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Development Of Two Different Spectrophotometric Methods For The Determination Of Atropine Drug In Pure Form And Pharmaceutical Preparations

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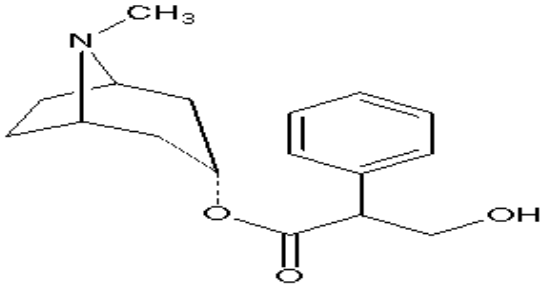
Abstract

Two methods have been applied for the spectrophotometric determination of atropine, in bulk sample and in dosage form. The methods are accurate, simple, rapid, inexpensive and sensitive. The first method depending on the extraction of the formed ion-pair complex with bromphenol blue (BPB) as a chromogenic reagent in chloroform, use phthalate buffer of pH 3.0; which showed absorbance maxima at 413 nm against reagent blank. The calibration graph is linear in the ranges of 0.5-40 µg.mL^{-1} with detection limit of 0.363µg.mL^{-1}. The second method depending on the measure of the absorbance maxima of the formed charge-transfer complex with 2,3-dichloro-5,6-dicyano-p-benzoquinone (DDQ) at 457 nm against reagent blank; with linearity range 2.5-50.0 µg.mL^{-1}, and detection limit of 2.143 µg.mL^{-1}. The results show the absence of interferences from the excipients on the determination of the drug. The proposed methods have been successfully applied for the determination of atropine in pharmaceutical preparations.

Keywords: Spectrophotometric, atropine, ion-pair, charge-transfer.

Introduction

Atropine(Scheme 1), was first isolated as an active principle from the roots of belladonna in 1831 by K. Mein, a German apothecary, [1], This compound, which have the chemical structure of tropane alkaloids[2], has two main types of actions, one on the central nervous system to cause respiratory stimulation, and the other, to suppress smooth muscles and secretary glands innervated by parasympathetic nerves[3]. It had been used as ingredients in many gastrointestinal drugs owing to their anticonvulsant and analgesic properties [1, 4]. Also it was used for bradicardia, following myocardial infection or over dosage of β-blockers and can be produced by typical application of anti cholinergic agent for treatment of irititis causing paralysis of ciliary muscle, leading to blurred vision [5]. For most of the alkaloids have special and distinct physiological properties and toxicity, the determination of atropine is of great importance not only in clinical application but also in pharmaceutical analysis.



Scheme 1: **The chemical structure of atropine**

Several methods have been reported for the determination of atropine in bulk and pharmaceutical dosage forms, these methods include high performance liquid chromatography [6-8] gas chromatography [9], potentiometry[10], Flow-injection post chemiluminescence [11] and thin-layer scanning method[12]. Some of these methods are time-consuming, tedious, and/or dedicated to sophisticated and expensive analytical instruments.

Spectrophotometry[13-19]; are most convenient techniques because of their inherent simplicity, adequate sensitivity, low cost and wide availability in all quality control laboratories.

The present work describes the utility of BPB and DDQ reagents for spectrophotometric determination of atropine in pure form as well as in dosage form. In addition, the optimization of chemical dependent variables of affecting absorbance has been studied.

Apparatus:

A Cintra 5 spectrophotometer with 1 cm quartz cells was used for absorbance measurements. PH-meter DW-9421 from Philips instrument, a Sartorius BL 210S balance, and a Pentium 4 computer (DELL) was used for data processing.

Experimental

Material and Reagents:

All Chemicals used were of analytical reagent grad unless otherwise is mentioned, Atropine sulfate standard powder (purity 99.8%) were kindly provided by the State Company for Drug Industries and Medical Appliances, Samara-Iraq (SDI).

Bromophenol blue (BPB) (Aldrich), 0.1% (w/v) solution was prepared by dissolving 0.1 g of the dye in 5 mL of methanol and then the solution was diluted to a final volume of 100 mL with distilled water. Working solutions were freshly prepared by subsequent dilutions.

2,3-dichloro-5,6-dicyano-p-benzoquinone (DDQ)(BDH); 0.1 %(w/v) solution was prepared by dissolving 0.01 g of the DDQ in 5 mL of acetonitrile and then the solution was diluted to a final volume 10 mL with acetonitrile. Working solutions were freshly prepared by subsequent dilutions. This solution is prepared daily using red- glass volumetric flask because it is a light sensitive reagent.

Hydrochloric Acid (Aldrich), ~0.1 M, a 0.85 mL of concentrated hydrochloric acid (37%, sp.gr1.18) was added to 50 mL distilled water and diluting to the mark in a 100 mL calibrated flask.

