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Comparative spectrophotometric determination of hriprolidine hydrochloride drug (TPH) using oxidizing agents Potassium permanganate & Potassium dichromate

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Abstract

This research deals with spectrophotometric method for determination of Triprolidine Hydrochloride drug (TPH) using two different oxidizing agents; Potassium permanganate and Potassium dichromate .The analytical results obtained are as follow :1- The alkaline media (NaOH): Potassium permanganate gives green solution which gives spectrum with amaximum absorption at 612nm its found that optimum conc. for alkaline solution is $1.2X10^{-1}$M and $1X10^{-3}$M for Potassium permanganate within 10 minutes oxidizing time. The statistical and other analytical results for drug concentration range (1-12) ppm are as follows: Correlation coefficient r = 0.9989 , molar absorption coefficient 26993 L/mol.cm and sandel's sensitivity is 0.0122μg/cm^2 at λ_{max} =612nm. This method is applied for the determination of TPH in pharmaceutical preparations with percentage recovery not less than 96.22 %. 2-The acidic media (HCl): Potassium dichromate gives yellow solution with amaximum absorption at 350 nm the optimum conc .for acidic solution is $1X10^{-2}$ M and $1.5X10^{-3}$M for Potassium dichromate during15 minutes oxidizing time. The statistical and other analytical results for drug concentration range (1-12) ppm are as follows: Correlation coefficient r = 0.9969, molar absorption coefficient 38559.5 L/mol.cm and sandel's sensitivity is 0.0086μg/cm^2 at λ_{max} =350nm This method is applied for the determination of TPH in pharmaceutical preparations with percentage recovery not less than 94.66%.

تقدير عقار ثلاثي بروليدين هيدروكلوريد(TPH) طيفياً بالاكسدة بعاملي برمنكنات البوتاسيوم وثنائي كرومات البوتاسيوم والمقارنة بينهما

أسماء أحمد محمد الراشدي

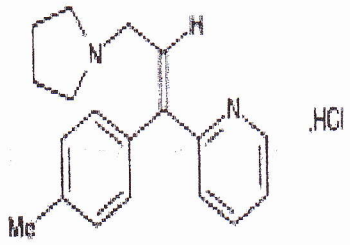
المستخلص

يتضمن البحث تقدير العقار الدوائي ثلاثي بروليدين هيدروكلوريد (TPH) بطريقتين باستخدام عاملي الاكسدة برمنغنات البوتاسيوم وثنائي كرومات البوتاسيوم بعد الحصول على مركب ملون للعقار مع كلا المؤكسدين وقياس طيف الامتصاص الجزيئي للمحاليل الناتجة كاختبارات أولية للوصول الى الطول الموجي الذي يسجل عنده اعلى امتصاص للطيف الناتج وكانت النتائج حسب الاتي :1-مع برمنغنات البوتاسيوم في وسط قاعدي (هيدروكيد الصوديوم) :حيث تكون محلول أخضر اعطى طيف امتصاص عند الطول الموجي612 نانوميتر وبعد دراسة الظروف التجريبية وجد أن أفضل تركيز للمحلول القاعدي $1.2X10^{-1}$ مولاري وللبرمنغنات $1X10^{-3}$ مولاري وبزمن قدره (10)دقائق ، أما النتائج الاحصائية والتحليلية لمدى تراكيز العقار (1-12)جزء من المليون فكانت كالاتي : معامل الارتباط r= 0.9989 ومعامل الامتصاص المولاري 26993.لتر /مول .سم وحساسية ساندل

