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Spectrophotometric method for the microdetermination of methyl and propyl paraben in some detergents through charge transfer complex

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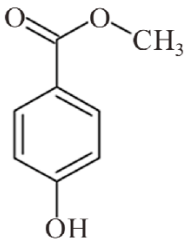
Abstrac

Simple Spectral method was developed to estimate the methyl and propyl paraben through charge- transfer (CT) complexes. The method involves the interaction of parabens compounds with o-chloranil as π-acceptor. Under the optimized conditions, the complexes were found to be absorbed at 415 and 385 nm with in the linearity range of 5-40 and 5-55 µg ml^{-1} respectively. The molar absorptivity values were in the range 3803.75 and 2342.6 $L.mol^{-1}.cm^{-1}$ respectively. And the lower limit of detection were in the rang 0.073 and 0.086 $\mu g/ml^{-1}$ for methyl and propyl respectively. The stoichiometry of the paraben compounds o-chloranil complexes was found to be 1:1 method. The procedure was characterized by its simplicity with accuracy and precision. The proposed method was applied successful in some detergents (shampoo and lotion) containing paraben compounds.

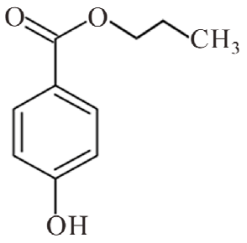
Key words: Spectrophotometric ,methyl and propyl paraben, Charge Transfer Complex, detergents

Introduction

Parabens (4-hydroxybenzoic acid esters) are synthetic chemical preservatives used in a wide range of cosmetic, food and pharmaceutical products because they are broad spectrum, antimicrobial and antifungal agents with a low toxicity to humans, have a good in situstability and are non-volatile [1]. In this work, the separation and quantification of tow commonly used parabens, as methylparaben (MP), and propylparaben (PP) (**Figure 1**), were proposed. These tow parabens were selected because they are the most frequently used in cosmetic products (e.g., creams, skin lotions or gels). Moreover, MP and PP are often used together due to their synergistic effects [2].



Propyl paraben



Methyl paraben

Several analytical methods for the detection and quantification of parabens have already been developed. Chromatographic methods are widely used, especially high- performance liquid chromatography (HPLC) [3] and gas chromatography (GC) [4]. Use of flow injection methods has also been reported [5,6].The present paper reports the spectrophotometric determination of some compound paraben based on their interaction, as *n*-donors, with *o-CA* as *π*-acceptor, in organic solution forming charge transfer complexes.

INSTRUMENTAION

1-A pparatus.

All spectrophotometric measurements are performed on Shimadzu UV V-530, UV Visible recording spectrophotometer,pHmeter (Jenway) 3310, Water bath Memmert – Germany.

2-MATERIALS

Methyl paraben solution(250µg/ml)

Prepared by dissolving 0.025g in small amount of ethanol and the volume was completed to 100ml with distilled water in a volumetric flask.

6,5,4,3, -tetra chloro-ortho-benzokynen (o- Chloranil) Solution(0.005M)

prepared by dissolving 0.123g of Ortho Chloranil in 100 ml of ethanol or methanol or acetone.

Potassium hydroxide solution(0.01M)

prepared by dissolving 0.14g of pure substance in 250ml of distilled water.

Interference solution(1000µg/ml)

prepared by dissolving 0.1g of Interference in amount of ethanol and the volume was completed to 100ml with distilled water in a volumetric flask.

General procedure

Accurately measured suitable volume of methyl, and propyl paraben were transferred from stock solution to 5 ml volumetric flasks and diluted to obtain 5-40 and 5-55 $\mu g.ml^{-1}$ for the compounds mentioned above respectively. To each flask containing compounds in the order mentioned above 0.7, and 0.6ml of *o-CA* and 0.4 and 0.6 ml of KOH were added. The solutions were diluted to the mark with acetone for methyl paraben and with water for propyl paraben. The absorbances were measured at 415 and 385 nm for methyl and propyl paraben versus their respective blanks respectively.

RESULTS AND DISCUSSIONS

Optimal conditions

Variables absorption of the complex formation studied to get the best conditions for the reaction between paraben compounds with o – Chloranil reagent were obtained as the following.

Effect of solvent type

The significant effect of the solvent on the stability of the complex formed between the donor and the acceptor electrons [7], in this study different solvents such as methanol, ethanol, acetone and water as medium for the reaction, used by adding (20µg/ml) of methyl , (15µg/ml) propyl paraben and 0.5ml of 0.005M of o – Chloranil and 0.Σml of potassium hydroxide concentration of 0.01M and volume completed in a 5ml volumetric flask by solvent to the mark. The solutions were kept in the laboratory temperature at (25 C°) for 10 minutes,then measured at the appropriate wavelength, As shown in table1. It was found that on the using water as solvent for methyl paraben and acetone for *o-CA* in the case of methyl and ethanol propyl in the presence of KOH and dilution with the same solvent were gave maximum color intensity.

Table (1) Effect of solvent type in the absorption of a complex methyl paraben - Ortho – Chloranil

Methyl paraben	O-Chloranil Dissolved in	Dilution	Methyl paraben λ_{max}(nm)	Absorbance	Propyl paraben λ_{max}(nm)	Absorbance
Ethanol	Ethanol	Water	-	Negative	-	Turbid
Water	Ethanol	Ethanol	343	0.161	322	0.014
Ethanol	Ethanol	Ethanol	355	0.122	334	0.051
Water	Ethanol	Water	323	0.04	385	0.201
Methano	Methanol	Methano	376	0.03	-	Negative
Water	Methanol	Methano	333	0.015	344	0.02
Methano	Methanol	Water	-	Turbid	-	Negative
Water	Methanol	Water	347	0.211	404	0.013
Water	Acetone	Acetone	415	0.355	366	0.06
Acetone	Acetone	Acetone	404	0.231	371	0.019
Water	Acetone	Water	388	0.163	366	0.017
Acetone	Acetone	Water	390	0.254	-	Turbid

Effect of acetone amount

Table(2) show the effect of acetone amount on the signal absorption, different percentages from 0.0% -100% were used in this study as shown in table (2), the maximum absorbance selected as optiumum concentration of acetone was 60%.