Potassium Hydroxide (fluka), ~ 0.1 M, was prepared by dissolving 0.56 g of potassium hydroxide in 25 mL distilled water and diluted to 100 mL in volumetric flask with distilled water.

Phthalate buffer 0.2M solution was prepared by dissolved 4.08 g of potassium hydrogen phthalate (MERCK) 25 mL distilled water and diluted to 100 mL in volumetric flask with distilled water, the pH was adjust to 5.5 by using few drops of 0.1M HCl and\or 0.1M KOH.

Atropine standard solution (250µg.mL^{-1})

For BPB method

It was prepared by dissolving weighed amount of salt equivalent to 25 mg of atropine base in 20mL distilled water and diluting to 100mL in a volumetric flask with distilled water. Working solutions were freshly prepared by subsequent dilutions.

For DDQ method

An accurately weighed amount of atropine salt equivalent to 25 mg of the base was dissolved in 20 ml distilled water. The solution was quantitatively transferred into a separating funnel, made alkaline (pH=9) with ammonia solution [20, 21] and shaken with four 20 ml portions of chloroform. The extracts were pooled by filtration through a filterer paper

containing anhydrous sodium sulphate into a 100 ml standard flask and made up to volume with chloroform. Working solutions were freshly prepared by subsequent dilutions.

General recommended procedure

For BPB method

A suitable amount of atropine standard solution was transferred into a series of 50 mL separating funnels, to each funnel 0.5 mL of phthalate buffer of pH 3.0 and 0.3 mL of 0.05% BPB reagent solutions were added. The separating funnels were shaken vigorously with 5 mL chloroform for 4 mints. The two phases were then allowed for clear separation and the absorbance of the yellow colored organic phase was measured at 413nm against a reagent blank prepared similarly without addition of atropine. The calibration graph was constructed by plotting the measured absorbance of the organic phase against the drug concentration.

For DDQ method

A Suitable volume of the standard stock solution of the drug were pipette into 5-mL calibrated flasks, 0.2 ml of 0.1% DDQ solution was added to each, and then diluted to volume with acetonitrile. Absorbance measurements of resulting solutions were done at the wavelength of maximum absorption at 457 nm against reagent blank which prepared by the same manner, but without addition of atropine.

Solution for the analysis of atropine in pharmaceutical preparations [22,23]

I. In Ampoules

For BPB method

The contents of 15 ampoules were mixed well. A volume equivalent to 10 mg of atropine base was quantitatively transferred into 50 mL volumetric flask and diluted to the mark with distilled water. Working solutions were freshly prepared by subsequent dilutions and analyzed by the recommended procedures.

For DDQ method

The contents of 15 ampoules were mixed well. A volume equivalent to 10 mg of atropine base was quantitatively transferred into 20 mL volumetric flask and diluted to the mark with distilled water, quantitatively transferred it into a separating funnel, made alkaline(pH=9) with ammonia solution, extract the drug base as under (standard solution). Working solutions were freshly prepared by subsequent dilutions and analyzed by the recommended procedure.

II. In Eye Drops

For BPB method

The volume of 5 drops was quantitatively transferred into 100 mL volumetric flask and diluted to the mark with distilled water. A volume equivalent to 25 mg of atropine base was transferred into 100 mL volumetric flask and diluted to the mark with distilled water. Working solutions were freshly prepared by subsequent dilutions and analyzed by the recommended procedure.

For DDQ method

The volume of 5 drops were quantitatively transferred into 100 mL volumetric flask and diluted to the mark with distilled water. A volume equivalent to 25 mg of atropine base was quantitatively transferred into a separating funnel, made alkaline (pH=9) with ammonia solution. Extract the drug base as under (standard solution).Working solutions were freshly prepared by subsequent dilution sand analyzed by the recommended procedure.

Results and discussion

Spectrophtometric procedures are popular for their sensitivity in the assay of drugs and hence, ion pair and charge transfer complexes formation has received considerable attentions for the quantitative determination of many pharmaceutical compounds [24-29].

Atropine reacts with BPB in acidic buffer to give yellow color chloroform soluble ion-pair complex, which exhibits absorption maxima at 413 nm against their reagent blank; (Figure1).

Some amines salts don't react with π-acceptors because they don't possess a lone pair of electrons[29]. Similarly, atropine sulfate unable to react with DDQ; unless it is extracted with chloroform in basic medium[29], resulting formation of atropine base in the chloroform layer; which acts (as n-donors) react with DDQ (as π-acceptors) to give red brown color acetonitrile soluble charge transfer complex, which exhibits absorption maxima at 457 nm against their reagent blank (Figure2). Under the experimental conditions the reagent blank showed in both cases negligible absorbance thereby permit good analytical conditions for quantitative determination of atropine in pharmaceutical dosage forms.

For BPB method

Effect of pH

In order to establish the optimum pH range, atropine was mixed with specified volumes of phthalate buffer. The pH was then adjusted to a value between (2.0 -4.5) with few drops of 0.1M KOH or 0.1M HCl. It was noticed that maximum color intensities and constant absorbance values were found at pH 3.0 (Figure 3). Low absorbencies were observed in solutions with higher or low pH than the optimum value. Hence, a pH of 3.0 was used in all the subsequent experimental work.

