Flow injection analysis for the photometric determination of promethazine-HCl in pure and pharmaceutical preparation via oxidation by persulphate using Ayah 3SX3-3D solar micro photometer

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Abstract:

The first flow injection spectrophotometric method is characterized by its speed and sensitivity which have been developed for the determination of promethazine-HCl in pure and pharmaceutical preparation. It is based on the in situ detection of colored cationic radicals formed via oxidation of the drug with sodium persulphate to pinkish-red species and the same species was determined by using homemade Ayah 3SX3-3D solar flow injection photometer. Optimum conditions were obtained by using the high intensive green light emitted diode as a source. Linear dynamic range for the absorbance versus promethazine-HCl concentration was 0-7 mmol.L^{-1}, with the correlation coefficient (r) was 0.9904 while the percentage linearity (r^2%) was 98.09%. the L.O.Q was 3.97 µg/sample, while L.O.D (S/N=3) = 0.2407µg/sample (5 µmol.L^{-1}) from the stepwise dilution for the minimum concentration of lowest concentration in the linear dynamic range of the calibration graph. The R.S.D% at 2 mmol.L^{-1} promethazine-HCl is less than 1% (eight replicates) using 150 µL sample volume. Throughput 30 sample.hr^{-1}. The method was applied successfully for the determination of promethazine-HCl in pharmaceutical preparation. By using paired t-test it was shown that there was no significant difference between the proposed method and official method and on that basis the new method can be accepted as an alternative analytical method.

Key words: Flow injection analysis, promethazine hydrochloride, spectrophotometric.

Introduction:

Promethazine-HCl, (2RS)-N,N-di-methyl-(10H-phenothiazin-10-yl) propan-2-amine hydrochloride is commonly known as neuroleptic tranquilizer and commonly used as a sedative, antihistamine, antiemetic and anaesthetic. (Fig.1.)
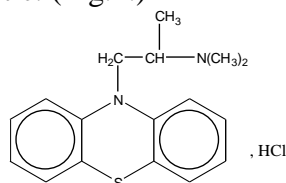

Fig.1: structure of promethazine-HCl

It is a white or faintly yellowish crystalline powder, very soluble in water and in alcohol [1]. Many analytical techniques have been employed for the determination of promethazine-HCl. Spectrophotometric procedures are based on the chemical oxidation of promethazine-HCl to form colored radical cation using oxidizing agents, such as cerium(IV) arsenite which is used as a strongly oxidizing solid-phase reactor[2], molybdopho-sphoric acid[3], iron (III) was used as oxidant and the iron (II) formed was

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measured as its 1,10-phenanthroline complex[4], iodic acid in the presence of sulphuric or phosphoric acid medium yielding a red colored product [5] and potassium persuphate to give a colored complex [6]. Various other methods, like oxidative-coupling reaction with 3-methyl-2-benzothi-azolinone hydrazone for determination of some anti-allergic agents [7], potentiometric sensor [8], turbidimetric method for determination of prometh-azine hydrochloride with bromophenol blue [9], nephelometric titration[10]. Titrimetric procedures are based on the redox properties of some N-substituted phenothiazine derivatives with hexa-amminecobalt (III)tri-carbonatocob-altate (III)[11]. Spectrofluorometric methods based on oxidation of promethazine-HCl to form its fluorescent sulphoxide derivative (12,13). Chemiluminescence reaction coupled with flow injection analysis, have also been employed [14,15]. Few methods applying flow injection have been reported: based on the use of a ceric ions in a sulphuric acid medium as an oxidant [16], other uses Fe(III)/Fe(II) as a redox system with biamperometric flow-through detector for the determination of phenothiazine derivatives in several commercial pharmaceutical preparations[17].

The present work is concerned with the determination of promethazine -HCl with the aid of a flow injection technique. The method is based upon the oxidation of promethazine-HCl by means of potassium persulphate in an aqueous medium. The oxidation product yields a pinkish red color measured at 515 nm. The procedure is simple, rapid and is proposed for the control analysis of the drug in pure and pharmaceutical preparations as an alternative analytical procedure.

Materials and Methods:
Chemicals

All chemicals used were of analytical reagent grade. Distilled water was used to prepare all the solutions. Freshly prepared solutions were always used. Standard solution (w/v) of promethazine-HCl ($C_{17}H_{20}N_2S$,HCl, 320.9 g.mol^{-1}, SDI, 10 mmol.L^{-1}) was prepared by dissolving 0.3209 gm of the drug in distilled water and diluting to the mark with the same in a 100 ml calibrated flask to give a stock solution, which was diluted further as required. A stock solution of sodium persulphate ($Na_2S_2O_8$, 238.10 g.mol^{-1}, BDH, 100 mmol.L^{-1}: 5.9525 g / 250 ml distilled water. Nitric acid, 1 mol.L^{-1},Dilute 128 ml of 70% HNO_3 (sp.gr. 1.42 g.ml^{-1}, BDH) with water to 2L in a calibrated flask, Sulphuric acid, 1 mol.L^{-1}. Dilute 111ml of 96 % m/m H_2SO_4 (sp.gr. 1.84 g.ml^{-1};BDH) with water in a 2 L calibrated flask. Hydrochloric acid,1 mol.L^{-1}. Dilute 176.50 ml of 35% HCl (sp.gr. 1.18 g.ml^{-1}, BDH with water in a 2L calibrated flask.