Table (2) Effect of acetone amount

Acetone (%)	0.0	20.0	40.0	60.0	80.0	100.0
Absorbance	0.091	0.155	0.254	0.367	0.342	0.355

Effect of bases solution

Different bases solution were prepared of concentration of 0.01M, Added constant volume of 0.4 ml of these solutions to the methyl paraben and at 0.6 ml propyl paraben and measured absorption at the 415 ,385 nm respectively. the results showing in Table (3).

Table (3) Effect of base types

Base(0.01M)	Absorbance (methyl paraben) 415nm	Absorbance (propyl paraben) 385nm
NaOH	0.388	0.223
KOH	0.402	0.229
Na_2CO_3	0.345	0.211
K_2CO_3	0.336	0.199

From the above table It was found that potassium hydroxide gave maximum color intensity for Methyl and propyl paraben therefor it was recommended for subsequent experiments.

Effect of the pH

Effect of the pH was studied by adding increasing amounts of potassium hydroxide concentration of 0.01M**,** Mixing 20µg/ml of methyl paraben and with 0.5ml of Ortho – Chloranil reagent, then added increasing volumes from the KOH in 5ml volumetric flask and completed to the mark by 60 % of of acetone solution, the high absorption shows at 415nm after leaving the solutions for 10 minutes at a laboratory temperature , then15µg/ml from complex of propyl paraben was mixied with 0.5ml of of Ortho – Chloranil reagent, Added increasing volumes of the base KOH in 5ml volumetric flask and completed to the mark with distilled water and then measured the absorption at 385nm after leaving the solutions for 10 minutes at laboratory temperature and the results showing in the table (4).

Table (4) Effect of pH

Volume of KOH Solution 0.01M(ml)	Absorbance (methyl paraben)415nm	Absorbance (propyl paraben)385 nm	Final pH(Methyl)	Final PH (propyl)
0.0	0.231	0.08	4.1	4.4
0.2	0.277	0.139	5.2	5.6
0.4	0.402	0.188	7.6	6.6
0.5	0.389	0.201	8.3	7.0
0.6	0.379	0.229	9.0	8.1
0.8	0.358	0.157	10.0	8.7
1	0.343	0.144	10.3	9.3

Effect of the amount of Ortho – Chloranil reagent

The amount of Ortho – Chloranil reagent as absorbing complexes , has been added to increasing volumes of a Ortho – Chloranil Solution (0.1-1.0 ml) then to the amount of the methyl paraben 20µg/ml and fixed volume of 0.4 ml of potassium hydroxid solution and15µg/ml propyl the presence of a fixed

volume of 0.6 ml of a potassium hydroxide in 5ml volumetric flask and completed to the mark with the appropriate solvent and the Solutions kept for 10 minutes at a laboratory temperature and measured the absorption and compared to blank solutions for methyl and propyl paraben at 415 ,385nm the result as shown in Table (5).

Table (5). Effect of the amount of Ortho – Chloranil reagen

o-Chloranil(0.005M) (Vol.ml)	Absorbance (methyl paraben) 415nm	Absorbance (propyl paraben) 385nm
0.1	0.092	0.044
0.2	0.130	0.078
0.3	0.226	0.014
0.4	0.311	0.128
0.5	0.402	0.229
0.6	0.478	0.237
0.7	0.480	0.210
0.8	0.432	0.189
0.9	0.382	0.145
1.0	0.333	0.111

Effect of surface active substances

Effect of the active surface substances studied on the intensity of the absorption of paraben - Ortho – Chloranil complexes, including sodium dodecyl sulphate (SDS), cetyltrimethylammonium bromide (CTAB), and Triton x-100 It was found that these surfactants decreased the absorbance of solutionso, so that did not depend in subsequent experiments as shown in the table (6).

Table (6) Effect of surface active substances

Surfactant 0.1%(1.0ml)	Absorbance (methyl paraben) 415nm	Absorbance (propyl paraben) 385nm
Without Surfactant	0.481	0.237
Sodium Dodecyl Sulfate	0.479	0.230
Cetyl Trimethyl ammonium bromide	Turibid	Turibid
Triton X-100	0.392	0.198

Effect of temperature on the stability and time of the complex

The temperature ranged between at room temperature and in thermostatically controlled water-bath at different temperatures.The absorbance was measured at 5 and 10 minutes intervals against reagent blank treated similarly. It was observed that maximum absorbance and stability was obtained at room temperature (25°C) for studied methyl and propyl paraben . It was found that complexes gave maximum absorption within 10-50 and 10-40 minutes for methyl and propyl paraben respectively and the color was fading slowly thereafter, as in the table (7).

Table (7), Temperature effect on the time and the stability formation of parabens - Ortho – Chloranil complexes

Tim	Absorbance (methyl paraben) 415nm			Absorbance (propyl paraben) 385nm		
	Temp(c°)			Temp(c°)		
	25	40	50	25	40	50
1.0	0.299	0.289	0.150	0.099	0.110	0.121
5.0	0.346	0.349	0.376	0.156	0.133	0.126
10	0.480	0.482	0.367	0.237	0.236	0.206
15	0.488	0.479	0.367	0.241	0.235	0.206
20	0.487	0.478	0.355	0.246	0.235	0.204
25	0.485	0.478	0.355	0.245	0.233	0.204
30	0.486	0.476	0.298	0.245	0.227	0.201
40	0.486	0.475	0.288	0.242	0.221	0.199
50	0.486	0.460	0.243	0.233	0.210	0.188
60	0.440	0.455	0.233	0.212	0.124	0.156
90	0.402	0.341	0.231	0.210	0.101	---------
Overnight	0.012	-------	----------	-------	-------	---------

Effect of order of addition

To obtain a higher sensitivity, studeid three order to getting the results as shown in the table (8) and (9).