0.0122عند الطول الموجي 612 نانوميتر وبعد تطبيق الطريقة على المستحضرات الصيدلانية وجد أن الاسترادادية المئوية لاتقل عن 96.22%-2. مع ثنائي كرومات البوتاسيوم في وسط حامضي (الهيدروكلوريك) :حيث نتج محلول ذا لون أصفر اعطى طيف امتصاص عند الطول الموجي 350 نانوميتر وبعد دراسة الظروف التجريبية وجد أن أفضل تركيز للمحلول الحامضي $1X10^{-2}$ مولاري وللثنائي كرومات $1.5X10^{-3}$ وبزمن قدره (15)دقيقة ، أما النتائج الاحصائية والتحليلية لمدى من تراكيز العقار فكانت (1-12)جزء من المليون فكانت كالاتي : معامل الارتباط r= 0.9969 ومعامل الامتصاص المولاري 38559.5لتر /مول .سم وحساسية ساندل 0.0086 عند الطول الموجي 350نانوميتر وبعد تطبيق الطريقة على المستحضرات الصيدلانية وجد أن الاسترادادية المئوية لاتقل عن 94.66 %.

Introduction

Triprolidine Hydrochloride,TPH [1] is of empirical formula $C_{19}H_{22}N_2,HCl,H_2O$ and molecular weight is 332. 9



TPH A white, crystalline powder; almost odorless. soluble in water; very soluble in chloroform; soluble in (96%) ethanol; practically insoluble in ether, m.p 60c° [2]. TPH is used antihistamine ,economize cure of sternutation ,prurigo ,lacrimation and desensitization of aspiratory system [3] but the side effect is apnoea and puke [4] . This important (TPH) drug has been determined by several spectrophotometric methods as determined by Mumtaz[5] using dichlorobenzene, linear calibrations were obtained from 50-150 ppm.Sahira used[6] organic complex $[PdI_4]^{-2}$, linear calibrations were obtained from1-10 ppm .In this study TPH drug determined by using potassium permanganate and potassium dichromate as oxidizing agents.

Experimental

Apparatus: Shimadzu UV-visible spectrophotometer UV-1650 PC was used to measure the absorbance with 1cm quartz cells, sartorius balance, and Isuzu hot air rapid drying oven ,were also used.

Solutions:

All materials used , solids and liquids are pure and the water used for preparing the solution is distilled water .

1- *Standard (TPH) Solution (1000ppm):*

0.1gm dissolve in few quantity of distilled water and transferred to volumetric flask 100ml then complete to the mark.

2- *Reagent solutions:-*

The following solutions were prepaerd as:

1.0 M NaOH (BDH) , 0.01M $KMnO_4$(fluka), 0.1 M HCl (Reidel – Dehen),

0.01M $K_2Cr_2O_7$ (BDH).

3-*procedure for deteremination of (TPH) in pharmaceutical preparation*

SDI has two products wich contain TPH drug as an effective material (Samafied syrup& Samafied tablet).

A-Samafied syrup by direct calibration

Transferred 12.5 ml from syrup to 50 ml volumetric flask and diluted to the mark with distilled water, from this solution diluted 6.25ml with distilled water in volumetric flask of 25ml.and we transferred several volumes 0.1-1.5ml in volumetric flask of 10ml and applied optimum condtion for both agents .

B- Samafied tablet by direct calibration

Ten tablets (average weight of one tablet is 0.2560g) were carefully crushed, and then 0.1522gm of the resulted powder

was dissolved in 30 ml of distilled water and diluted to the mark in a volumetric flask of 50ml and then treated such as syrup.

Results and Discussion

I: Spectrum studies

I.1- Drug Spectrum:1.0 ml of TPH 100ppm was transferred into 10ml volumetric flask diluted to the mark with distilled water. This solution shows two maximum absorbtions at 236 and 276 nm Fig (1).