Apparatus and manifold:

The flow system used for the determination of promethazine-HCl,shown schematically in fig. 2.

A peristaltic pump:one channel, variable speed (Ismatec, Switzerland), 6-port medium pressure injection valve (IDEX corporation, USA) with a sample loop (0.7 mm i.d., Teflon, variable lengths) used for sample injection. The instrument response was measured by Ayah 3SX3-3D solar FI photometer (homemade) by using super bright blue, green and red light emitted diode (LED) as source with a detection using solar cell [18]. The output signals were recorded by potentiometric recorder (Siemens, Germany, range (1-500) mV or (1-500) Volt. Peak height was measured for each signals. UV-Vis (Cary-100 conc)

spectrophotometer digital double beam type (Japan) was also used to scan the spectrum of product of reactants using 1cm glass cell.
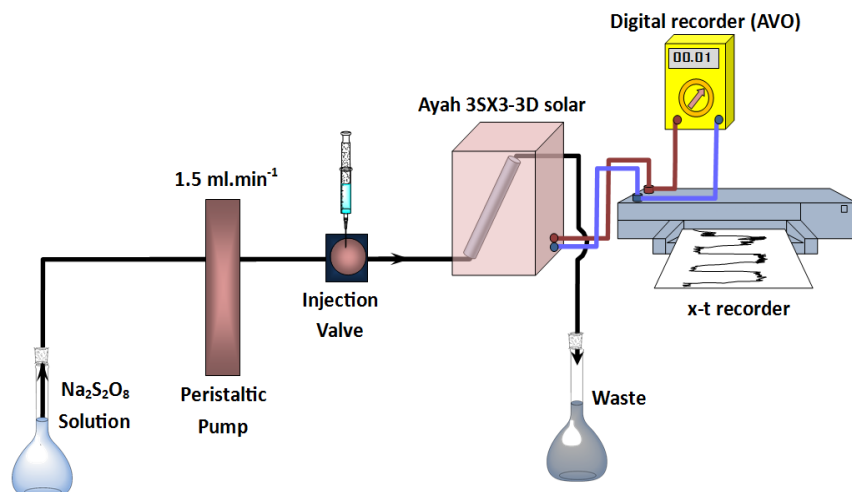

Fig. 2: Schematic diagram of flow injection analysis system.

Methodology:

The whole reaction manifold system for promethazine-HCl determination via directly oxidizes promethazine-HCl to colored cationic radical with sodium persulphate is shown in fig. 2. The manifold system is simple and it is composed of one line supplied sodium persulphate (30 mmol.L^{-1}) at 1.5 ml.min^{-1} which leads to the injection valve to carry promethazine-HCl sample of 150 µL (loop length 39 with 0.7 mm I.D). The absorbance peak of the resulting pinkish-red product is followed by using Ayah 3SX3-3D solar photometer and the variation of response was monitored by using green light emitted diode (LED) throughout the reaction. Each solution was assayed in triplicate. A proposed mechanism of oxidation of promethazine-HCl in aqueous medium is presented in fig.[19]
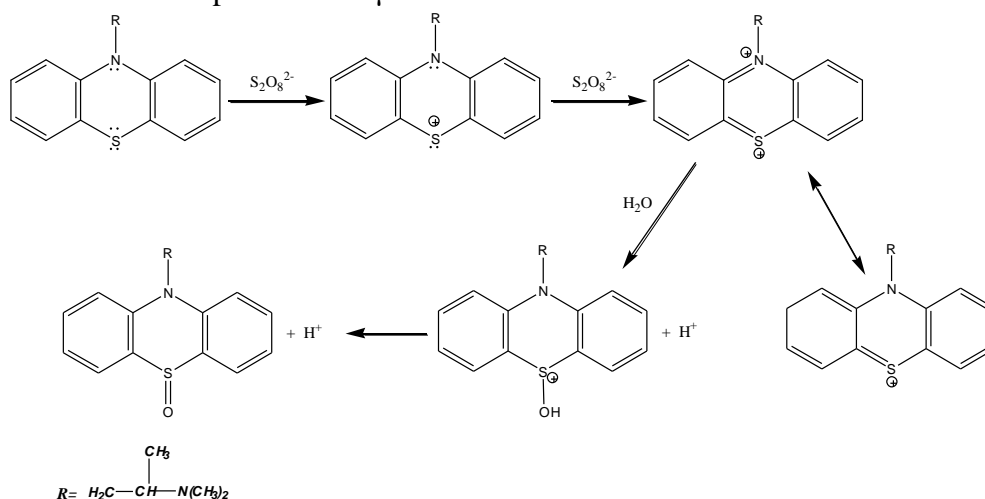

Fig. 3: Proposed mechanism of reaction between promethazine-HCl and sodium persulphate.