Table (8) order of addition

Order number	Reaction components	Absorbance Methyl paraben 415nm	Absorbance propyl paraben 385nm
1	A+C+B	0.303	0.139
2	A+B+C	0.488	0.246
3	B+C+A	0.218	0.098

A=Methyl andpropyle paraben , B=o-Chloranil , C=KOH.

In the above table that the second order indicates that the presence of potassium hydroxide increase the sensitivity and this confirms that the potassium hydroxide is the appropriate base medium for complex after transmission of the charge and quickly leads to the formation of free radicals as described in paragraph (The Suggested chemical reaction)

Table (9) Optimum conditions for the determination of methyl and propyl paraben with o-CA reagent

Experimental conditions	Methyl paraben	Propeyl paraben
λ_{max} nm	415	385
O-Chloranil 0.005M (ml)(X ml)	0.7	0.6
KOH (0.01M ,Xml)	0.4	0.6
Temperature(C°) (°C)	25	25
Development time (min)	15	20

Charge transfer electronic spectra.

Absorption Spectra for complexes of paraben compounds as n-donors with Ortho – Chloranil as π-acceptor after demonstrating optimal conditions shown in the table(9) The electronic spectra were scanned against their respective blank reagents.New bands with maximum absorption at 415 and 385 nm methyl and propyl paraben respectively. Fig. (1) and (2) shows the final draw of absorption spectra.

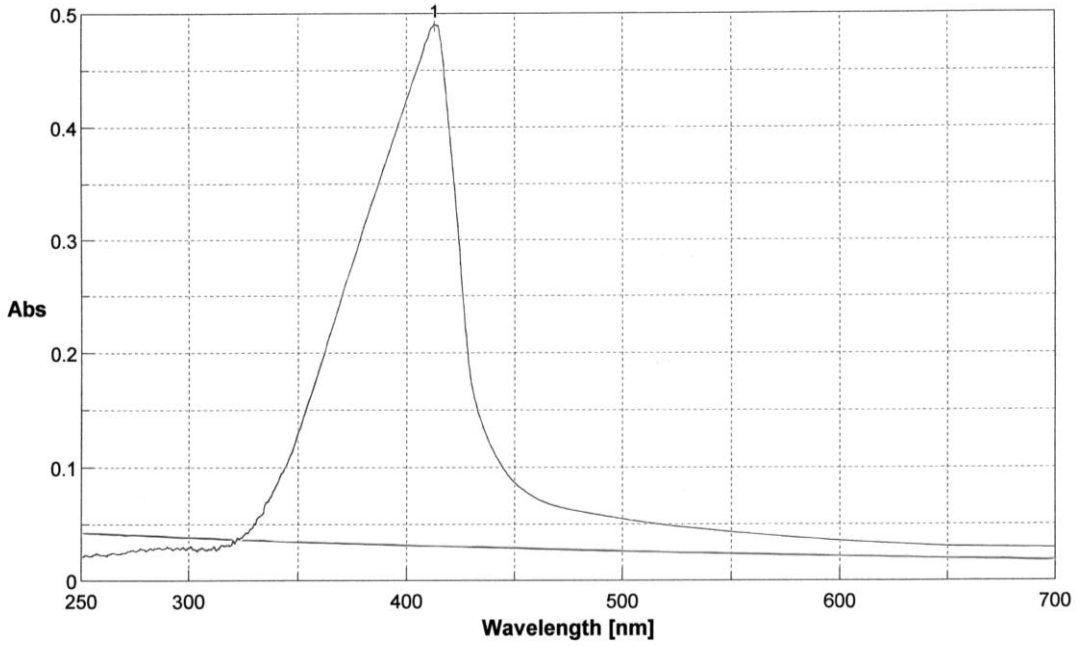


Fig. (1) Absorption spectrum of methyl parabens - Ortho – Chloranil complexes

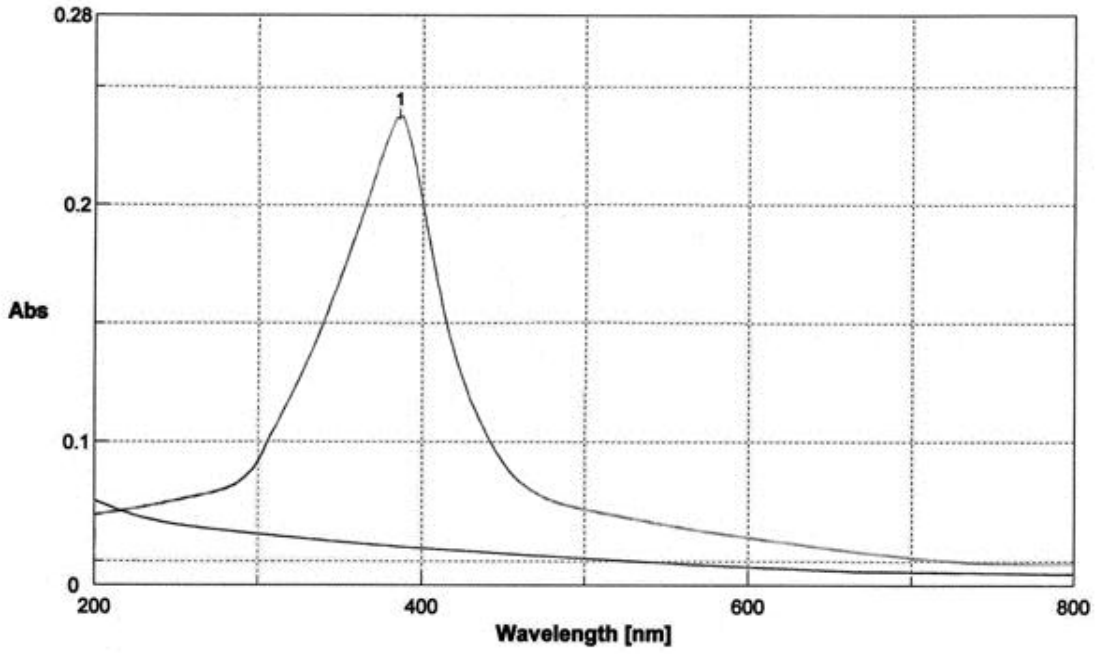


Fig. (2) Absorption spectrum of propyl parabens - Ortho – Chloranil complexes

Calibration curve.

