



SPECTROPHOTOMETRIC DETERMINATION OF BENDIOCARB AFTER SYNTHESIS A NEW COLORED COMPOUND WITH A PARA-AMINO PHENOL IN ENVIRONMENTAL WATER SAMPLES

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ABSTRACT

A simple, sensitive, rapid and accurate spectrophotometric method was developed for the analysis of parts per million levels of widely used carbamate pesticide bendiocarb. The proposed method was based on alkaline hydrolysis of the bendiocarb pesticide, and the resultant hydrolysis product of bendiocarb was coupled with para-amino phenol to give blue color product with λ_{\max} (630 nm). This compound shows an absorption maxima at a wavelength of (630 nm) with apparent molar absorptivity ($2.018 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$) and obeyed Beer's law in the concentration range of (0.1-10 $\mu\text{g/mL}$). Sandell's of the color reaction are $0.01106 \mu\text{g cm}^{-2}$. The effect of the non-target species on the determination of bendiocarb was studied. The formation of colored derivatives with the coupling agents is and stable for 3-6 hr.

Key words: Spectrophotometry, Bendiocarb, Para-amino phenol, Environmental samples.

INTRODUCTION

Pesticides are extensively worldwide used for agriculture and for non-agricultural purposes. The major environmental concern of used pesticides is their ability to leach down to subsoil and contaminate the ground water, or, if they immobile, they could persist on the top soil and become harmful to microorganisms, plants, animal and people^{1,2}. Harmful pesticide residues can contaminate the environment and accumulate in ecosystems than entering the human food chain³⁻⁵. Pesticides have various characteristics that determine how act once in soil where it could accumulate to toxic level. Generally, soil and groundwater pollution are the major consequences environmental effects of pesticides application. Pesticides can reach water through surface runoff from treated plants and soil, there for we can needed a simple way to determination the trace reside of this pesticide. Bendiocarb was

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one of these harmful pesticides therefore we need to detect its amount in the environmental water samples bendiocarb, (2,2-dimethyl-1,3-benzodioxol-4-yl-methylcarbamate) and other chemical name of bendiocarb, (2,2-dimethyl-2 H-1,3-benzodioxol-4-yl methyl carbamate), (2,2-dimethyl-1,3-benzodioxol-4-yl *N*-methylcarbamate). (2,3-Isopropylidenedioxy phenyl methylcarbamate) empirical formula ($C_{11}H_{13}NO_4$) relative molecular mass (223.25 g/mole)⁶.

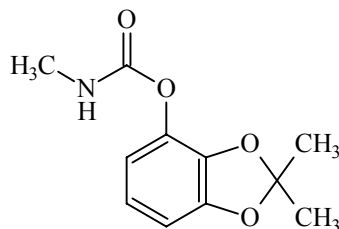


Fig. 1: The structure of bendiocarb

p-Amino phenol is also called p-hydroxyaniline, 4-amino-1-hydroxy benzene and p-amino phenol (Czech). The trade names are aZoli; Rodinal; Ursol P, it has molecular formula (C_6H_7NO), molecular weight (109.13)⁷.

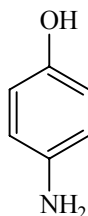
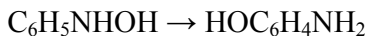
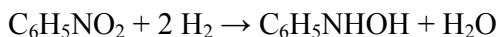


Fig. 2: The structure of para-amino phenol

It is produced from phenol by nitration followed by reduction with iron. Alternatively, the partial hydrogenation of nitrobenzene affords phenyl hydroxylamine, which rearranges primarily to 4-aminophenol⁸.



Alternatively, it may be produced by the reduction of nitrobenzene with iron filings in acid, or by catalytic hydrogenation. This reagent was stable conditions to avoid, may discolor on exposure to light may discolor on exposure to air. In compatibilities are strong oxidizing agents; Hazardous combustion or decomposition products; carbon monoxide, carbon dioxide, nitrogen oxides⁹. In industry, p-aminophenol is widely used in the production

of colored textiles, treatment of leathers, photography development process, pharmaceutical formulations, and as antioxidants additive in oil industry. In addition, p-aminophenol as phenol derivative, which contains in its structure as basic group and hydroxyl group, it was suggested as a catalyst for different reactions¹⁰. Several techniques have been reported for the determination of bendiocarb, such as spectrophotometry¹¹⁻¹⁶, voltammetry¹⁷, liquid chromatographic¹⁸, gas chromatographic^{19,21} and HPLC²²⁻²⁴. A novel method²⁵ and different chromatographic techniques were used. The aim of this search was to determination of bendiocarb in different neutral water by action it with reagent (PAP) and syntheses a color compound in alkaline medium, which easily detect the percent of pollutions environmental water samples with pesticides (bendiocarb).