Results and Discussion:
spectroscopic study of promethazine -HCl-$S_2O_8^{2-}$ system.

A dilute aqueous solution of promethazine-HCl when mixed with sodium persulphate as oxidizing agent an intense pinkish-red colour product was formed immediately, the product shows a maximum absorption at 515 nm against reagent blank (promethazine-HCl) as shown in fig. 4.

The colour product of the oxidized species formed by promethazine-HCl as mentioned previously was measured by using Ayah 3SX3-3D solar FI photometer at three different light emitted diode (LED) [blue (470 nm), green (525 nm), and red (635 nm)], a maximum response measured in mV obtained when using the high intensity green light emitted diode (525 nm) as source as shown in fig. 5.

Optimum conditions for oxidation of promethazine-HCl with sodium persulphate by using homemade Ayah 3SX3-3D solar FI photometer.

Chemical variable
- **Effect of sodium persulphate concentration:**

A series of sodium persulphate solutions were prepared by ranging (5-50) $mmol.L^{-1}$ to establish the optimum concentration that can be used. The study was conducted that 30 $mmol.L^{-1}$ of sodium persulphate was necessary to achieve the maximum colour intensity of the product (fig. 6). Above and below this concentration, the colour intensity diminished. The probable mechanism is that sodium persulphate instantaneously oxidizes the promethazine-HCl at room temperature giving a pinkish-red coloured species that is believed to be a radical cation. Most probably, the excess of persulphate causes an irreversible oxidation of the coloured product to a colourless sulphoxide with the loss of one or more electrons.

- **Effect of acidic media on the absorbance of promethazine-HCl-$S_2O_8^{2-}$ system:**

The oxidation of promethazine-HCl with persulphate was studied in different acidic media (nitric, hydrochloric and Sulphuric acid) at 30 $mmol.L^{-1}$ concentration in addition to the aqueous phase medium yielding immediately a pinkish-red colour. The results are depicted in fig. 7. In which that nitric acid increases absorbance which might be explained as it is a powerful oxidizing agent which increases the coloured species; in addition to persulphate effect. While using hydrochloric acid absorbance is dcreased due to the reducing effect of the chloride ions which in turn consume part of the available of persulphate ion. While for Sulphuric acid no significant difference was found compared to the aqua medium. On the basis of the outcome of the results obtained from this section; aqueous medium was chosen as the optimum medium to conduct the reaction pattern.
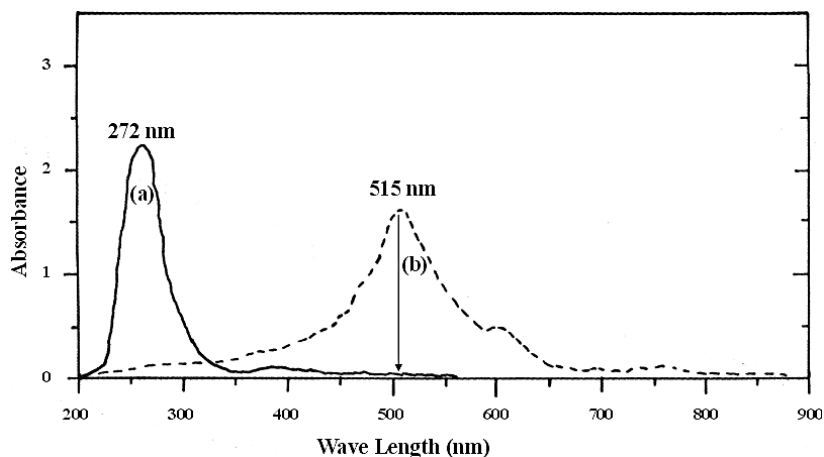
Fig. 4: Absorbance spectra for pinkish-red species formed by promethazine-HCl (1 mmol.l^{-1})-S$_2$O$_8^2$(7 mmol.l^{-1}) system; a: Absorbance spectra (—) for promethazine-HCl, b: Absorbance spectra (---) for promethazine-HCl-S$_2$O$_8^{-2}$ system using promethazine-HCl as a reference blank.
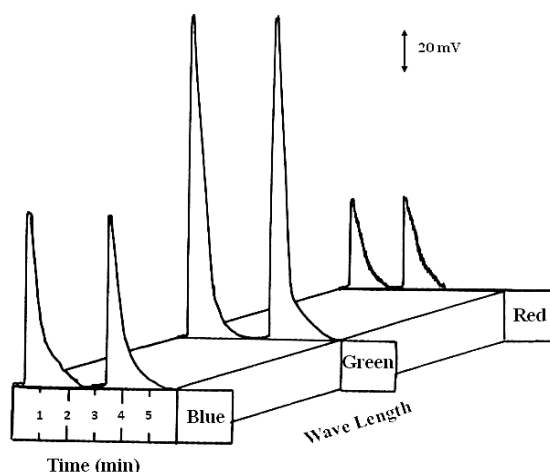