Depending on the optimal conditions, constructed calibration curve to estimate the methyl and propyl paraben as the following: -

To a series of solutions containing increasing amounts of methyl, and probyl paraben and in to 5 ml volumetric flasks and diluted to obtain 5-40 and 5-55 µg ml^{-1} for the compounds mentioned above respectively. To each flask containing componds in the order mentioned above, 0.7,and 0.6 ml of 0.005M o-CA and 0.4and 0.6 ml of0.01M KOH were added. The solutions were diluted to the mark with 60% acetone for methyle and with water for propylparaben. and the Solutions left at a temperature and time shows in table (7), The absorbances were measured at415 and 385 nm respectivel. (Fig 3) and (Fig4) showed that linear standard curves, The Beer's law limits , molar absorptivity ,Sandel and Limit of detection (LOD) values were evaluated and given in table 10.

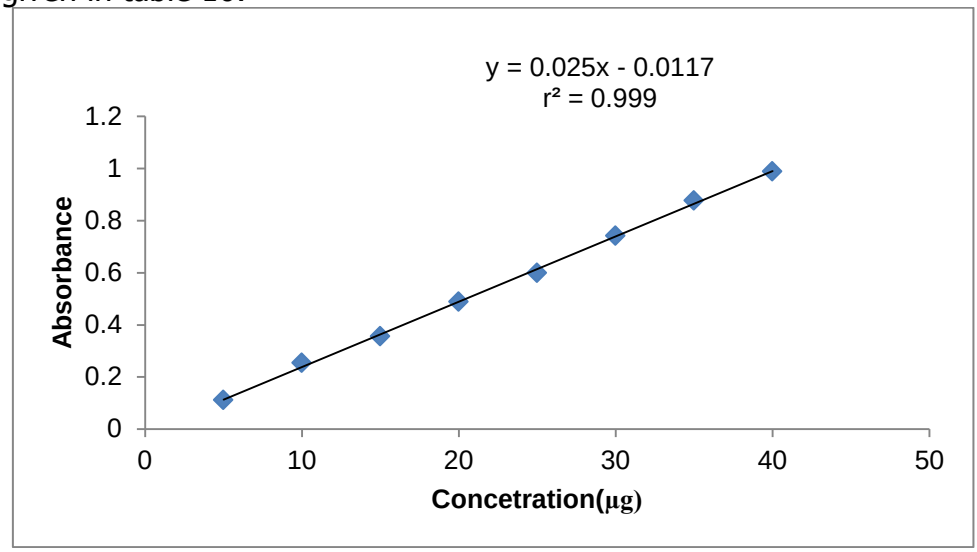


Fig.(3) Calibration curve for methyl paraben determination

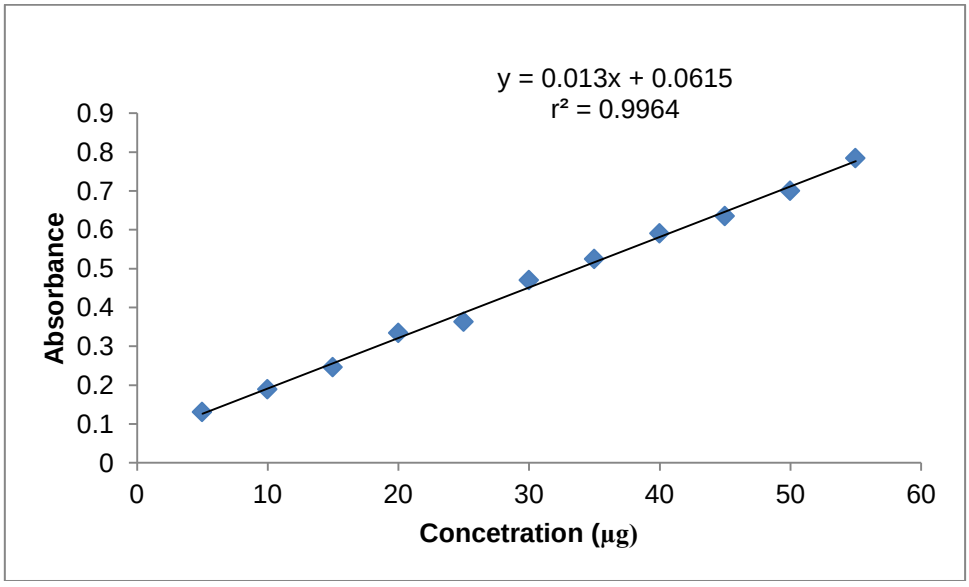


Fig.(4) Calibration curve for propyl paraben determination.

Table (10) Summary of the values of the standard curves of the resulting complexes.

Parameter	Value Methyl paraben	Value propeyl paraben
Linearity range (μg .ml^{-1})	5-40	5-55
r	0.9989	0.9957
r^2	0.9978	0.9914
ε, L mol^{-1} cm^{-1}	3803.75	2342.6
S, μg cm^{-2}	0.040	0.077
Intercept a	0.0117	0.0615
the slope b, mL	0.025	0.013
limit of detection LOD, (μg .ml^{-1})	0.07	0.086
(t) test	77.378>>2.361	46.936>>2.262

Accuracy and precision

To check the accuracy and precision of the calibration curve, methyl and propyl paraben was determined in three different concentrations .The results shown in Table (11) indicated that the calibration curve was satisfactory.