EXPERIMENTAL

Instrumentation

A Shimadzu 1800 pc Model Double-beam UV-Vis Spectrophotometer (2000) (Japan) working at wavelength of 190-1100 nm (± 0.3 accuracy and ± 0.1 repeatability) spectral band width of < 2 nm and equipped with 10 mm optical. Path cell was used for the scanning of absorption spectra of all reagents and complexes throughout this study. While absorbance measurements in the optimization study were conducted with spectrophotometer SEDICO (1665 U.K.) double-beam working at wavelength of 190-1100 nm.

Chemicals and reagent

Bendiocarb (purity 99.9%) was obtained from Accustandard (USA). A standard stock solution of $20 \mu\text{g mL}^{-1}$ of bendiocarb was prepared by dissolving 0.02 g of the solid pesticide in (1000 mL) deionized water. This solution was kept in Refrigerator to prevent any hydrolysis or exposing to sun light which being stable for more than two months. A standard stock solution of sodium carbonate Na_2CO_3 (1 M) was prepared by dissolving (10.6 g) of the solid product in (100 mL) of deionizer water. A standard stock solution of para-amino phenol PAP (4.58×10^{-4} M) was prepared daily by dissolving (0.005 g) of para-amino phenol PAP in (100 mL) and filled to mark with deionized water potassium periodate KIO_4 (0.2 M) was prepared by dissolving (4.6 g) of the solid product in (100 mL) of deionizer water.

Collection of environmental spiked water samples

A (500 mL) of real water samples were directly taken from tap, irrigation water was irrigation collected from Tigris River under Jadria bridge. Underground water samples were collected from well in Baghdad (Jadria). All the environmental water samples were kept in glass containers.

4-Recommended procedure in neutral water samples

A series of solution were prepared by taking a known volume of bendiocarb into a 25 mL volumetric flask and added 1.5 mL of (1 M) sodium carbonate Na_2CO_3 and (5 mL) of (4.58×10^{-4} M) para-amino phenol and added 3 mL of (0.2 M) potassium periodate KIO_4 and filled to the mark with environmental water sample to obtained (2, 4, 6, 8, 10, 12 ppm) concentrations of bendiocarb in alkaline medium.

RESULTS AND DISCUSSION

Spectrum of bendiocarb after coupling with para-amino phenol

The UV-Vis spectrum of the complex obtained by the reaction of bendiocarb with para-amino phenol have λ_{max} of the color compound 630 nm (Fig. 3c). This caused by charge transition between $\text{C} = \text{N}$ and conjugated benzene ring, it is also the reason of the blue color of the solution. The maximum absorption of the color compound at 630 nm is used in all subsequent experiments.

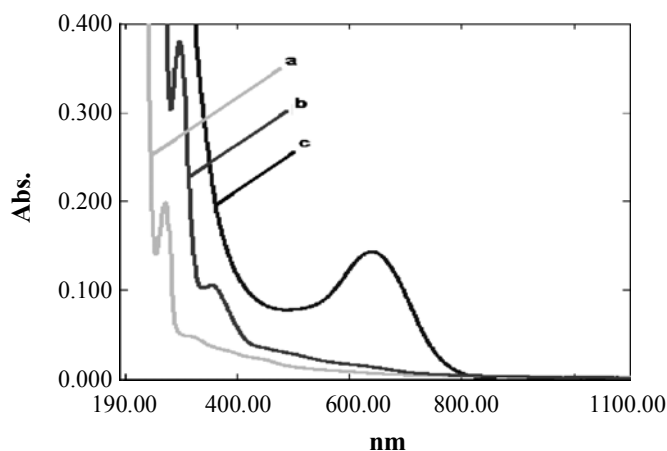


Fig. 3: Absorption spectrum (a): Bendiocarb versus deionized water (b) Para-amino phenol versus deionized water (c) The complex obtained by the reaction bendiocarb with para-amino phenol

Optimization of the color product with alkaline concentration

A series of solution were prepared by take (12.5 mL) of (20 ppm) bendiocarb and added different volumes (0.5, 1, 1.5, 2, 2.5, 3.75 mL) of (1 M) of sodium carbonate Na_2CO_3 in to 25 mL volumetric flasks, 5 mL of 50 ppm para-amino phenol and 3 mL of (0.2 M)

potassium periodate KIO_4 and filled to the mark with deionized water. The absorbance measurements were carried out at a wavelength at (630 nm).