Fig. 5: A maximum response measured in mV of pinkish-red species formed by injection 100 μL(1 mmol.L^{-1}) of promethazine-HCl at three different light emitted diode which is blue (470 nm), green (525 nm) and red (635 nm) as source using homemade Ayah 3SX3-3D solar FI photometer.
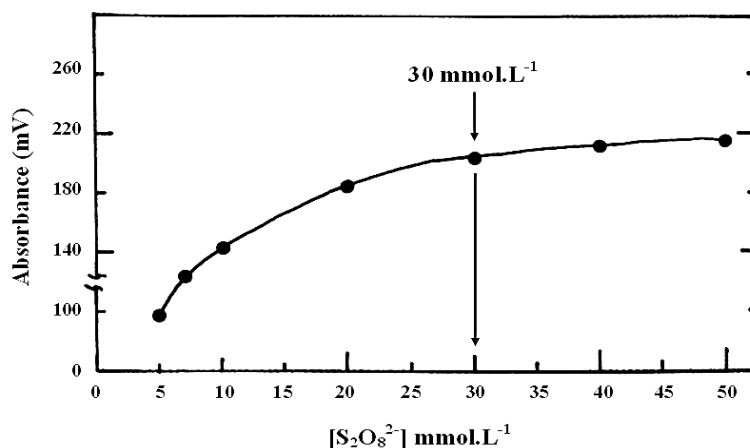

Fig. 6: Effect of variation of sodium persulphate concentration on absorbance using promethazine-HCl (2 mmol.L^{-1})-S$_2$O$_8^{2-}$, with sample volume 100 μL, flow rate: 1ml.min^{-1}.
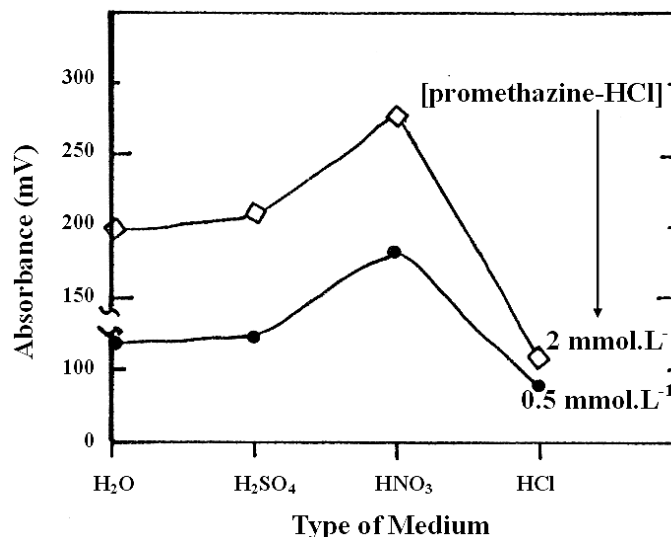
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Fig. 7: Effect of the acid medium on the oxidation of promethazine-HCl (2 mmol.L^{-1}) with S$_2$O$_8^{2-}$ (30 mmol.L^{-1}), each of acid at 30 mmol.L^{-1} concentration.

Physical variables

• Effect of flow rate of sodium persulphate:

A set of experimental was carried out for the optimization of the preferred flow rate of sodium persulphate as a carrier (30 mmol.L^{-1}) that extent (0.9-3) ml.min^{-1} using 100 μL of 2 mmol.L^{-1} of promethazine-HCl and 15 second as purge time for the sample segment. The results obtained were summarized in fig. 8. A, B. It was noticed that at slow flow rate, there is increase in dilution and dispersion which might cause an increase in base of response (Δt$_B$) as show in fig. 8 A.