Effect of reaction time

The optimum reaction time was determined by following the color development at ambient temperature (25±2). It was found that the reaction was instantaneous. Hence the product attained maximum and constant absorbencies immediately after atropine have been mixed with BPB and the developed color, remained strictly unaltered for at least 24 hours.

Effect of reagent volume

The influences of reagent volume on the absorbance of complex are illustrated in (Figure 4). 0.3 mL of 0.05% solutions of BPB were found to be optimum to develop the maximum color intensities for atropine ion-pair complex, after which no more increase in absorbance values was obtained; therefore, the cited volume of BPB solution were used.

Effect of shaking time

The optimum shaking times for the complete extraction of the formed ion pair complex with chloroform were studied for the period of 1-5 minutes (Table 1). It was found that the optimum shaking times for complete extraction of atropine ion pair complex, at room temperature for minutes.

Effect of the extraction solvent:

Several organic solvents, such as, chloroform, toluene, carbon tetrachloride, benzene, 1, 2-dichloroethane and Dichloro methane were examined for their ability to extract the drug-BPB ion-pair complex. It was found to be chloroform the most suitable solvent in terms of extraction efficiency (Table 2). On the other hand, it was observed that only a single extraction with 5 mL portion of chloroform was adequate to achieve a quantitative recovery of the complex.

For DDQ method

Effect of pH

The effect of pH on the development of the colored complex, between the cited drug and DDQ was investigated by adjusting the pH to a value between 6.0 and 10 with few drops of 0.2M NH_4OH or 0.1M HCl. It was noticed that maximum color intensities and constant absorbance values were found at pH 9.0 (Figure 5). Low absorbencies were observed in solutions with higher or low pH than the optimum value. Hence, a pH of 9.0 was used in all the subsequent experimental work.

Effect of reaction time:

It was found the reaction was instantaneous. Hence the product attained maximum and constant absorbancies immediately after atropine have been mixed with DDQ and the developed color, remained strictly unaltered for at least 8 hours in drake place.

Effect of reagent Volume

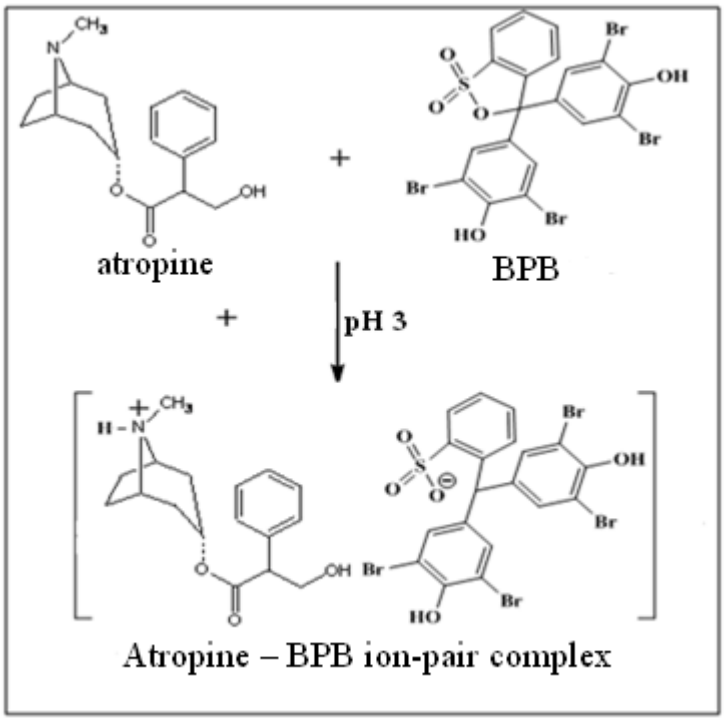
The effect of the volume of DDQ on the color development was studied by adding different volumes(0.05- 0.30) mL of 0.1%DDQ solution to 20µg.ml of atropine .The results revealed the fact that 0.2 ml of 0.1%DDQ solution was required to achieve the maximum intensity of the color (Figure 6).