Table 1: The effect of the sodium carbonate Na_2CO_3 absorbance measurements

Volume of Na_2CO_3 mL	Absorbance			Mean Absorbance
	R1	R2	R3	
0.5	0.120	0.122	0.124	0.122
1.0	0.144	0.142	0.140	0.142
1.5	0.182	0.184	0.180	0.182
2.0	0.168	0.166	0.164	0.166
2.5	0.146	0.142	0.140	0.142
3.75	0.110	0.112	0.114	0.112

As it shows the best volume of the base sodium carbonate Na_2CO_3 were (1.5 L) from the Table 1 and Fig. 4.

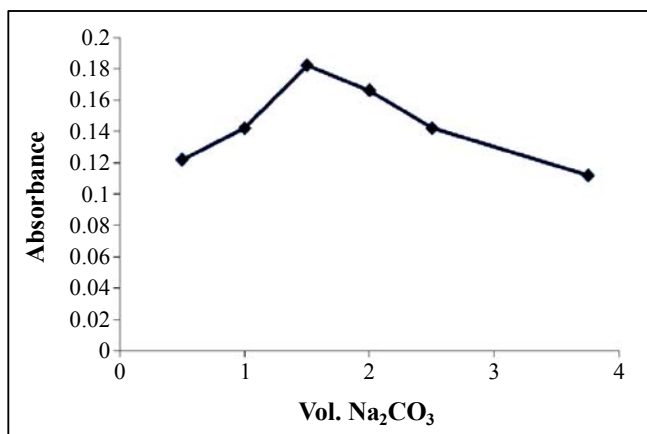


Fig. 4: Optimization of the concentration of Na_2CO_3

Optimization of the reagent para-aminophenol concentration (PAP)

A series of solution were prepared by take (12.5 mL) of 20 ppm bendiocarb (1.5 mL) of (1 M) sodium carbonate Na_2CO_3 , 3 mL of 0.2 M the potassium periodate and different volumes (1, 2, 3, 4, 5, 6, 7 mL) of (50 ppm) para-amino phenol in to 25 mL volumetric flasks and filled to the mark with deionized water. The absorbance measurements were carried out at a wavelength at 630 nm.

The best volume of para-amino phenol PAP were obtained (5 mL), as shown from the Table 2 and Fig. 5.

Table 2: The effect of the para-aminophenol PAP of color compound absorbance measurements

Vol. of (PAP) mL	Absorbance			Mean Absorbance
	R1	R2	R3	
1.0	0.113	0.112	0.111	0.112
2.0	0.134	0.135	0.133	0.134
3.0	0.145	0.146	0.147	0.146
4.0	0.165	0.164	0.163	0.164
5.0	0.181	0.183	0.182	0.182
6.0	0.163	0.162	0.164	0.163
7.0	0.155	0.154	0.153	0.154

