and had higher sensitivity and steadier baseline. Therefore the reversed flow injection system was adopted in this study. The effect of the FI-Cl parameters on the analytical response were studied by changing each variable keeping the others constant.

Optimizations were started using the following preliminary values; chemical parameters: ASA (15 µg/mL), 0.0001 M Fe(III), 0.005 M H_2O_2, and 0.0003 M luminol; and physical parameters: flow rate of all lines 2.0 mL/min, coil 1: 10 cm, coil 2: 20 cm, and injection volume of luminol solution 100 µL at room temperature (25 °C).

Effect of Fe (III) concentration

The influence of potassium hexacyanoferrate concentration was studied in the range 0.0001 to 0.0005 M. The results showed (Fig. 3) that the CL intensity corresponding to 15 µg/mL ASA increased with increasing concentration of Fe (III) up to 0.0003 M, above which it remained constant. Consequently, a 0.0003 M Fe (III) was chosen.

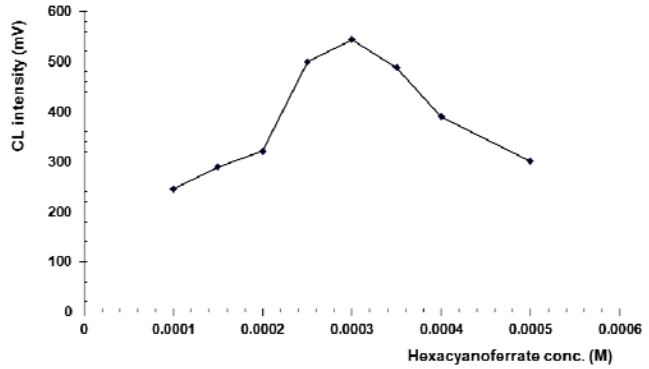

Figure (3): Effect of Fe(III) concentration on the CL intensity of 15 µg/mL of ASA.

Effect of hydrogen peroxide concentration

The effect of the hydrogen peroxide concentration on the performance of the method was evaluated in the range 0.003 to 0.040 M. In a series of measurements, a solution with 15 µg/mL ASA was analyzed, the results of these measurements are shown in Fig. 4, where the sensitivity is represented by the peak height (mV). The concentration of 0.008 M was chosen as optimal.

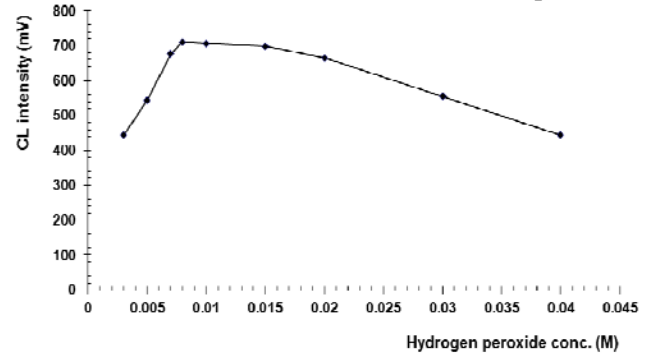

Figure (4): Effect of hydrogen peroxide concentration on the CL intensity of 15 µg/mL of ASA.

Effect of luminol concentration

The effect of luminol concentration on the CL intensity was investigated in the concentration range of 0.00001 to 0.0005 M and the results are shown in Fig. 5. When the concentration of luminol is higher than 0.0003 M the CL intensity decreased. Hence, the optimum concentration of luminol for the determination of ASA was 0.0003 M.

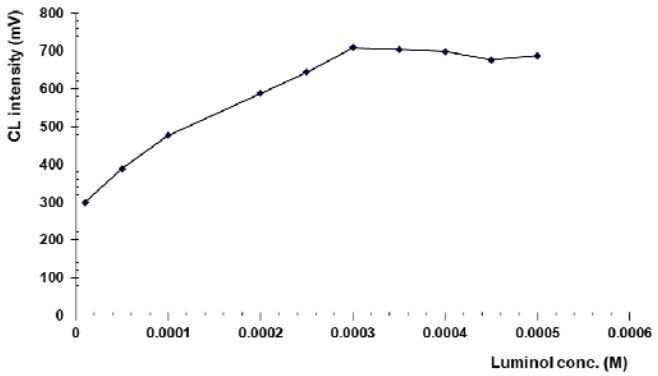

Figure (5): Effect of luminol concentration on the CL intensity of 15 µg/mL of ASA.

Effect of flow rate on the CL intensity

The effect of flow rate on the CL intensity was also investigated (Fig. 6). The CL intensity increases with increasing flow rate over the range of 0.5–5.0 mL/min probably because flow rates (<2.0 mL/min) resulted in lower CL emission, while flow rate more than 4.0 mL/min led to greater consumption of reagents and unacceptable reproducibility. Therefore, a flow rate of 3.0 mL/min was chosen for further studies.