Effect of solvent

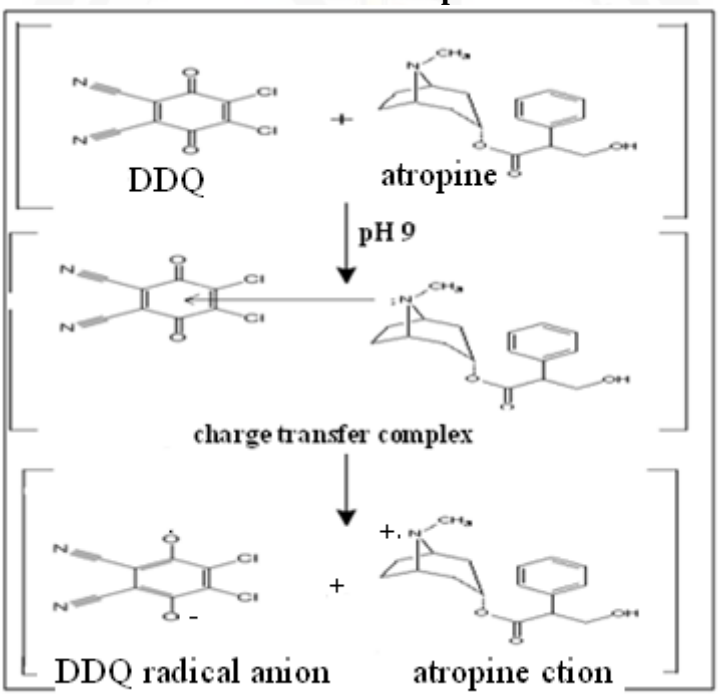
Several organic solvents such as acetonitrile , acetone, methanol, chloroform, 1,2-dichloro ethane, and dichloro methane was studied to choose the preferred diluting solvent for the quantitative measurements.(Table3). The results show that Acetonitrile was considered as an ideal diluting solvent as it gives good solvating capacity for atropine, and gives the highest yield of the radical anion.

Stoichiometry of the complexes

To establish molar ratio for both complexes, Job's method of continuous variation has been used (Figures 7 and 8). The results showed that 1:1 ratios for both complexes were formed; through the electrostatic attraction between the positive protonated atropine with the anion of BPB for ion pair complex formation[30], and the complex between the studied drugs, as n-donors, with DDQ, as π acceptors for charge transfer complex formation[20],(Scheme 1 and 2).



Scheme 2: Proposed reaction pathway between atropine –BPB ion pair complex, under recommended procedure.



Scheme 3: Proposed reaction pathway between atropine –DDQ charge transfer complex. under optimum recommended procedure.

Calibration graphs:

Employing the experimental conditions, linear calibration graphs for both complexes; were obtained (Figures 9 and 10), which show that Beer's law was obey in the concentration range of 0.5-40 and 2.5-50 $\mu g.mL^{-1}$ for atropine BPB ion pair and atropine DDQ charge transfer complexes respectively.

Spectral characteristics of the proposed methods:

According to the optimum experimental conditions of the proposed methods, the regression plots showed linear dependence of absorbance signals on the concentrations of the studied drug in the range given. The regression equations, correlation coefficients, molar absorptivities, detection limits and sandell sensitivities in addition to other parameters are given in Table 4.

Accuracy and precision:

The accuracies of the proposed methods were confirmed by analyzing five replicate analyses of three different amounts of the drug (within Beer's law) by calculating the relative error percentage (Table 5). The results indicated good accuracies for both of the methods. The precision was determined in each case by calculating the percentage relative standard deviation (RSD %) for five determinations at each of the studied concentration level and were found to be in the range of 1.352-1.826% and 1.778-2.103% for atropine BPB ion pair and atropine DDQ chrge transfer complexes respectively. The values of the mean error(x_i-μ) were less than the values of indeterminate error ($\pm ts/\sqrt{n}$), indicating that no significant differences between the mean and the true values; at 95% confidence level.

Interferences Study:

The results showed that no interferences were found in the presence of 250 μg of the studied excipients (lactose, sucrose, starch, glucose, magnesium stearate, sodium citrate, and sodium chloride) in the determination of atropine for both methods, (Table 6).

Analysis of dosage forms:

It is evident from the aforementioned results that the proposed methods gave satisfactory results with the investigated drug. Thus, their pharmaceutical dosage forms were subjected to analysis of their contents of the active ingredient by the proposed methods (ion-pair and charge transfer complexes formation). The results given in (Table 7 and 8) were satisfactory.

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Table(1): Effect of shaking time on extraction of 20 µg.mL^{-1} Atropine; 0.3mL of 0.05%BPB, pH(3.0).

Shaking time(minute)	Absorbance
1	0.4121
2	0.4168
3	0.4202
4	0.4329
5	0.4325
6	0.4325

Table (2): Effect of type of extraction solvent on absorbance of 20 µg.mL^{-1} atropine; 0.3mL of 0.05%BPB, pH(3.0).

Extraction solvent	Absorbance
Chloroform	0.4329
Toluene	0.0221
Carbontetrachloride	0.0297
Benzene	0.0096
1,2-Dichloro ethane	0.3054
Dichloro methane	0.2153

Table (3): Effect of type of organic solvent on absorbance of 20 µg.mL^{-1} atropine; 0.2mL of 0.1%DDQ, pH(9.0).

Organic Solvent	Absorbance
Acetonitrile	0.3031
Acetone	0.2322
Methanol	0.0799
Chloroform	0.1413
1,2-dichloroethane	0.0976
Dichloro methane	0.2155

Table(4): Spectral characteristics and statistical data of the regression equations for determination of atropine by ion–pair and charge transfer complexes formation.