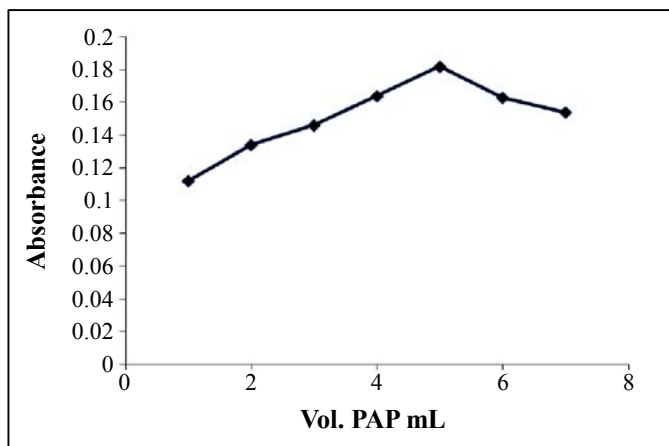


Fig. 5: Optimization of the concentration of PAP

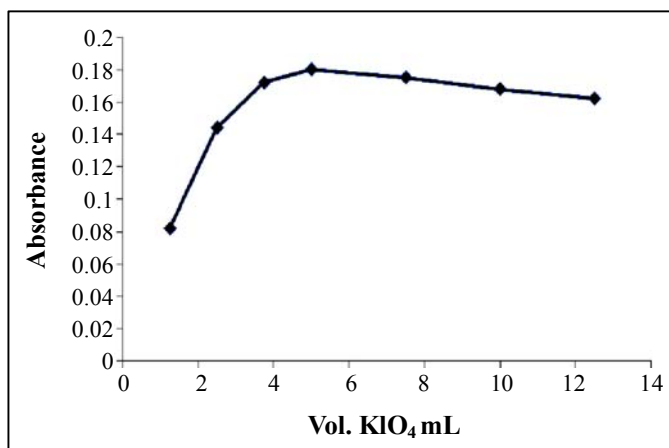
Optimization of the potassium periodate (KIO_4) concentration

A series of solution were prepared by taking 12.5 mL of 20 ppm bendiocarb 1.5 mL of 1 M sodium carbonate Na_2CO_3 , 5 mL of 50 ppm para-amino phenol and taking a different volumes 1.25, 2.5, 5, 7.5, 10, 12.5, 25 mL of 0.2 M potassium periodate KIO_4 . The absorbance measurements were carried out at a wavelength at 630 nm.

Table 3: The effect of potassium periodate KIO_4 of the absorbance measurements were obtained of color compound

Vol. of KIO_4 mL	Absorbance			Mean Absorbance
	R1	R2	R3	
1.25	0.082	0.083	0.081	0.082
2.5	0.145	0.144	0.143	0.144
3.75	0.172	0.173	0.171	0.172
5.0	0.180	0.181	0.179	0.180
7.5	0.176	0.175	0.174	0.175
1.0	0.169	0.168	0.167	0.168
12.5	0.161	0.162	0.163	0.162

The best volume of potassium periodate KIO_4 were obtained 5 mL, which is shown from the Table 3 and Fig. 6.

**Fig. 6: Optimization of the concentration of potassium periodate (KIO_4)**

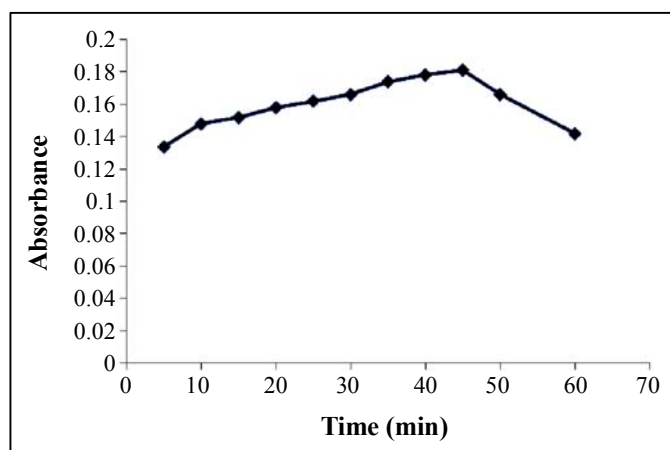
Optimization of hydrolysis time of the color compound

A solution was prepared by using 12.5 mL of 20 ppm bendiocarb 1.5 mL of 1 M sodium carbonate Na_2CO_3 , 5 mL of 50 ppm para-amino phenol and 5 mL of 0.2 M potassium periodate (KIO_4) into 25 mL volumetric flask and filled to the mark with deionized water and the absorption measurements were carried at 630 nm in triplicate manner and calculated the best time that take best absorbance.