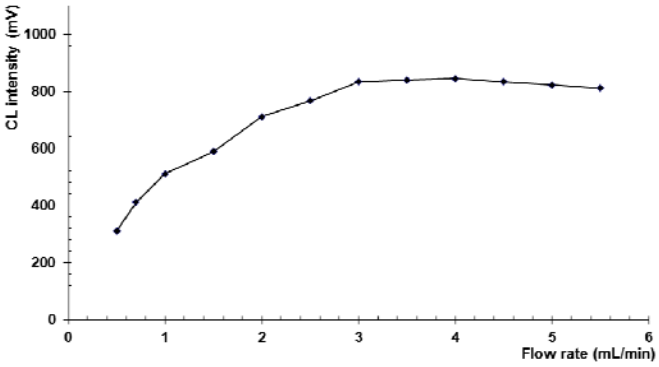

Figure (6): Effect of flow rate on the CL intensity of 15 µg/mL of ASA.

The effect of coil length

In order to improve the sensitivity, the hydrogen peroxide–luminol mixing coil length (coil 1) was also optimized. The result is shown in Fig. 7, indicating that the largest change in CL intensity was obtained using 10 cm mixing coil, which is chosen for the subsequent experiment.

The effect of coil 2 length was investigated in the range from 5 to 40 cm, for 15 µg/mL ASA solution (Fig. 7). This coil was used to increase the reaction time between Fe (III) and ASA. The peak heights strongly increase with the increases of the coil length up to 30 cm. A 30 cm coil 2 length was chosen, taking into account sensitivity of the procedure.

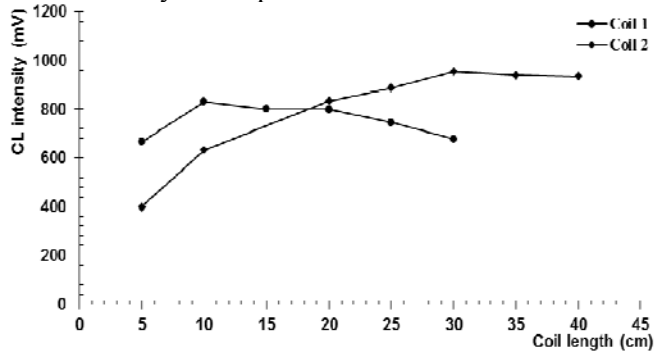

Figure (7): Effect of coil lengths on the CL intensity.

The effect of injected volumes

The effect of injected luminol volumes from 25 to 175 µL on the analytical signal for 15 µg/mL ASA solution was evaluated (Fig. 8). The increase of the reagent volume increase of CL intensity up to 100 µL. Therefore, a 100 µL volume of luminol solution was selected because it resulted in higher sensitivity and reasonable analytical frequency.

Table 1 illustrates summary of optimum chemical and physical conditions for the determination of ASA using rFIA-CL system.

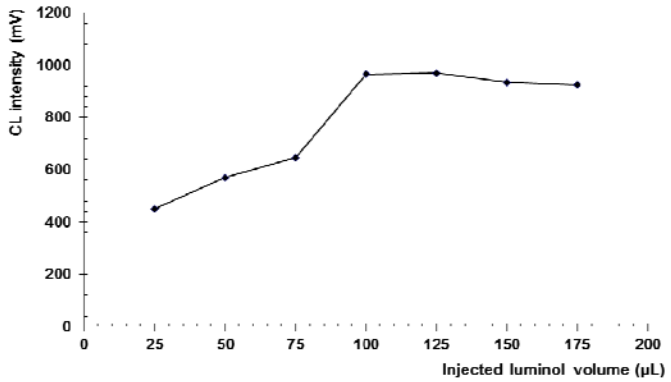

Figure (8): Effect of injected sample volume on the CL intensity using 15 µg/mL ASA.

Table (1): Summary of optimum chemical and physical conditions for the determination of ASA.

Parameters	Optimum value
Ferric nitrate	0.0003 M
Hydrogen peroxide (H_2O_2)	0.008 M
Luminol	0.0003 M
Flow rate	3.0 mL/min
Coil length	10 cm 30 cm
Injected volume	100 µL

VALIDATION ANALYSIS METHOD

Linearity

The linearity, of the developed rFI-CL method employed for quantitative determination of ASA was evaluated under optimum reaction conditions recorded in Table 1. The calibration graph was constructed by plotting the concentration in µg/mL of the ASA against the differences in CL-intensity in the form of peak height (ΔCL), between that of the blank (absence of ASA) CL1 and in the presence of ASA CL2 (i.e., ΔCL= CL1 - CL2). Three injections were used for each concentration. It was found that the calibration curve was linear in the concentration range within 2.5-125.0 µg/mL (Fig. 9), and the regression coefficient calculated by least squares produced for the calibration equation was 0.9985 with a detection limit of 0.5 µg/mL and analytical frequency of 75 determinations per hour.