Parameter	Ion–Pair Complex Formation	Charge Transfer Complex Formation
λ_{max} (nm)	413	457
Color	Yellow	Red-brown
Linearity range (µg.mL^{-1})	0.5 – 40	2.5 – 50
Molar absorpitivites(l.mol^{-1}.cm^{-1})	5787.6	4051.32
Regression equation	A = 0.020 x + 0.037	A= 0.014x + 0.012
Calibration Sensitivity	0.020	0.014
Sandell's Sensitivity(µg.cm^{-2})	50.000	71.439
Correlation of Linearity (R^2)	0.9998	0.9982
Correlation coefficient (R)	0.9999	0.9991
Detection limit (µg.mL^{-1})	0.363	2.143

Table (5): Evaluation of accuracies and precisions of the two proposed procedure.

Method	Drug Concentration (µg.mL^{-1})		Relative Error %	R.S.D.* %	x_i-µ	±ts/√n
	Taken	Found*				
Ion–Pair Complex	2.5	2.488	-0.480	1.352	-0.012	0.039
	10	9.932	-0.680	1.414	-0.068	0.161
	30	30.205	+0.683	1.826	+0.205	0.634
Charge Transfer Complex	5	4.962	-0.760	1.833	-0.038	0.105
	20	19.871	-0.645	1.778	-0.129	0.406
	40	40.283	+0.708	2.103	+0.283	0.974

*Average of five determinations

t = 2.571 for n=5 at 95% confidence level.

Table(6): Percent recovery for 20 µg.mL^{-1} of atropine in the presence of 250 µg.mL^{-1}of Excipients by ion–pair and charge transfer complexes formation.

Excipients	Ion–Pair Complex Formation Method		Charge Transfer Complex Formation Method	
	Conc. Fund (µg.mL^{-1})	Recovery%	Conc. Fund (µg.mL^{-1})	Recovery%
lactose	19.779	98.895	19.828	99.140
Sucrose	20.231	101.155	20.197	100.985
Starch	19.888	99.444	20.302	101.510
Glucose	19.862	99.310	20.340	101.700
Magnesium Stearate	20.316	101.580	19.863	99.315
Sodium Citrate	19.803	99.015	20.265	101.325
Sodium Chloride	20.334	101.670	19.798	98.990

*Average of three determinations.

Table (7): Spectrophotometric determination of atropine in pharmaceutical compounds by ion–pair complex formation.

Ion–Pair Complex	Drug Concentration (µg.mL^{-1})		Recovery%	R.S.D.* %
	Taken	Found*		
ATROPINE Sulfate (1mL Ampoules) 1mg/1mL PAYAL - Uk	2.5	2.555	102.200	1.572
	10	9.989	99.890	1.521
	30	30.562	101.873	1.884
ATROPINE Sulfate (1mL Ampoules) 1mg/1mL BELCO - India	2.5	2.576	103.040	1.776
	10	9.883	98.830	1.902
	30	29.505	98.350	2.091
Atropina 0.5% 8mL Eye Drops Atropine sulfate 5mg/1mL Dalta Co.Syria	2.5	2.468	98.720	1.769
	10	9.876	98.760	1.814
	30	29.658	98.860	2.143
Apitropine1% 10mL Eye Drops Atropine sulfate 10mg/1mL API - Jordan	2.5	2.622	104.88	1.852
	10	10.264	102.640	2.056
	30	30.792	102.650	2.442

*Average of five determinations.

Table (8): Spectrophotometric determination of atropine in pharmaceutical compounds by charge transfer complex formation.

Charge Transfer Complex	Drug Concentration (µg.mL^{-1})		% Recovery	R.S.D.* %
	Taken	Found*		
ATROPINE Sulfate (1mL Ampoules) 1mg/1mL PAYAL - Uk	5	5.146	102.900	1.989
	20	20.315	101.575	2.183
	40	40.472	101.180	2.552
ATROPINE Sulfate (1mL Ampoules) 1mg/1mL BELCO - India	5	5.178	103.560	2.286
	20	20.617	103.085	2.602
	40	40.992	102.480	2.814
Atropina 0.5% 8mL Eye Drops Atropine sulfate 5mg/1mL Dalta Co.Syria	5	5.123	102.460	1.792
	20	20.287	101.435	2.521
	40	40.790	101.975	2.746
Apitropine1% 10mL Eye Drops Atropine sulfate 10mg/1mL API - Jordan	5	5.222	104.440	2.473
	20	20.451	102.255	2.756
	40	41.243	103.108	2.967

*Average of five determinations.