Table (11) Accuracy and Precision

Compo-unds	(μg/ ml)	Abs	Average *	S.d	RSD %	t-test confidence limits 95%	Rer%	Recov-ery%
Methyl paraben	١٠	0.2383	0.2408	0.0087	3.612	0.2408±0. 011	-1.05	101.04
	٣٠	0.7383	0.7374	0.0048	0.654	0.7374±0.005 9	0.0012	99.87
	٤٠	0.9883	0.9872	0.00083	0.085	0.9872±0.001 04	0.0011	99.88
Propyl paraben	٢٠	0.3215	0.3260	0.0073	2.243	0.326±0.0091 0	-1.399	101.39
	35	0.5165	0.5198	0.0047	0.914	0.5198±0.005 8	-0.638	100.63
	٥٥	0.7776	0.7794	0.0042	0.541	0.7794±0.005 2	-0. 231	100.23

*Average of five/determination

Nature of methyl and propyl paraben- Ortho – Chloranil reagent complex.

Job`s method[8]and the mole ratio[9] method have been used in the determination of the reaction ratio of methyl paraben with Ortho – Chloranil reagent.The obtained result (Fig 5,6,7and8) showed that 1:1 methyl paraben to Ortho – Chloranil reagent ratio was obtained.

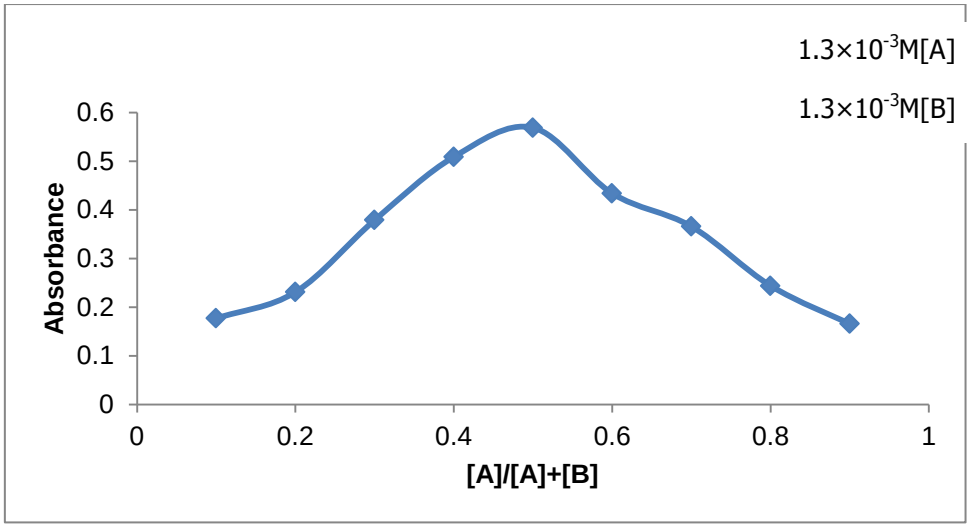


Fig.(5) Job s plot method for methyl paraben Ortho – Chloranil reagent.