Table 4: The effect of time for hydrolysis of the color compound

Time (min)	Absorbance			Mean absorbance
	R1	R2	R3	
5	0.134	0.132	0.136	0.134
10	0.146	0.148	0.150	0.148
15	0.150	0.152	0.154	0.152
20	0.160	0.158	0.156	0.158
25	0.164	0.162	0.160	0.162
30	0.166	0.165	0.167	0.166
35	0.174	0.172	0.174	0.174
40	0.177	0.178	0.179	0.178
45	0.182	0.181	0.180	0.181
50	0.168	0.166	0.164	0.166
60	0.142	0.144	0.140	0.142

From Table 4 and Fig. 7, we can see the best time of the hydrolysis of the color compound was 45 min to obtain the best absorbance of the color compound.

**Fig. 7: The effect of time for hydrolysis of the color compound**

Calibration curve of color compound in deionized water

A series of solution were prepared by using known volumes of bendiocarb 0, 2.5, 5, 7.5, 10, 12.5, 15 mL in the concentration 4.48×10^{-4} M and 1.5 mL of 1 M sodium carbonate, 5 mL of 50 ppm para-amino phenol and 5 mL of 0.2 M potassium periodate in to a 25 mL volumetric flask and filled to the mark with deionized water and the measurements were carried at 630 nm in triplicate manner The absorbance measurements were obtained illustrated in Table 5 and Fig. 8.

Table 5: The absorbance measurements of standard solution of color compound

Con. of bendiocarb (ppm)	Absorbance			Mean Absorbance
	R1	R2	R3	
2	0.190	0.192	0.193	0.192
4	0.365	0.364	0.363	0.364
6	0.546	0.545	0.547	0.546
8	0.724	0.722	0.726	0.724
10	0.912	0.910	0.914	0.912
12	1.087	1.088	1.089	1.088

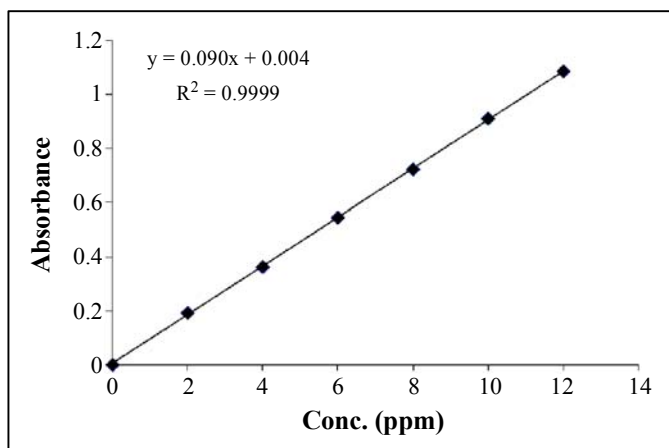


Fig. 8: Calibration curve of bendiocarb in deionized water

Optical characteristic feature of the calibration curve are given in Table 6. The main features of part of the calibration curve carried out by using the optimum condition.

Table 6: Optical characteristic features of calibration curve

Parameter	Values
Color	Blue
Wave length λ_{\max} (nm)	630
Beer's Law limit ($\mu\text{g mL}^{-1}$)	0.1-10
Molar absorptivity ($\text{Lmol}^{-1}\text{cm}^{-1}$)	2.018×10^4
Limit of detection ($\mu\text{g mL}^{-1}$)	0.265
Sandell's sensitivity ($\mu\text{g.cm-2/0.001 A.U}$)	0.01106
Slope (m)	0.0904
Intercept (C)	0.0040
Regression equation ($Y = m X + C$)	$0.090x + 0.004$
Correlation coefficient (r^2)	0.9999

From the above data in Table 6, we can see that the method is suitable for the determination of bendiocarb in environmental water samples after action with PAP.

Effect of interferences

To study the effect of the some interference probable in the water to calculate the selectivity, the proposed method has been reported. A series of solution were prepared by using 12.5 mL of 20 ppm bendiocarb and 1.5 mL of 1 M sodium carbonate Na_2CO_3 , 5 mL of 50 ppm para-amino phenol and 5 mL of 0.2 M potassium periodate (KIO_4), in to 25 mL volumetric flask and added 1 mL of 10 ppm the each one of the interferences, which prepared by take suitable weight of the solid substance and dissolved in to 100 mL deionized water and filled to the mark with deionized water and measured the absorbance of each solution. Another solution were prepared by used 12.5 mL of 20 ppm bendiocarb 1.5 mL of 1 M sodium carbonate Na_2CO_3 , 5 mL of 50 ppm para-amino phenol and 5 mL of 0.2 M potassium periodate (KIO_4), in to a 25 mL volumetric flask as a blank but with out any addition of the interferences and calculated the mean absorbance and relative stander deviation (RSD) of all solution, as Table 8 shows height absorbance of the solution with out any addition of the interference. The Table 7 shows the effect of interferences too.