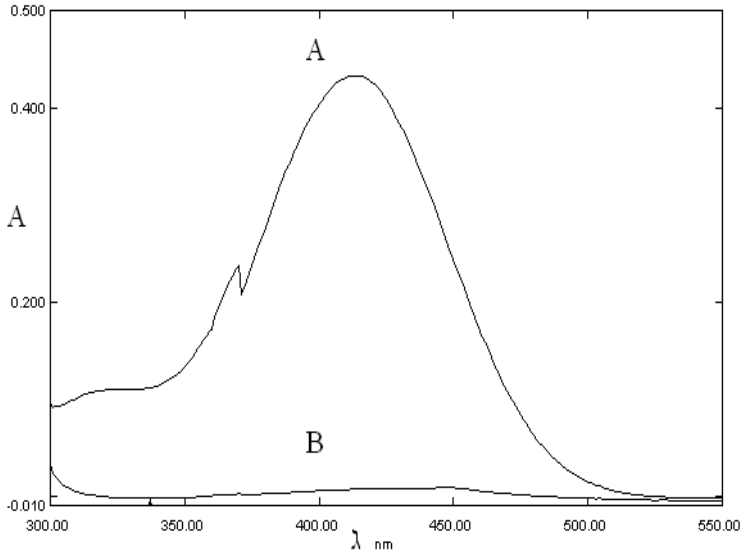

Fig. (1): Absorption spectra of A: 20 µg.mL^{-1} atropine –BTB Ion –Pair Complex against reagent blank, B: reagent blank against chloroform, under optimum conditions.

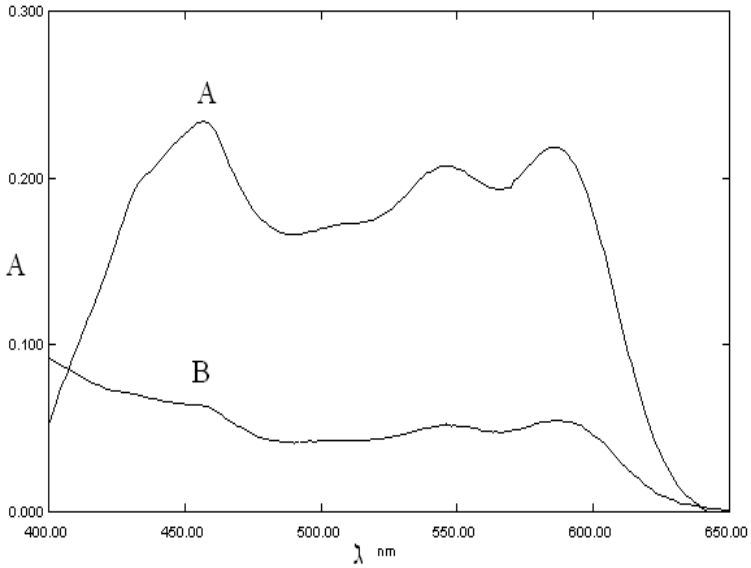

Fig. (2): Absorption spectra of A: 15 µg.mL^{-1} atropine–DDQ charge transfer complex, against reagent blank, B: reagent blank against acetonitrile, under optimum conditions.

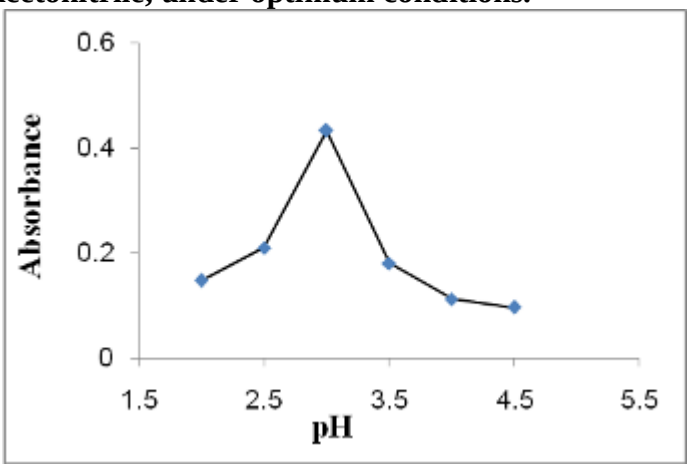

Fig. (3): Effect of pH on the absorbance of 20 µg.mL^{-1}atropine; 0.05% BPB.

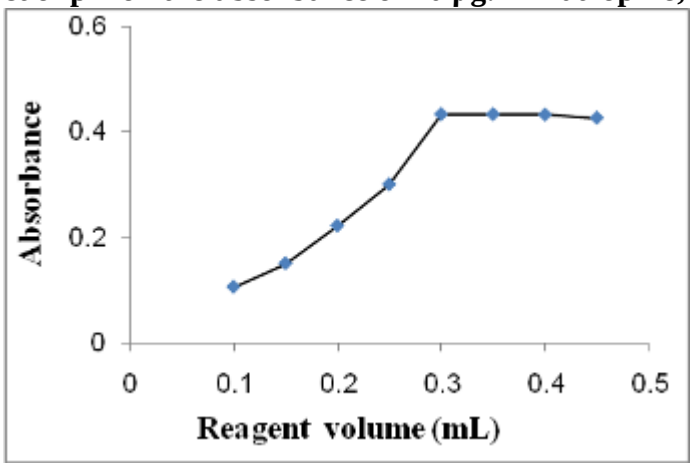

Fig. (4): Effect of reagent volume (0.05% BPB) on the absorbance of 20 µg.mL^{-1} atropine; pH 3.0