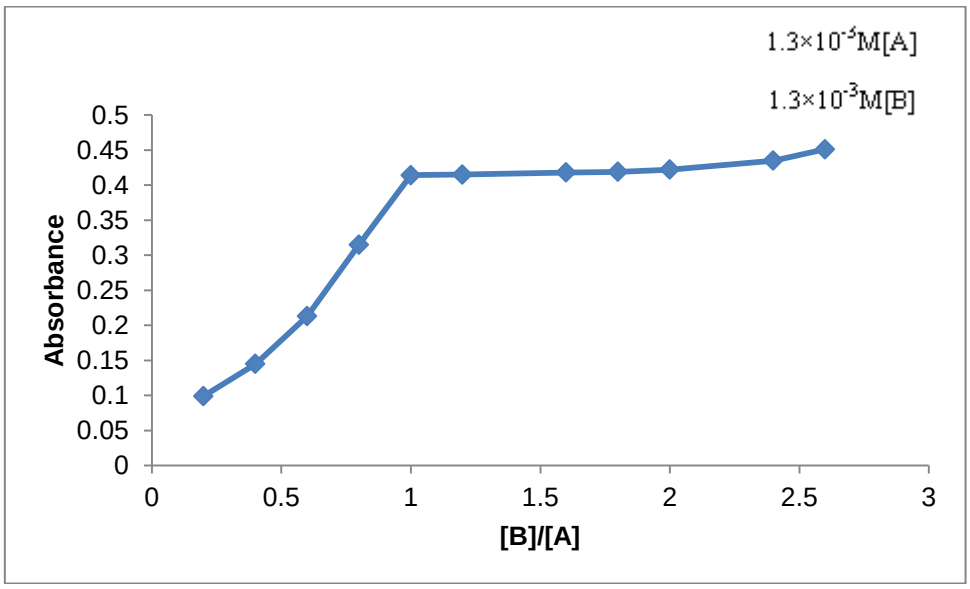


Fig.(6) Mole ratios plot method for methyl paraben Ortho – Chloranil reagent

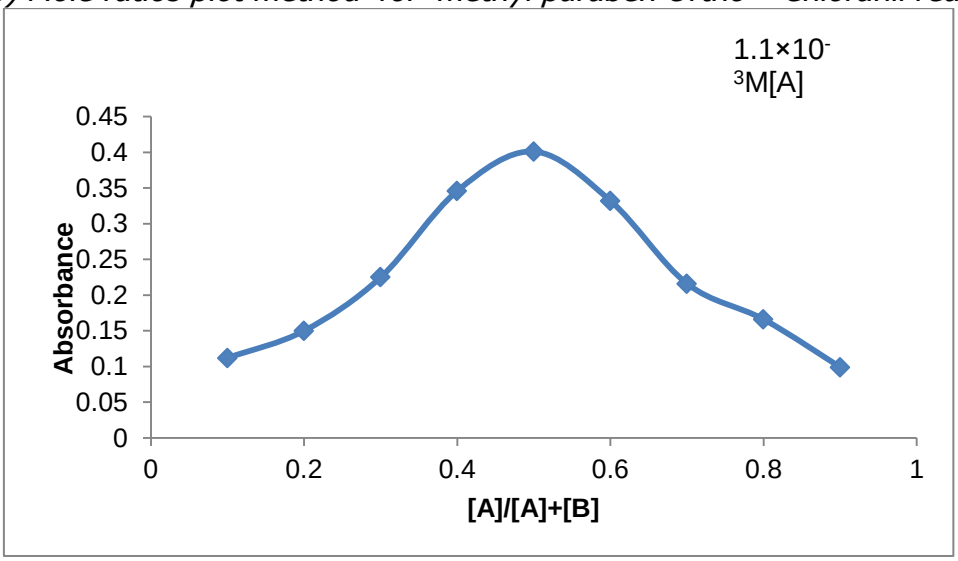


Fig.(6) Job s plot method for propyl paraben Ortho – Chloranil reagent

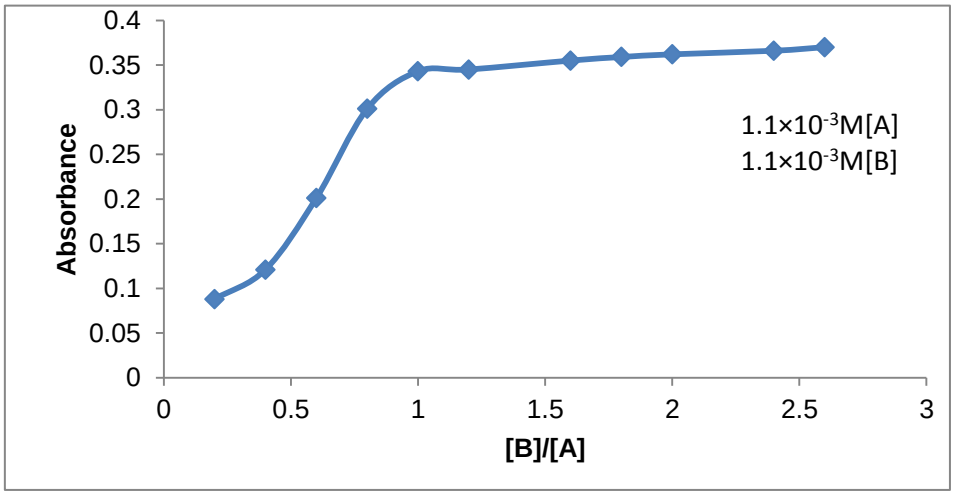
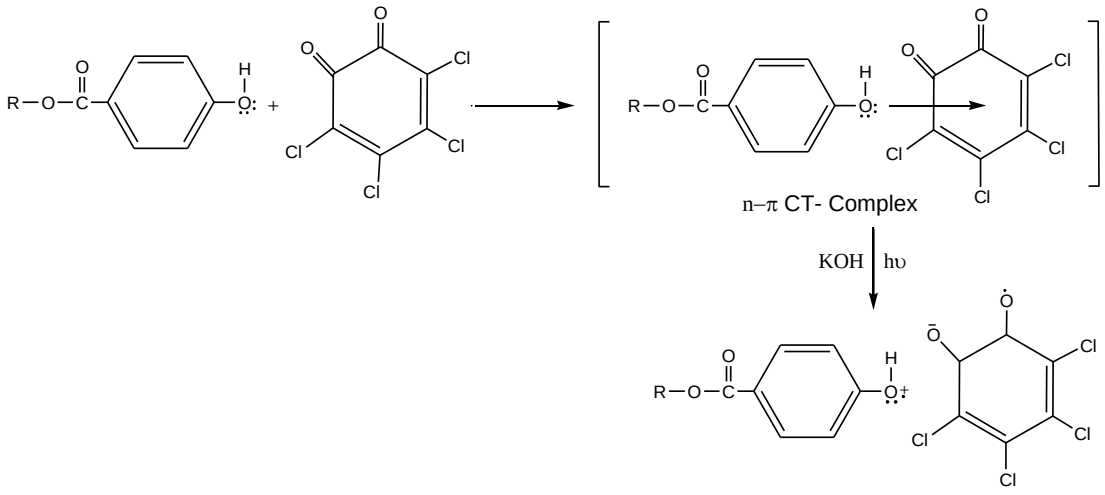


Fig.(8) Mole ratios plot method for propyl paraben Ortho – Chloranil reagent

The Suggested chemical reaction

The O - Cloranil is a reagent which is acceptor π electrons and strongly interaction with Nitrogen atom and other atoms containing a pair of non bounding electrons[10]. The resulting complex ratio was 1:1 hydroxyl: o – Cloranil, that Indicated, the involvement of the hydroxyl group in the formation of the complex, They appear a new peak as a result of charge transfer that differ from the absorbance peak of both the donor and acceptor and the new peak formation a n-π CT Complex, when Occurrence of this complex to radiation in the visible region or UV leads to the formation of a pair of free radicals ions $D^+ A^-$ [11]. The presence of KOH quickly formation of free radicals., And it can be expressed as a complex formation reaction transfer charge and free radicals as follows.



R=CH_3 group to methyl paraben.

R= CH_3-CH_2CH_3 group to propyl paraben.

Interference effect.

The Interference effect to absorbance of 20µg/ml methyl and 15µg/ml propyl paraben was stuided , Added increasing amounts of interference and measured dependent to conditions in the table (9) and the absorption was measured at 415 and 385nm respectively. the table (12)shows the results.

Table (12) Interference effect

Foreign Compound	Foreign added (µg/ml)	Recovery%	
		Methyl paraben	proyl paraben
Sucrose	50	99.79	98.37
	100	99.59	100.81
	200	99.38	101.21
Glucose	50	100.20	99.18
	100	100.41	100.41
	200	100.82	100.81
Lactose	50	97.33	99.59
	100	98.15	98.78
	200	96.72	97.15
NaCl	50	97.13	100.40
	100	98.97	97.96
	200	97.54	96.75
KCl	50	100.20	98.78
	100	100.82	98.37
	200	101.43	98.78

Above results show the absence of interference significantly when estimating paraben compounds using optimal conditions for the proposed method where the ratios between retrospective 101.43%, and 96.72%.