while at higher flow rate (>1.5ml.min^{-1} in fig. 8 A), although the effect on physical parameter was not very crucial on the response obtaining regular responses and sharp maxima but it is not very high due to departure of reactants from measuring cell prior to completion of reaction. Fig. 8 B shows that an increase in the flow rate led to reach the coloured species to the measuring cell in a minimum time (T), therefore a flow rate of 1.5 ml.min^{-1} was adopted for promethazine-HCl determination to obtain a maximum response and less Δt$_B$ as shown in fig. 8 A.
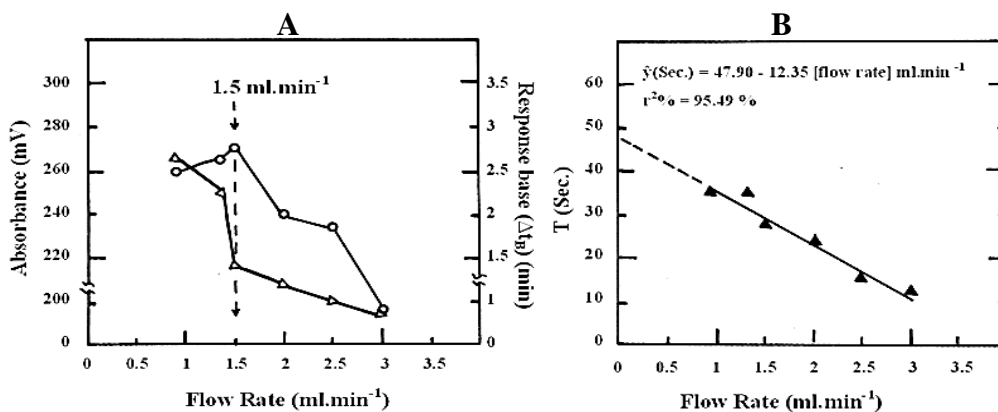
Fig. 8: Effect of variation of flow rate on:
A- (–○–): Absorbance for pinkish-red species & (–△–) response base width (Δt$_B$).
B- T (time for the departure of sample segment from injection valve reaching to the measuring cell).
Using parameter: promethazine-HCl (2 mmol.L^{-1})-S$_2$O$_8^{2-}$ (30 mmol.L^{-1}), sample volume 100 μL.

- **Effect of sample volume:**
    Using the optimum flow rate of 1.5 ml.min^{-1} with a various volumes (76-190) µL were injected using open valve mode i.e. allowance for concentration purge of sample segment from the sample loop in the injection valve. The data obtained were plotted as shown in fig. 9 A showing that the

optimum sample volume is 150 µL for better response as shown in fig. 9 B, while increasing sample volume more than 150 µL gave broadening of the base as well as the peak maxima which was most probably attributed to continuous long time duration of colour segment in front of detector.
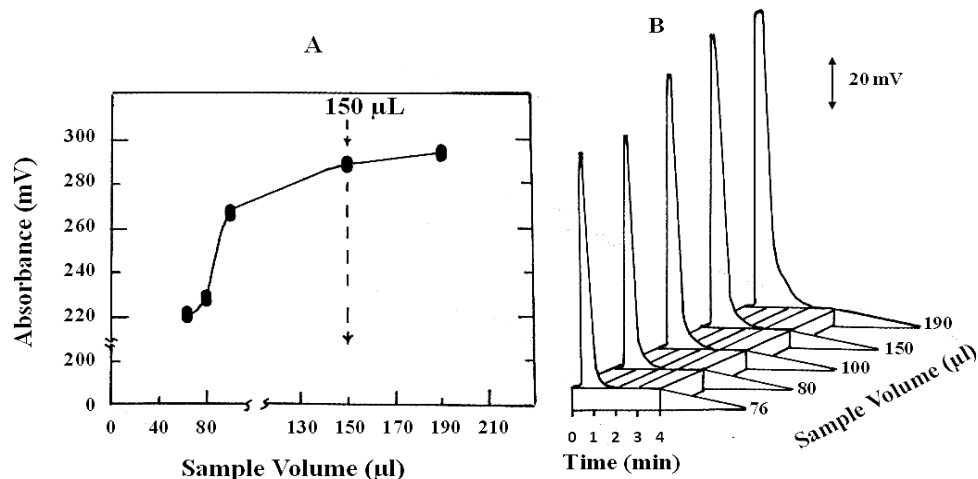

Fig. 9: Effect of variation of sample volume on:
A- Absorbance.
B- Response profile of Ayah 3SX3-3D solar FI photometer for pinkish-red species.

- **Effect of purge time:**
    Using different purge time for the sample segment i.e. using 10 to 35 seconds allowed permissible time for the carrier solution to passing through the injection valve in injection mode followed by turning the injection valve to the load position. Sample volume of 150 µL was used. Fig. 10 shows the continuation of the increase in absorbance with increase of injection time up to 20 seconds then followed by a decrease with the increase of injection time. The decrease in absorbance when using less than 20 seconds was attributed to the incomplete purge of the sample from sample loop in the injection valve. Above 20 seconds the decrease was attributed to the resistance of flow due to the passage through the injection

valve, therefore 20 seconds as purge time was chosen as optimum time to the complete purge of the sample from sample loop.

- **Stability of the coloured species with time:**
    Using the optimum parameters achieved at previous sections: promethazine-HCl (2 mmol.L^{-1})-S$_2$O$_8^{2-}$ (30mml.L^{-1}), the coloured species was formed immediately and the stability of the colour product with respect time was depicted in fig. 11. in which absorbance were recorded starting from time = 10 seconds up to 12 hr shows that the coloured species were faded in colour starting at time 4 hrs, followed by a continuous decrease up to 10 hrs where it completely disappears.