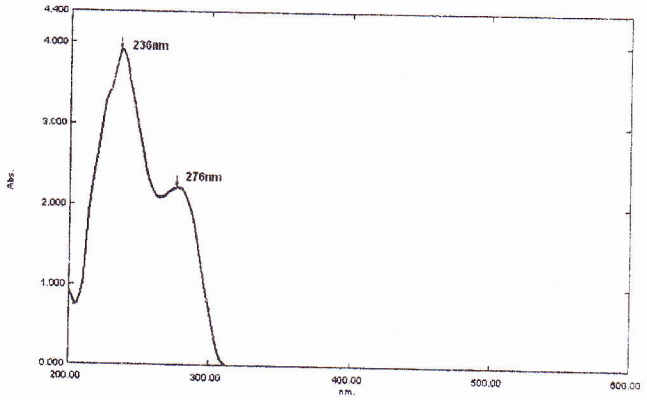


Fig (1):- Absorption spectrum of TPH drug

I.2-drug - $KMnO_4$ Spectra

A- primary test :

1.0 ml of 0.01M $KMnO_4$ was mixed with 1.5ml of 1.0M NaOH and 1.0ml of 100ppm TPH in 10ml volumetric flask complete to the mark by distilled water then measure absorption against the water (blank) at wave length 612nm the absorption spectrum as in Fig (2).

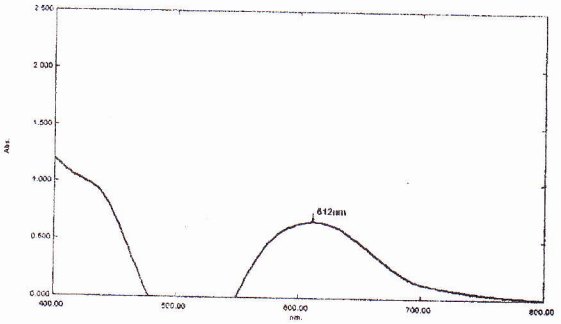


Fig (2):- Absorption spectrum of TPH - $KMnO_4$

B- Optimum conditions

The following tables illustrate the optimum condition of NaOH and $KMnO_4$ conc. In adition to optimum oxidizing time that give best absorbance .

Table (1): Effect of NaOH concentration

conc. of NaOH	A
$1X10^{-2}$ M	0.176
$2X10^{-2}$ M	0.192
$4X10^{-2}$ M	0.553
$8X10^{-2}$ M	0.865
$1X10^{-1}$ M	0.911
$1.2\ X10^{-1}$ M	0.936
$1.4\ X10^{-1}$ M	0.930
$1.6\ X10^{-1}$ M	0.919
$1.8\ X10^{-1}$ M	0.855

Table (2): Effect of $KMnO_4$ concentration

Conc. of $KMnO_4$	A
$1X10^{-4}$ M	0.061
$2X10^{-4}$ M	0.134
$3X10^{-4}$ M	0.226
$4X10^{-4}$ M	0.341
$5X10^{-4}$ M	0.478
$7X10^{-4}$ M	0.758
$9X10^{-4}$ M	0.791
$1\ X10^{-3}$ M	0.887
$1.2X10^{-3}$ M	0.853
$1.4X10^{-3}$ M	0.831

Table (3): Oxidation time

Absorbance at preparation	0.887
Absorbance after 5 minutes	0.898
Absorbance after 10minutes	0.902
Absorbance after 15minutes	0.858

Table (4):Summary of optimum conditions of drug - $KMnO_4$

Studied Conditions	Optimum Conditions
effect of NaOH concentration	1.2 X10 $^{-1}$ M
effect of $KMnO_4$ concentration	1 X10^{-3} M
oxidation time	10 min

I.3-Calibration curve

At the optimum conditions, the calibration graphs were constructed (Fig 3) .The linear range for TPH is (1–12ppm). The important statistical and analytical results are summarized in table 5.