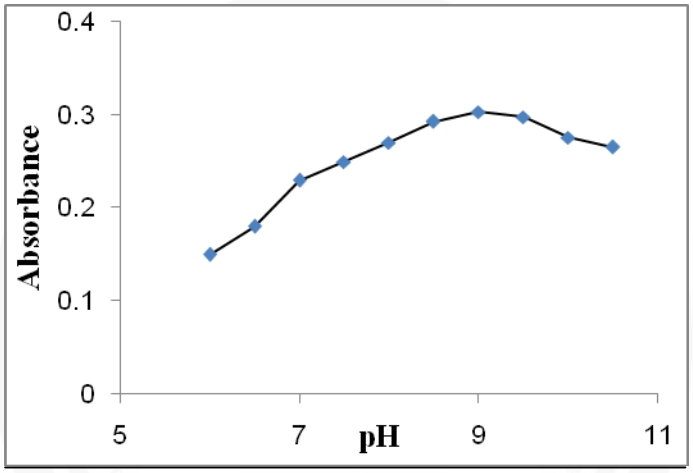

Fig. (5): Effect of pH on the absorbance of 20 µg.mL^{-1} atropine; 0.1% DDQ.

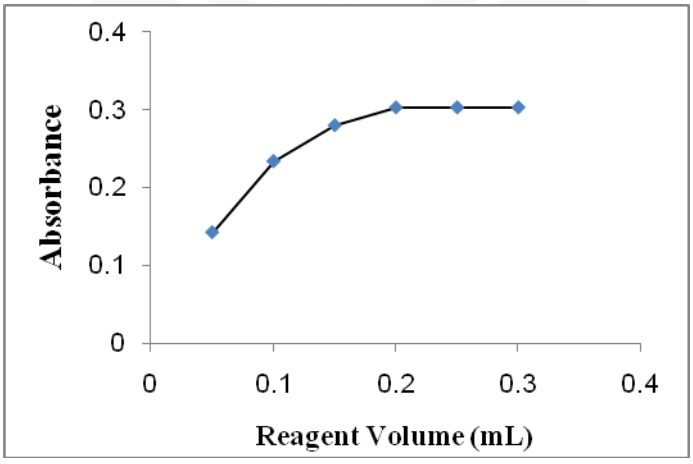

Fig. (6): Effect of reagent volume (0.1% DDQ) on the absorbance of 20 µg.mL^{-1} atropine; pH 9.0

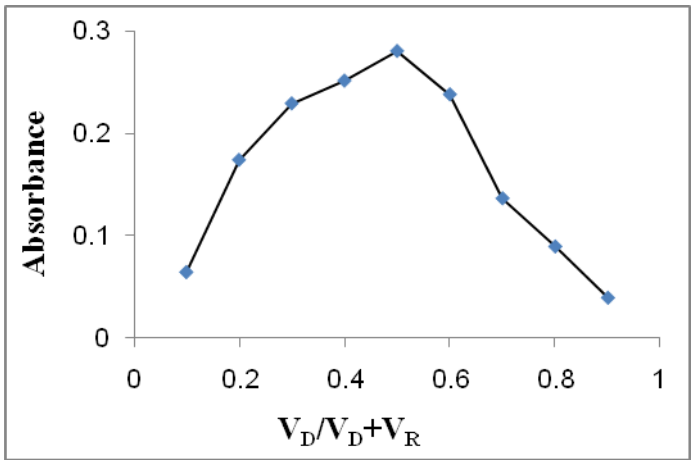

Fig. (7): Continuous variation of atropine –BPB ion pair complex, (each $3.456x10^{-4}$M), pH 3.0.

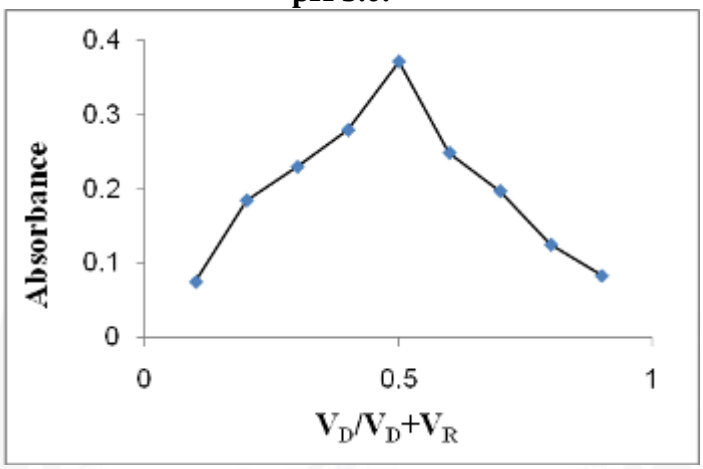

Fig. (8): Continuous variation of atropine –DDQ charge transfer complex, (each $3.456x10^{-4}$M), pH 9.0