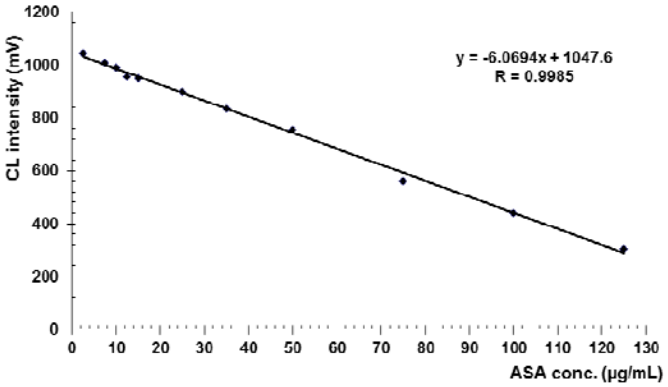

Figure (9): Calibration graph for the determination of ASA using FIA-CL system.

Accuracy and precision

To determine the accuracy and precision of the proposed method, five replicate were made of five different concentrations of standard ASA. The accuracy was checked with a relative error (RE%), while the precision of the method is checked with a relative standard deviation (RSD%) of the same solutions. The results are shown in Table 2 indicate good accuracy and precision.

Table (2): Accuracy and precision of the method.

Drug	Added (µg/mL)	Found (µg/mL) *	Error%	RSD%
ASA	10	9.80	2.00	3.05
	25	24.80	0.80	1.78
	50	49.80	0.40	0.91
	75	74.92	0.11	0.76
	100	100.12	-0.12	0.13

* Mean of five replicates (n=5)

Robustness

The evaluation of robustness was performed for system suitability to ensure the validity of analytical procedure. Some parameters including flow rate, reagent injection volume, and coil length were changed within a realistic range, and the quantitative influence of the variables was determined for the pre-analyzed sample solution, contains 20.0 µg/mL ASA. The results obtained, as shown in Table 3, illustrate that the proposed method is robust since the results are unaffected by changes in the experimental conditions.

Table (3): Robustness results of the method

Parameters		ASA (µg/mL)			Recovery%	RSD%
		Sample	**Added**	**Found***		
Flow rate (mL/min)	2.8	20	15	34.64	98.97	1.05
		20	30	49.83	99.66	1.23
	3.2	20	15	35.11	100.31	1.11
		20	30	51.01	102.02	1.17
Reagent injection volume (µL)	90	20	15	34.44	98.40	1.28
		20	30	49.04	98.08	2.70
	110	20	15	34.67	99.06	1.19
		20	30	49.65	99.30	1.37
Coil length (cm)						
Coil 1	5	20	15	34.51	98.60	3.32
	15	20	30	49.79	99.58	3.01
Coil 2	20	20	15	35.08	100.23	2.56
	40	20	30	50.63	101.26	2.17

* Mean of three replicate (n=3)

INTERFERENCES

Effects of some common foreign species which can be found in typical pharmaceutical preparations were examined for the determination of ASA. Table 4 shows maximum tolerable concentrations of the various compounds indicating that the contents of interferences in pharmaceuticals are lower than their tolerable concentrations, therefore, the proposed method could be used selectively to determine ASA in pharmaceutical dosage forms.

Table (4): Effect of interferences on the CL intensity of 25 µg/mL ASA.

Interfering species	**MAIC** [a] **(µg/mL)**	**ASA (µg/mL)**		**Error %**	**TCR**[c]
		Added	**Found** [b]		
Glucose	250	25.00	25.47	-1.89	10.0
Sucrose	250	25.00	24.48	2.07	10.0
Galactose	250	25.00	24.81	0.75	10.0
Lactose	250	25.00	25.14	-0.57	10.0
Fructose	250	25.00	25.31	-1.23	10.0
Starch	250	25.00	25.14	-0.57	10.0
Magnesium streate	125	25.00	23.82	4.70	5.0
Mixture of interferences	Above concentrations	25.00	24.98	0.09	

[a] *Maximum Allowable Interference Concentration (µg/mL)*
[b] *Mean of three replicate analyses*
[c] *Tolerable Concentration Ratio [Conc. Interferent (µg/mL) / Conc. ASA (µg/mL)]*

APPLICATION

The proposed reverse flow injection chemiluminescence system was applied to determine ASA in commercial pharmaceutical formulations. The results of the analysis of ASA are presented in Table 5. The obtained results were compared with those obtained with the recommended method in British Pharmacopoeia that used as a reference method (*British Pharmacopoeia*, 2009). The results of two methods are compared using the F-test and t-test. From the values of F-calculated (2.75) of the experiment and F-value from the table (6.39) with a confidence limit of 95%, the results indicated that there is no significant difference between the precision of two methods. From the values of t-calculated and t-table (Harris, 2009; Skoog, West, Holler, & Crouch, 2004) (t_{exp}= 0.087 and t_{table} = 2.776) with a confidence limit of 95% the results indicated that there is no significant difference between the accuracy of the two methods.