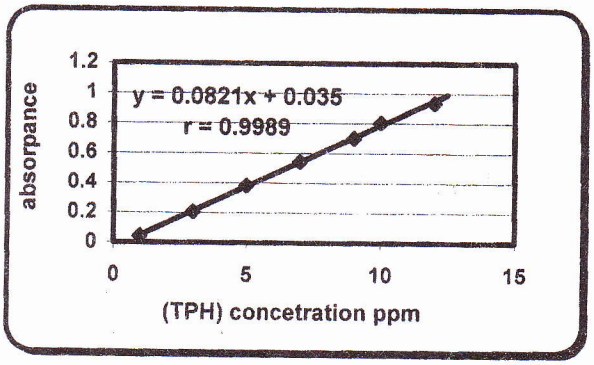


Fig (3):- Calibration curve at 612 nm for TPH -$KMnO_4$

Table (5): λ_{max}, Sandell`s index (µg / cm^2),ε (L / mol. Cm)[7], r^2,r

λ_{max}	S(µg / cm^2)	ε(L / mol. Cm)	r^2	r
612	0.0122	26993	0.9978	0.9989

Table (6): Accuracy ,precision[8,9] and detection limit

λ_{max} nm	Prepared quantity ppm n=3	Measured quantity ppm	RSD	Rec%	E_{re}%	D.L ppm
612	5	4.77	0.544	95.4	3.77	0.49

From the above table it was found that the sensitivity is very high according to the lower detection limit , comparsion of calculated (t) test with tabulated (t)test at 95%confidence was (3.18) illustrate the method used has high value which is (42.59) and high accuracy

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Table (7): Application of drug-$KMnO_4$ method for determination of samafied syrup & tablet (SDI/Iraq).

Pharmaceutical	λ_{max}	Conc.ppm Prepared	measured	Rec.%	Erel.%
Syrup	612 nm	4.50	4.33	96.22	-3.77
Tablet	612nm	10.00	9.77	97.70	2.3

II:drug - $K_2Cr_2O_7$ Spectra

II.A- primary test :

1.0 ml of 0.01M $K_2Cr_2O_7$ was mixed with 1.0 ml of 0.1M HCl and1.0ml of 100ppm TPH in 10ml volumetric flask and complete to the mark by distilled water then measure absorption against the water (blank) at wave length 350 nm the absorption spectrum as Fig (4)

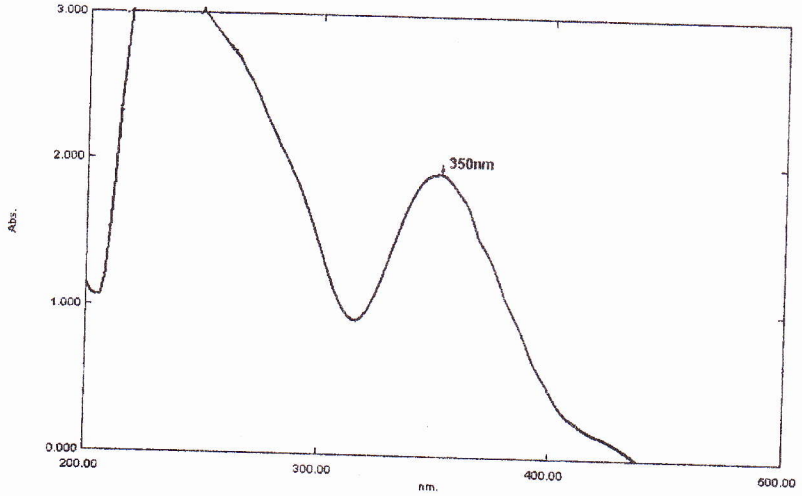

Fig (4):- Absorption spectrum of TPH - $K_2Cr_2O_7$

B- Optimum conditions

The following tables illustrate the optimum condition of NaOH and $K_2Cr_2O_7$ conc.In adition to optimum oxidizing time that give best absorbance .

Table (8): Effect of HCl concentration

Conc. of HCl	A
$1X10^{-3}$ M	0.093
$2 X10^{-3}$ M	0.172
$3 X10^{-3}$ M	0.272
$4 X10^{-3}$ M	0.403
$5 X10^{-3}$ M	0.560
$7 X10^{-3}$ M	0.811
$8 X10^{-3}$ M	0.853
$9 X10^{-3}$ M	0.892
$1 X10^{-2}$ M	0.923
$1.2 X10^{-2}$ M	0.906