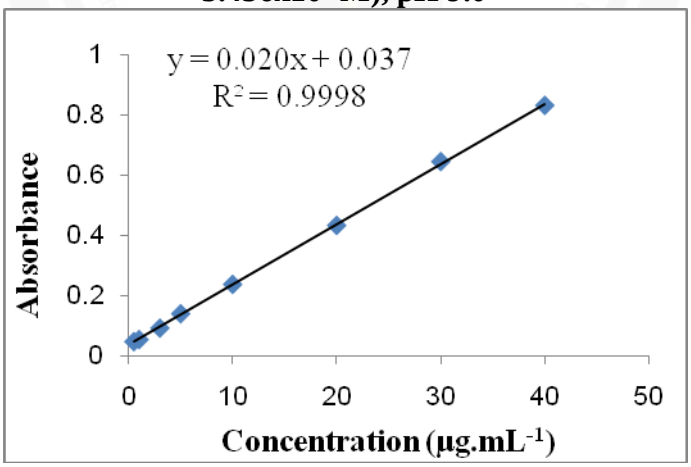

Fig. (9): Calibration graph of atropine-BPB ion-pair complex, under optimum recommended Procedure.

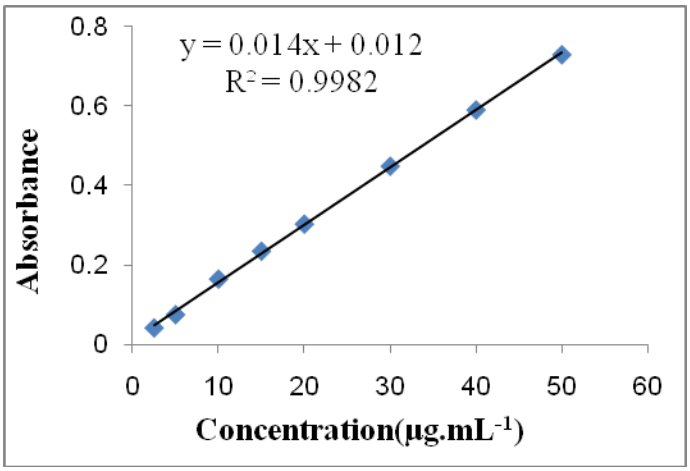

Fig. (10): Calibration graph of atropine –DDQ charge transfer complex, under optimum recommended Procedure.

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تطوير طريقتين طيفيتين مختلفتين لتقدير دواء الأتروبين بصورته النقية وفي المستحضرات الصيدلانية

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قسم الكيمياء ، كلية التربية - ابن الهيثم ، جامعة بغداد

استلم البحث في : 15 نيسان 2012 قبل البحث في : 17 حزيران 2012

الخلاصة

اقترحت طريقتان طيفيتان لتقدير دواء الأتروبين في عينات نقية وبعض المستحضرات الصيدلانية بالاعتماد على تكوين معقدات الأزدواج الأيوني و أنتقال الشحنة. كانت الطريقتان اعلاه دقيقة ، وبسيطة، وسريعة، وغير مكلفة وحساسة. اعتمدت الطريقة الاولى على استعمال الكلوروفورم في أستخلاص معقد الازدواج الايوني المتكون بين العقار اعلاه والكاشف بروموفينول الازرق من وسط مائي عند دالة حامضية مقدارها 3.0، أذ اظهر المعقد المتكون اقصى امتصاص له عند الطول الموجي413 نانومتر، ضد محلول الخلب، وأظهر منحني المقايسة علاقة خطية لمدى من التراكيز تراوحت بين 0.5-40 مايكروغرام/مل و بحد كشف 0.363 مايكروغرام /مل. في حين اعتمدت الطريقة الثانية على قياس اقصى امتصاص لمعقد انتقال الشحنة المتكون بين العقار قيد الدراسة(كواهب للالكترونات) و2و3- داي كلورو – 5-6- داي سيانو – بارا- بنزوكوينون (كمستقبل للالكترونات) عند الطول الموجي 457 نانومتر ضد محلول الخلب وبخطية تراوحت بين 2.5- 50 مايكروغرام/مل، وبحد كشف 2.143 مايكروغرام/مل. أظهرت الدراسة أيضاً أن الطريقتين المقترحتين خالية من تأثير المتداخلات المتعارف على وجودها في المستحضرات الصيدلانية، فقد أمكن تطبيق الطريقتين بنجاح لتقدير الأتروبين في بعض تلك المستحضرات.

الكلمات المفتاحية: طيفي، اتروبين، الأزدواج الأيوني ،انتقال الشحنة