Table (5): Determination of ASA in commercial drug formulations using the proposed and the standard methods.

Formulation	Company	Amount nominal (mg per tablet)	Drugs found (mg) per tablet		E%
			Proposed method*	**Standard method**	
Aspirin - Gastro-Resistant Tablets (tablet)	Bristol Ltd- UK	75.00	74.95	74.67	-0.38
Ataspin (tablet)	Turkey	80.00	78.95	79.43	0.60
Aspirin Protect (tablet)	Bayer HealthCare AG - Saudi Arabia	100.00	99.52	99.01	-0.52
CorAspin (tablet)	Bayer HealthCare AG – Turkey	300.00	296.08	296.47	0.13
Acetylsalicylic acid (tablet)	ASA – Iran	325.00	323.43	323.65	0.07

* Average of three measurements (n=3)

CONCLUSIONS

A reverse flow injection chemiluminescence method was proposed for the determination of ASA based on inhibition of luminol-hydrogen peroxide catalyzed Fe(III) system. The proposed method is simple, precise, accurate, and sensitive for the determination of ASA in pharmaceutical formulations. It is free from many disadvantages that are common in spectrophotometric methods, such as complex sample treatment, critical working conditions, heating of the reaction mixture, expensive chemicals and instrumentation and high time consuming, etc. In addition, it decreases the possibility of interference caused by common foreign species. The results obtained by this method are compared well with those obtained by reference method in British pharmacopoeia.

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دەرزی لێدانی ڕۆیشتووی پەیوەست بە بریسکەی کیمیایی بۆ خەملاندنی ترشی ئەسیتایل سالسیلیك لە ناو پێکهاتە دەرمانسازیەکان

پوختە:

ئەم توێژینەوەیە ڕێگایەکی سادەی هەستیار بۆ خەملاندنی ترشی ئەسیتایل سالسیلیك (ئەسپرین) بە بەکارهێنانی دەرزی لێدانی ڕۆیشتووی پەیوەست بە بریسکەی کیمیایی دەگرێتە خۆ. ڕێگاکە بەندە لەسەر کپ کردنی بریسکەی کارلێکی کیمیایی نێوان لۆمینۆل–پیرۆکسیدی هایدرۆجین و شەشەم سیانیدی ئاسنیکی پۆتاسیۆم. بەها پراکتیکیە جیاکانی پەیوەندی دار بە بریسکەی کیمیایی ئەنجام دران بۆ دەست نیشان کردنی شیاوترین بار. سنووری ناسینەوە بریتی بوو لە 0,5 مایکرۆگرام/ملیلتر ترشی ئەسیتایل سالسیلیك و مەودای 2,5–125 مایکرۆگرام/ملیلتر بە هاوکۆلکەی بەستنەوەی 0,9985 وە بە خێرایی 75 خەملاندن لە کاتژمێرێکدا. ڕێگاکە بە سەرکەوتوویی ئەنجام درا بۆ خەملاندنی پێکهاتە دەرمانسازیەکان، وە ئەنجامەکان نزیکبوون لە ئەنجامەکانی ڕێگای ستاندەردی بەریتانی.

الحقن الجريانى المرتبط بالبريق الكيميائي لتقدير حامض الأسيتايل سالسيليك في المستحضرات الصيدلانية

الخلاصة:

يتضمن هذا البحث طريقة بسيطة و حساسة لتقدير حامض الأسيتايل سالسيليك (الأسبرين) في المستحضرات الصيدلانية من خلال الحقن الجرياني المرتبط بالبريق الكيميائي. تعتمد الطريقة على تثبيط حامض الأسيتايل سالسيليك لإشارة البريق الناتج من تفاعل اللومينول–بيروكسيد الهيدروجين مع بوتاسيوم سداسي سيانيد الحديديك. تم دراسة العوامل المختلفة التي تؤثر على البريق الكيميائي. قيمة حد الكشف كانت 0,5 مايكروجرام/مللتر حامض الأسيتايل سالسيليك، و كانت العلاقة الخطية بين تركيز الدواء و إشارة البريق في مدى 2,5 – 125 مايكروجرام/مللتر، معامل الارتباط 0,9985 و بمعدل 75 نموذج في الساعة. تم مقارنة نتائج تقدير المستحضرات الصيدلانية مع النتائج المحصلة من تقدير نفس النماذج بالطريقة القياسية البريطانية والتي أظهرت دقة و تطابقية جيدة للطريقة المقترحة.