The development of pinkish-red colour that fades continuously is in accordance with the work of duchinski[20]who established that oxidation proceeds in two-1-electron stages. The first stage results simply from the loss of an electron from the parent compound and produced the corresponding free radical cation which is intensely pinkish-red. The second stage is a further 1-electron oxidation of the free radical to a colourless sulphoxide.
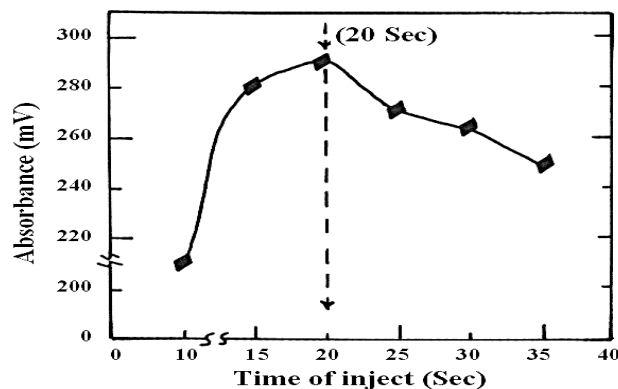
Fig. 10: Effect of variation of injection time on the oxidation of promethazine-HCl with $S_2O_8^{2-}$. Using optimum parameter: promethazine-HCl (2 mmol.L^{-1})-$S_2O_8^{2-}$ (30 mmol.L^{-1}), flow rate(1.5ml.min^{-1}), sample volume (150 µL).
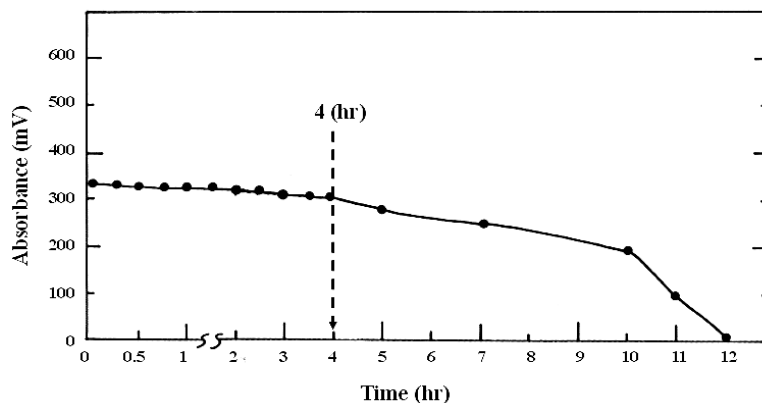
Fig. 11: Effect of time on stability of the colour species.

Variation of absorbance versus concentration of promethazine-HCl

Various concentrations (0, 0.03, 0.05, 0.1, 0.3, 0.5, 0.7, 1, 2, 3, 5, 7 and 10 mmol.L^{-1}) of promethazine-HCl were prepared by using the parameters established above. Each measurement was repeated three times. The absorbance of the average peak height (mV) was plotted against the concentration of promethazine-HCl, a straight-line graph (fig.12) from 0-7 mmol.L^{-1} of promethazine-HCl was obtained. Above 7 mmol.L^{-1} the value for correlation coefficient will deviate from linearity most probably due to the high intensity of the coloured species in front of detector. The obtained results were tabulated in table no. 1.

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Table 1: Summery of calibration graph results for the determination of promethazine-HCl using sodium persulphate as oxidizing agent.

Measured [promethazine-HCl] mmol.L^{-1}	Linear dynamic range mmol.L^{-1} n=12	$\hat{y}$(mV) = a ± S$_a$t + b ± S$_b$t [x] mmol.L^{-1} at confidence interval at 95%, n-2	r, r^2	t$_{tab}$	$t_{cal} = \dfrac{\lvert r \rvert \sqrt{n-2}}{\sqrt{1-r^2}}$ at 95%, n-2
0-10	0-7	61.79±28.83+107.85±10.61[X] mmol.L^{-1}	0.9904 98.09%		2.228<< 22.67

x= promethazine-HCl.

Limit of detection for promethazine-HCl was conducted through four methods as tabulated in table no. 2. at injected sample volume of 150 µL, also L.O.Q was reported.

Table 2: Limit 0f detection and limit of quantitation for promethazine-HCl at optimum parameter.