Application of the method

This method developed by applieding to the detergent which contain the methyl and propyl paraben (shampoo pert and lotion On Coconut shower gel) using Standard additions method, by pulling 1ml of shampoo (unknown concentration) to five container (5ml volumetric flask) adding ,increasing volumes, (0.5-1.5-1-2ml) of standard solution concentrate (25µg/ml) methyl and (30µg/ml) propyl the last volumetric flask remaind without any addition, and the solutions treatment with same method above the calibration curve drawing and then measured the absorbance at 415 and 385nm wavelength, Figure (9,10,11,and 12) shows the results of standard addition method.

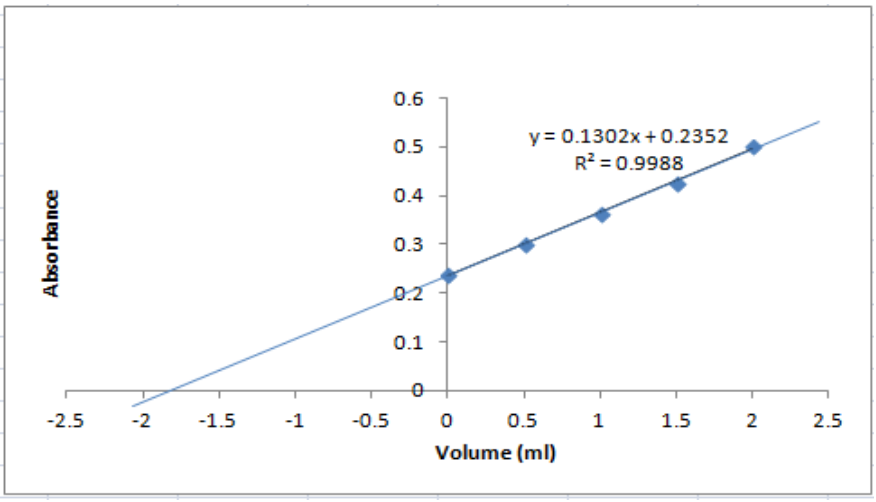


Fig. (9) Standard addition method, curve to estimate methyl paraben in detergents (Shampoo pert) (concentration of standard solution 25µg/ml and 1ml of the unknown solution, all the readings were the average of four measurements).

From the equation of a straight line when y= 0, van x=-1.806, the observed concentration is 9.03µg and using the relationship:

$C_\circ V_\circ = -C_s V_s$

$C_\circ$ =Shampoo concentration

$V_\circ$= 1ml Shampoo Volume

C_s= Concetration of the Standard solution of pure methyl added =25µg/ml

V_s =methyl Standard solution

In the same method as above, estimated methyl paraben in Shampoo(SNCI s.a.l. Lebanon) refreshing shower gel, and measured the solution at 415nm wavelength, and the results show in Figure (6).

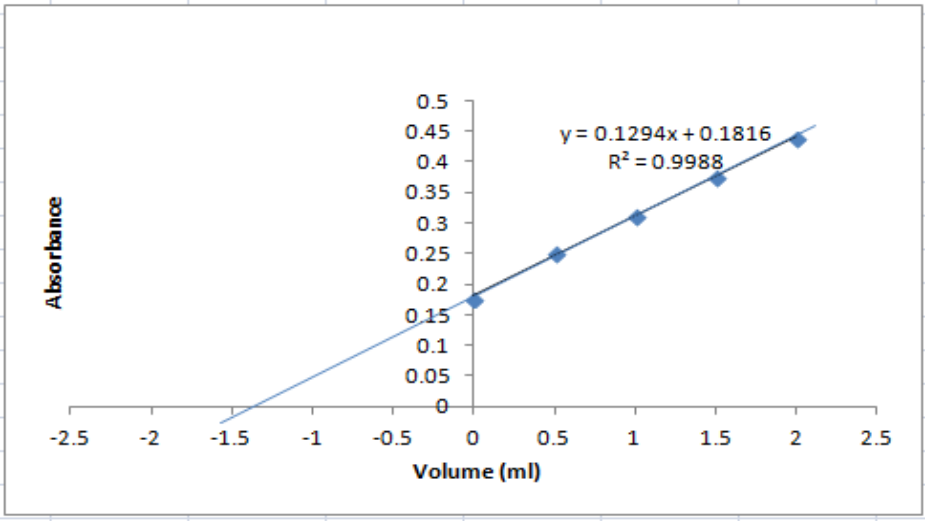


Fig.(10) Standard addition method, curve to estimate of methyl paraben in detergents (On Coconut) (concentration of standard solution 25µg/ml and 1ml of the unknown solution, all the readings were the average of four measurements).