From the above table, as we show there are no presence of the interference of the optimizations condition as illustrated above. Therefore, we can apply the proposed method to determine the bendiocarb in the environmental water sample sensitivity.

Table 7: Effect of interferences

Type of interferences 10 ppm	Absorbance			Mean	RSD%
	R1	R2	R3		
Non-addition	0.183	0.181	0.182	0.182	0.549
p-nitro phenol	0.096	0.097	0.095	0.096	1.473
Pyrocatechol	0.090	0.088	0.086	0.087	1.625
Hydroquinone.	0.094	0.096	0.092	0.093	1.521
Resorcinol	0.096	0.095	0.094	0.095	1.488
o-cresol	0.055	0.056	0.057	0.056	1.251
Urea	0.085	0.086	0.087	0.086	1.644
Fructose	0.078	0.076	0.074	0.075	1.885
Glucose	0.082	0.081	0.083	0.082	1.724
Lactose	0.061	0.062	0.063	0.062	1.131
Sodium acetate	0.075	0.074	0.076	0.075	1.885

Stoichiometric determination of color compound**Continuous variation method (Job`s method)²⁶**

A series of solution 1, 2, 3, 4, 5, 6, 7, 8, 9 mL of 4.48×10^{-4} molL⁻¹ Bendiocarb was pipette into each of 10 mL volumetric flask then 9, 8, 7, 6, 5, 4, 3, 2, 1 mL of 4.48×10^{-4} reagent, the absorbance of the solution was measured by UV-Vis spectrophotometer at λ_{\max} (630 nm). The stoichiometric ratio between bendiocarb with reagent 1:1 results are shown in the Table 8 and Fig. 9.

Table 8: The role between the absorbance by continuous variation method with bendiocarb and the legend

VB (mL)	VR (mL)	VB/VT	Absorbance at $\lambda = 630$ for color compound
1	9	0.1	0.048
2	8	0.2	0.088
3	7	0.3	0.124

Cont...

VB (mL)	VR (mL)	VB/VT	Absorbance at $\lambda = 630$ for color compound
4	6	0.4	0.158
5	5	0.5	0.182
6	4	0.6	0.157
7	3	0.7	0.122
8	2	0.8	0.089
9	1	0.9	0.050

Plotting the value of absorbance versus the VB/VT is shown in Fig. 9.

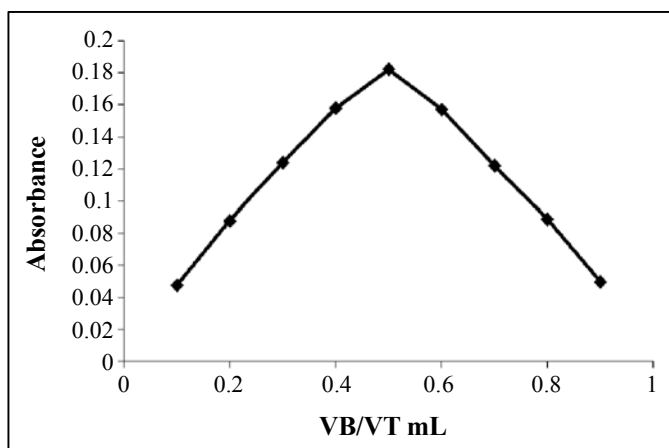


Fig. 9: Continuous variation method plot

VB: Volume of the compounds (bendiocarb).

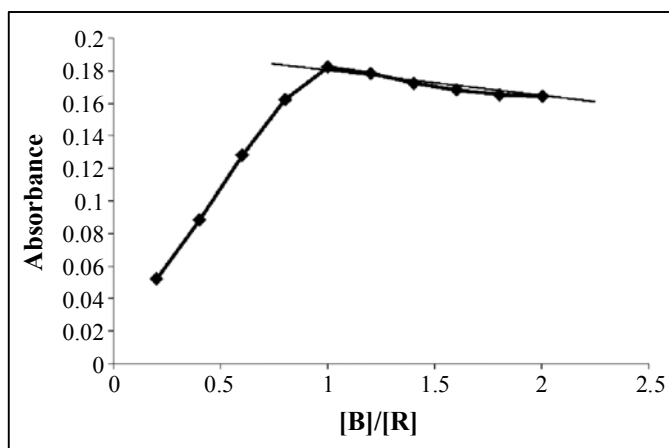
VT: Total volume of the bendiocarb and the reagent.