General dilution for the minimum conc.	Based on dilution factor (df)	Based on the value of slope $x = \dfrac{3S_B}{Slope}$	Linear equation $\hat{y}$(mV) = y$_B$+3S$_B$	L.O.Q= $\hat{y}$(mV) = y$_B$+10S$_B$
0.2407 µg	0.0401 µg	0.402 µg	47.89 µg	3.97 µg

df= 6
x: value of L.O.D. based on slope
S$_B$: standard deviation of blank solution
y$_B$: average response for the blank solution (equivalent to intercept in straight line equation)
L.O.D: limit of detection
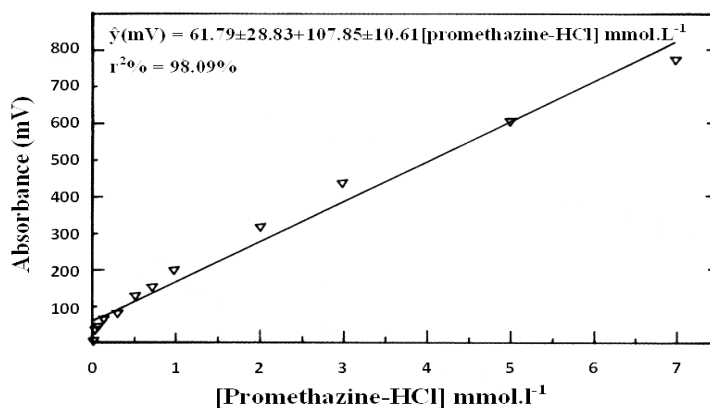*L.O.Q: limit of quantitation*

Fig. 12: Effect of variation of promethazine-HCl on absorbance at optimum conditions: promethazine-HCl (variation of conc.)-S$_2$O$_8^{2-}$ (30 mmol.L^{-1}), flow rate:1.5 ml.min^{-1}, sample volume 150 µL, injection time 20 sec.

The repeatability was studied via measurements of R.S.D% for some selected concentration of promethazine-HCl (n=8) tabulated in table no. 3. The response profile at concentration 0.5, 3 mmol.L^{-1} of eight successive injected sample measurements as shown in fig. 13.

Table 3: Repeatability of promethazine-HCl results.

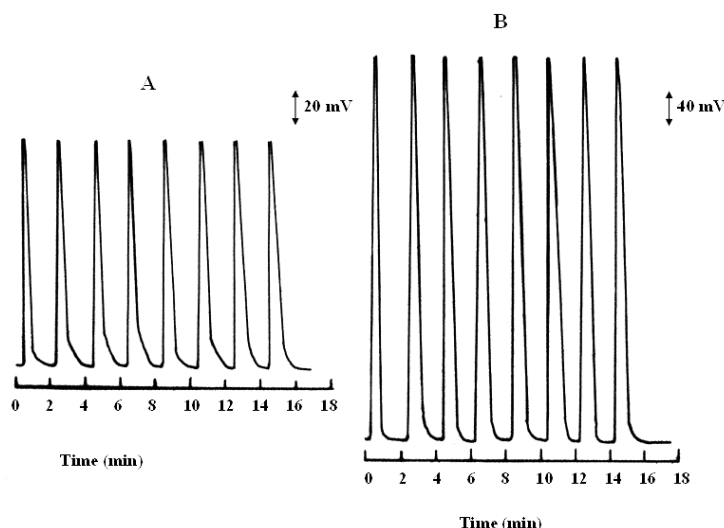| [Promethazine-HCl] mmol.L^{-1}	$\bar{y}_i$ (n=8) (mV)	Repeatability RSD%	Confidence interval of the mean at 95% $\bar{y}_i \pm t^*_{0.025,n-1} \dfrac{\sigma_{n-1}}{\sqrt{n}}$
0.5	130	0.68	130 ± 0.74
2	270	0.99	270 ± 1.67
3	440	0.30	440 ± 1.10

* t$_{0.025, n-1}$= 2.365 , n=8

Fig. 13: Successive repeatable measurement for promethazine-HCl; A: 0.5 mmol.L^{-1},   B: 3mmol.L^{-1}.

Analysis of pharmaceutical preparation

The proposed method achieved in this work was used for the analysis of promethazine-HCl in pharmaceutical preparation and was compared with the official method [21] . Thirteen tablets were weight, crushed and grinded. Tablets containing 25 mg of promethazine-HCl were weighted 0.51396gm(96.27mg active ingredient) (3mmol.l^{-1}) from pharmaceutical preparation, dissolved in as a little water, followed by filtration to get rid of undissolved materials and completed the volume to 100 ml with distilled water; 5ml drown to each of five 100 ml volumetric flasks followed by the addition of gradual volumes of standard promethazine-HCl (0, 1, 1.5, 2, 2.5) ml of 10 mmol.L^{-1} to obtained (0.15, 0.25, 0.3, 0.35, and 0.4) mmol.L^{-1}. Flask no. 1. is the sample. The measurement was conducted by proposed method and the results were mathematically treated for the standard addition method. The results were tabulated in table no. 4. at confidence interval 95%, paired t-test[22,23] was used as shown in table no. 5. The obtained results indicate clearly that there was no significant difference between newly developed method (FIA-Spectrophotometer) with official method[21] at 95% confidence level because calculated t value is less than tabulated t value.