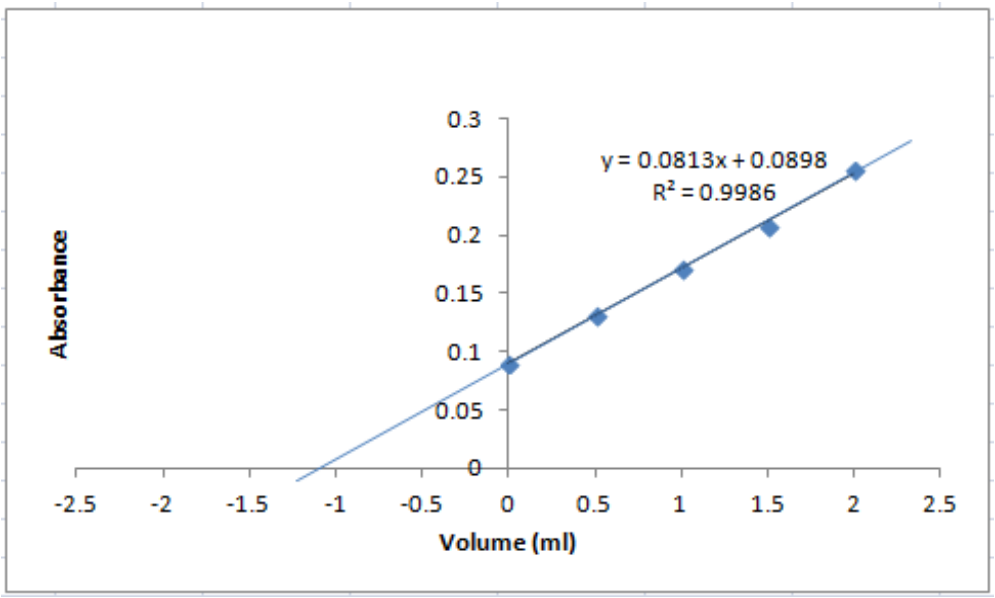


Fig. (11) Standard addition method, curve to estimate of propyl paraben in detergents (*Shampoo pert*) (concentration of standard solution 30µg/ml and 1ml of the unknown solution, all the readings are the average of four measurements).

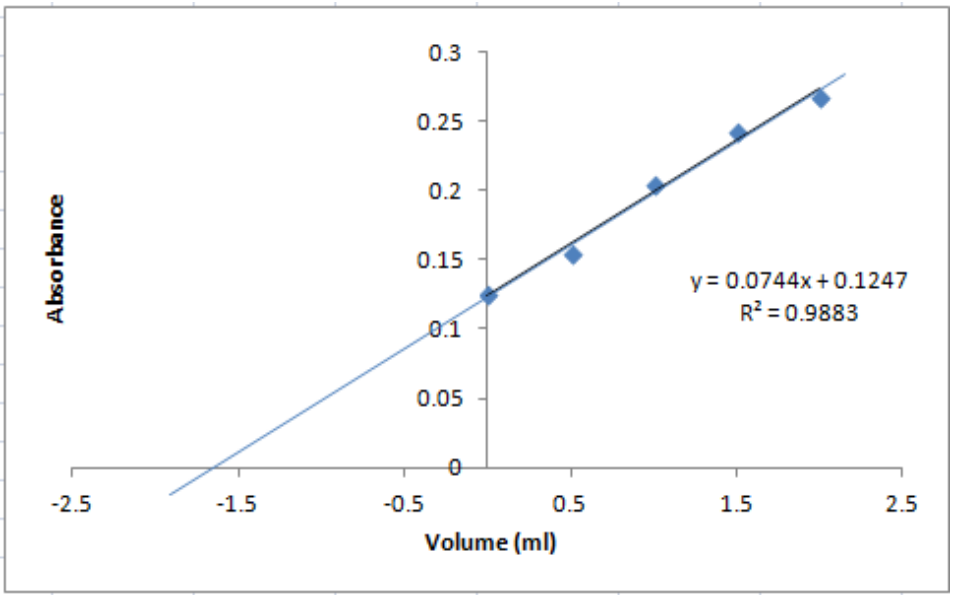


Fig. (12) Standard addition method, curve to estimate of propyl paraben in detergents (On Coconut) (concentration of standard solution 30µg/ml and 1ml of the unknown solution, all the readings are the average of four measurements).

The results above curve observed to estimate methyl paraben in detergents The concentrations within the limits of Beer's law to a standard curve for methyl paraben.

Table (13) Determination of methyl paraben in detergents by a standard addition method.

Detergent	methyl paraben amount µ	methyl paraben Found	propyl paraben amount µg	propyl paraben Found	Recovery%	
					methyl	propyl
shampoo pert	٩	9.03	6.5	6.627	100.33	101.95
lotion On Coconut shower gel	٧	7.015	١٠	10.056	100.21	100.56

The results above curve observed to estimate methyl paraben in detergents the concentrations within the limits of Beer's law to a standard curve for methyl paraben.

Conclusion

The proposed spectrophotometric method was sensitive (trace amounts can be determined), accurate and simple since it does not need neither temperature control nor solvent extraction step.The method was besed on the CT- Complex formation reaction of methyl and propyl paraben as donors with Tetrachloro –o-benzoquinone reagent as π acceptor measured at 415 and 385nm.

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طريقة طيفية للتقدير المايكروي لمركبات المثل والبروبيل بارابين في بعض مستحضرات التنظيف من خلال معقدات انتقال الشحنة

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الخلاصة

طورت طريقة طيفية ومبسطة لتقدير مركبات المثيل وبروبيل بارابين من خلال معقدات انتقال الشحنة (CT). اذ تضمنت الطريقة تفاعل مركبات الباربين المستخدمة مع حامض الكلورانيك كمستقبل π . كانت قيمة الامتصاصية في الظروف المثلى للمثل والبروبيل باربين هي ٤١٥ و ٣٨٥ نانو ميتر على التوالي وكانت حدود المدى الخطي للمثل والبروبيل باربين هي ٥-٤٠ و ٥-٥٥ مايكرو غرام .مللتر$^{-١}$ على التوالي. ومعامل الامتصاص المولاري للمثل والبروبيل باربين هي 3803.75 و 2342.6 لتر. مول$^{-١}$. سم$^{-١}$.والحد الادنى لحد الكشف للمركبين بين 0.073 و0.086 مايكرو غرام.مللتر$^{-١}$ على التوالي. وتبين الحسابات الاحصائية نسبة تكوين المعقدات لمركبات الباربين المستخدمة مع حامض الاورثو كلورانيل هي ١:١. وفي هذه الدراسة تم حساب الدقة والتوافقية للمعقدات المتكونة . وطبقت الطريقة بشكل ناجح في بعض مستحضرات التنظيف الحاوية على مركبات الباربين المستخدمة في هذه الدراسة.

الكلمات الدالة : مثيل وبروبيل بارابين، معقدات انتقال الشحنة ، مستحضرات التنظيف