Mole-ratio method²⁷

Aliquots of 10 mL solution of ($4.48 \times 10^{-4} \text{ molL}^{-1}$) of bendiocarb and increasing concentrations of para-amino phenol $0.358 \times 10^{-7} \text{ molL}^{-1}$ reagent at optimum pH were taken. The absorbance of the solutions were measured by UV-Vis spectrophotometer versus blank at $\lambda_{\text{max}} = 630 \text{ nm}$. The stoichiometric ratio between 1:1 results are shown in the Table 9 and Fig. 10.

Table 9: The mole-ratio method results for the bendiocarb complexes

[R]	[B]/[R]	Absorbance at $\lambda = 630$ for reagent
3.58×10^{-7}	0.2	0.052
7.16×10^{-7}	0.4	0.088
1.07×10^{-6}	0.6	0.128
1.43×10^{-6}	0.8	0.162
1.79×10^{-6}	1	0.182
2.14×10^{-6}	1.2	0.178
2.51×10^{-6}	1.4	0.172
2.87×10^{-6}	1.6	0.168
3.23×10^{-6}	1.8	0.165
3.58×10^{-6}	2	0.164

**Fig. 10: Mole ratio plot of color compound**

[B] The concentration of bendiocarb.

[R] The concentration of reagent PAP.

Stability constant of reaction product²⁷

The conditional or apparent stability constant of the 1:1 (reagent and bendiocarb) product was evaluated as described.

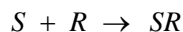
The stability constant [K] of colored product formed by imputation of (Reagent : bendiocarb) is as follows :

A series of solution was prepared containing three different concentrations of reagent and bendiocarb (1:1) and the concentration was $4.48 \times 10^{-4} \text{ molL}^{-1}$ for each reagent and bendiocarb. When formed imputation under this condition is easily hydrolyzed and the intensity absorption was very low.

A series of solution was prepared containing three different concentrations of reagent and bendiocarb but with abundance of the reagent (the best concentration). The complex was prepared and no decomposition was found. Using the intensity absorption A_m , and the relation given below, the value of degree of decomposition (α) can be calculated as follows:

$$\alpha = \frac{A_m - A_s}{A_m}$$

Stability constant [K] calculated as follows:



$$\alpha c \quad \alpha c \quad (1 - \alpha)c$$

$$K = \frac{[SR]}{[S][R]}$$

$$K = \frac{(1 - \alpha)c}{(\alpha c)(\alpha c)} = \frac{1 - \alpha}{\alpha^2 c}$$

K; Stability constant

C; The concentration of the product complex and it is equivalent the concentration of bendiocarb. The result are shown in the below Table.

Table 10: The absorption of the result complex

Volume of bendiocarb (mL)	Absorption at λ_{max} 630 nm			
	A_s	A_m	α	K ($\text{mol}^{-2} \cdot \text{L}$)
1	0.102	0.124	0.1774	5.088×10^4
3	0.128	0.162	0.1486	8.606×10^4
5	0.146	0.180	0.1888	5.07×10^4

CONCLUSION

A simple, sensitive, rapid and accurate spectrophotometric method was developed for the analysis of parts per million levels of widely used carbamate pesticide bendiocarb. The proposed method was based on alkaline hydrolysis of the bendiocarb pesticide, and the resultant hydrolysis product of bendiocarb was coupled with para-amino phenol to give blue color product with λ_{\max} (630 nm). This compound shows an absorption maxima at a wavelength of (630 nm) with apparent molar absorptivity ($2.018 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$) and obeyed Beer's law in the concentration range of (0.1-10 $\mu\text{g/mL}$). Sandell's of the color reaction are $0.01106 \mu\text{g cm}^{-2}$. The effect of the non-target species on the determination of the bendiocarb was studied the formation of colored derivatives with the coupling reagent is instantaneous and stable for 3-6 hr.

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