Table 4: Results for the determination of promethazine-HCl in pharmaceutical preparation using proposed method.

Commercial name, content and country	Confidence interval for the average weight at 95% $\overline{w} \pm 1.96 \frac{\sigma_{n-1}}{\sqrt{n}}$ (g)	Sample weight (4.8135 mg) equivalent to 0.15 mmol.L^{-1} of active ingredient (g)	confidence interval for the theoretical content of active ingredient at 95% n=∞ (mg)	Practical content of active ingredient at 95% n=∞ (mg)	Recovery %
Histazine 25 mg United pharmaceutical Jordanie	0.1335 ± 0.00088	0.0257	25 ± 0.1648	24.6705 ± 2.693	98.68%

Table 5: Paired t-test results for FIA-Spectrophotometer proposed method with official method [21] for determination of promethazine-HCl in pharmaceutical preparation.

Purposed method FIA-spectrophotometer ($\bar{x}$)	Official method 'cuoted value' (μ)	σ_{n-1}	n	$\|t_{cal.}\| = \dfrac{(\bar{x} - \mu)\sqrt{n}}{S}$	t_{tab} at 95%, n-1
24.67 mg	25 mg	2.38	8	$\|$ -0.39 $\|$ = 0.39 << 2.365	

Conclusion:

A spectrophotometric FIA procedure is proposed for the determination of promethazine-HCl with application in the control analysis of pharmaceutical formulations. The method is based on the oxidation of promethazine-HCl with sodium persulphate to pinkish-red product. From the experimental point of view, the manipulation is very simple and sequential measurement was permitted with sample frequency up to 30 samples per hour. The proposed method uses cheaper instruments and reagents compared with classical spectrophotometry[6], flourometry and other FIA methods with different oxidizing reagents. In this paper a more effective faster determination was achieved by using Ayah 3SX3-3D solar FI photometer, light emitted diode as source with a detection using solar cell. The R.S.D% was < 1% observed for all samples, which is an indication of satisfactory precision of the proposed method. The standard addition method was used to avoid matrix effects. Also this method can be applied to micro determination of promethazine-HCl in pure as well as in pharmaceutical preparation and offers the advantages of high sensitivity without the need for heating or extraction. The statistical analyses are in good agreement with those of the British pharmacopoeia.

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التحليل بالحقن الجرياني للتقدير الطيفي للبروميثازين-هيدروكلورايد في المستحضرات الصيدلانية والنقية من خلال الاكسدة باستخدام بيرسلفات الصوديوم ومنظومة Ayah 3SX3-3D solar الطيفية

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الخلاصة:

تم تطوير طريقة تحليلية طيفية سريعة وحساسة باستخدام تقنية الحقن الجرياني لتقدير بروميثازين – هيدروكلورايد في المستحضرات الصيدلانية والنقية. استندت الطريقة على التحسس الاني بالجذور الايونية الملونة والمتكونة من خلال اكسدة الدواء باستخدام بيرسلفات الصوديوم لتكوين اصناف بلون احمر – برمنغناتي وقياس الامتصاصية باستخدام منظومة مصنعة محلياً طيفية للتحليل بالحقن الجرياني المستمر Ayah 3SX3-3D solar FI photometer . تم دراسة الظروف المثلى باستخدام ثنائي الوصلة باعث للضوء الاخضر وبشدة عالية كمصدر للتشعيع. المدى الخطي الفعال لعلاقة تغير الممتصية مع التركيز للبروميثازين – هيدروكلورايد يمتد (0-7)مللي مول.لتر$^{-1}$ وبمعامل ارتباط r= 0.9904 بينما نسبة الخطية r^2%= 98.09. حد التقدير الكمي (L.O.Q) 3.97 مكغم/انموذج بينما حدود الكشف (L.O.D) (S/N= 3) = 0.2407 مكغم /انموذج (5 مايكرومول.لتر$^{-1}$) من التخفيف التدريجي لاقل تركيز في منحني المعايرة. الانحراف القياسي النسبي المئوي RSD% لتركيز 2 مللي مول.لتر$^{-1}$ من البروميثازين – هيدروكلورايد اقل من 1% (تكرارية (n=8)) باستخدام حجم لانموذج محقن 150 مايكرولتر. معدل النمذجة = 30 انموذج/ساعة. طبقت الطريقة بنجاح لتقدير الدواء في المستحضرات الصيدلانية.

باستخدام اختبار t–المزدوج لوحظ انه لا يوجد فرق جوهري بين الطريقة المقترحة والطريقة القياسية، وعلى هذا الاساس بالامكان استخدام الطريقة المقترحة للتحليل بالحقن الجرياني الطيفي كبديل للطريقة القياسية.