Table (9): Effect of $K_2Cr_2O_7$ concentration

Conc. of $K_2Cr_2O_7$	A
$1X10^{-4}$ M	0.127
$2X10^{-4}$ M	0.185
$4X10^{-4}$ M	0.341
$6 X10^{-4}$ M	0.634
$8 X10^{-4}$ M	0.793
$9 X10^{-4}$ M	0.352
$1 X10^{-3}$ M	0.923
$1.2 X10^{-3}$ M	0.947
$1.3 X10^{-3}$ M	0.960
$1.5 X10^{-3}$ M	0.979
$1.7 X10^{-3}$ M	0.952

Table (10): Oxidation time

Absorbance at preparation	0.979
Absorbance after 5 minutes	0.982
Absorbance after 10minutes	0.991
Absorbance after 15minutes	0.996
Absorbance after 20minutes	0.990

Table (11):Summary of optimum conditions of drug - $K_2Cr_2O_7$

Studied Conditions	Optimum conditions
effect of HCl concentration	$1X10^{-2}$ M
effect of $K_2Cr_2O_7$ concentration	$1.5\ X10^{-3}$ M
oxidation time	15 min

3-Calibration curve

At the optimum conditions, the calibration graphs were constructed (Fig5). The linear range for TPH is (1–12ppm). The important statistical and analytical results are summarized in table12

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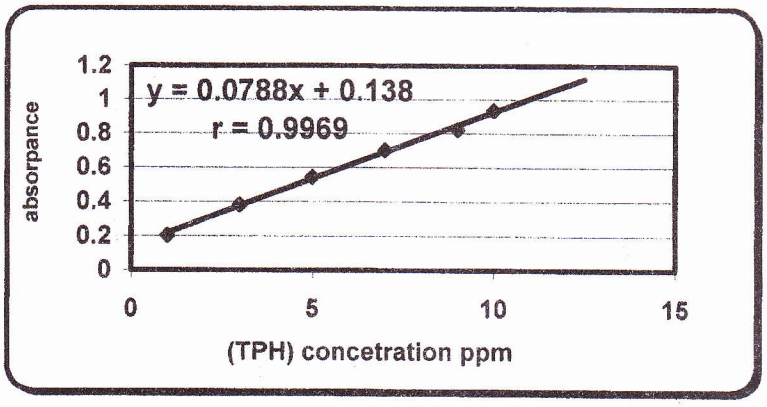

Fig (5):- Calibration curve at 350 nm for TPH - $K_2Cr_2O_7$

Table (12): λ_{max}, Sandell's index ($\mu g / cm^2$),ε (L / mol. Cm)[7], r^2,r

λ_{max}	S($\mu g / cm^2$)	ε(L / mol. Cm)	r^2	r
350nm	0.0086	38559.50	0.9938	0.9969

Table (13): Accuracy ,precision[8,9] and detection limit

λ_{max}	Prepared quantity ppm n=3	Measured quantity ppm	RSD	Rec%	E_{re}%	D.L ppm
350nm	5	5.13	0.544	102.60	2.72	0.49

From the above table it was found that the sensitivity is very high according to the lower detection limit , comparsion of calculated (t) test with tabulated (t)test

at 95%confidence was (3.18) illustrate the method used has high value which is (25.33) and high accuracy.

Table (14) :Application of drug-$K_2Cr_2O_7$ method for determination of samafied syrup & tablet (SDI/Iraq).

Pharmaceutical	λ_{max}	Conc.ppm		Rec.%	Erel.%
		Prepared	measured		
Syrup	350nm	4.50	4.26	94.66	-1.55
Tablet	350nm	10.00	10.30	103.00	-3

III : F test

In determination comparison of TPH between two producers it was found that F value as in table (15) .

Table (15): F test calculated

drug	(F) table test of 95%	F test
TPH	6.26	0.875

From the above table the F value calculated is lower than that of tabulated value ; which means there's no difference between two methods [12].

Conclusions

TPH drug give high result to form colour compound with both oxidizing agent potassium permanganate & potassium dichromate, wich means that we develope two new methods for determining this drug .

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