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Spectrophotometric Determination Of Bismuth (iii) Via Pyrocatecol Violet Dye As Chromogenic Reagent

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ABSTRACT

Direct, rapid, simple, precise and an accurate spectrophotometric method for the estimation of bismuth (III) ion was proposed. The reaction of bismuth (III) ion with pyrocatecol violet in aqueous solution in the presence of cetyl trimethyl ammonium bromide(CTAB)to form at pH 3.4 a colored complex which was showing maximum absorption at 648 nm. The linearity of the proposed method was obeyed Beer's law over the concentration range of 5-200 µg /25 ml i.e (0.2-8.0) ppm. The molar absorptivity factor found to be 1.72×10^4 l/mol.cm and, Sandall's sensitivity index was 0.01215 µg/cm. The proposed method was successfully applied for the determination of bismuth (III) in water and pharmaceutical preperation (Tablet).

INTRODUCTION:

Chemical element bismuth has a symbol Bi with atomic number 83. Element bismuth naturally may occur, an impartant commercial ores of bismuth sulfide and oxide have been formed. The free element as deuse as lead is 86%. Bismuth is a brittle and silvery white color metal when it produced freshly, the surface oxidation of bismuth give it a pink tinge. Bismuth is diamagnetic element and radioactive slightly, also bismuth has the lowest thermal conductivity values among metals. A lthough bismuth was confused with tin and lead, also bismuth sharing some physical properties with that elements. The etymology was uncertain, while possibly it comes from arabic bismid, that meaning bismuth having the properties of anti mony element[(1)] also the wbismuth or weibe masse which was German words meaning white mass was translated in the mid 16th century to new latin bisemutam[(2)].

Bismuth is the last chemical element in group (Va) of periodic table with an important properties and usages[(3)]. In the environmental bismuth is one of the most common element due to its consumption for avariety of purpose. It has two main oxidation state as Bi(III) and Bi(V)[(4)]. Out of these, pentavalent state of bismuth was thermodynamically unstable while trivalent state of bismuth was stable[(5)]. In the earth crust, the existence of element bismuth in the sea is about 8.0 µg.Kg^{-1} and 0.2 µg.L^{-1}. Bismuth had many application in variety of field which include pharmaceuticals such as antiacids, antiseptic, antibacterial, antiulcer, anti-HIV and radio therapeutic agents and ulcer[(6)].

Several spectrophotometric methods was used for the determining of Bi, such as determining it by cloud point extraction[(7)].. A simple strategy has been developed for determination and speciation of bismuth (III) and (V) coupled dual wave β-correction spectrophotometry[(8)]. An electromembrance-microextraction (EME) followed by microcell UV-

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VIS spectrophotometric –detection was developed for fast and sensitive determination of bismuth in bismuth subcitrate and human plasma sample[9]A dispersive liquid-liquid microextraction method for the determination of bismuth in various samples by flame atomic absorption spectrometry[10]. A highly selective and sensitive spectrophotometric method has been developed for the determination of trace amounts of bismuth in various samples[11].

Determination of bismuth by electrothermal atomic absorption spectrometry using cationic microstructures of cetyltrimethyl ammonium bromide (CTAB) and sodium dodecyl sulfate (SDS) surfactants in human blood serum and hair sample [12], bismuth determining in pharmaceutical samples and synthetic mixture by extractive method[4]. A simple and reliable supramolecule-aggregated liquid solid microextraction method is described for preconcentration and determination of trace amounts of bismuth in water as well as human blood serum and hair samples[13]. Also bismuthcan be determined in human plasma by ICP-MS and its use in bioquivalence studies has been reported [14]. Gallacetophenone phenyl hydrazone was used for amperometric determination of Bi in wood's alloy as an analytical reagent [15].

Sevral reagents have been used for the spectrophotometric determination of bismuth such as dithiozone, diethyldithiocarbamate, xylenol orange, iodide, thiourea and azo reagents,[16]. Kinetic determination of bismuth by using the catalytic effect of bismuth on the oxidation of phenyl fluorone by H_2O_2 in ammonia buffer, the method was confirmed by determining Bi(III) in a drug of stomach ulcer[17]. Extractive determination of Bi(III) carried out with 1-amino-4,4-6-trimethyl(1H,4H) pyrimidine-2-thiol as an analytical reagent in alloy samples of Bi(III) also has been reported[18].

The present work is devoted to the spectrophotometric study of the colored complex of bismuth with pyrocatecol violet in the presence of cetyl trimethyl ammonium bromide (CTAB) as an attempt to increase the sensitivity and selectivity of bismuth determination.

EXPERIMENTAL:

Spectral and absorbance measurement were carried out using 1-cm matched cells and Jasco V-630 Spectrophotometer computerized double beam spectrophotometer. Also HANNA pH 24 was used for pH measurements.

Reagents

All chemical materials used were highest purity available.

Stock bismuth (III) solution (1000µg/ml): 0.231 g of $Bi(NO_3)_3.5H_2O$ (Fluka) was dissolved in 5 M nitric acid and diluting with distilled water in a 100 ml of a dry and clean volumetric flask [4].

Working bismuth (III) solution (100µg/ml): This solution was prepared by diluting 10 ml of bismuth stock solution with distilled water using a 100 ml clean and dry volumetric flask.

Pyrocatechol violet (PCV) reagent solution (1×10^{-3} M): 0.038 g of PCV (BDH) was dissolved in distilled water and the final volume was bring to be 100 ml using a suitable volumetric flask, then, it was transferred to be storage in a dark brown bottle where it remains stable more than 6 days.

Cetyltrimethyl ammonium bromide (CTAB) solution (1×10^{-3} M): 0.036 g of CTAB (Fluka) was dissolved in a suitable amount of distilled water, then the final volume was diluted to 100 ml with distilled water in a dry and clean volumetric flask.

Cetyltrimethyl pyridinium chloride (CPC) solution (1×10^{-3} M): 0.0358 g of CPC (Fluka) was dissolved and diluted to 100 ml with distilled water using a dry and clean volumetric flask.

Sodium dodecyl sulphate (SDS) solution (1×10^{-3} M): 0.028 g of SDS (Fluka) was dissolved in distilled water, then the final volume was diluted in a 100 ml dry and clean volumetric flask with distilled water.

Triton X-100 (1%): 1 g of Triton X-100 (Fluka) was dissolved in a 100 ml volumetric flask using distilled water.

Procedure for dosage form (Tablets)

Ten tablets (262 mg Bi(III)/tablet) of the drug were weighed, crushed with well mixed and weighed a portion equivalent to 0.0100 g of bismuth was dissolved in 50 ml of distilled water, shaken well, filtered and diluted with distilled water in a 100 ml volumetric flask. An aliquot of the drug solution was then treated as the recommended procedure.

RESULTS AND DISCUSSION

Preliminary studies of the reaction of bismuth (III) with PCV reagent and CTAB indicate that the reaction proceed immediately. The maximum absorption of blue colored complex was measured at 648 nm, while 430 nm was the selective wavelength of the reagent blank.

Optimum conditions

Various parameters were effected on the intensity of the colored complex absorption therefore, the effect of that parameters were studied and the reaction conditions were optimized.

Effect of pH

The effect of pH on color intensity of the complex was studied with solution containing 100µg of Bismuth (III) in two serious of flasks and various volume of 0.01 M of HNO_3 and NaOH solution,1ml of (0.001 M) of PCV reagent and 8.0 ml of (1×10^{-3}M) CTAB. Finally, all flasks were diluted to 25 ml with distilled water.

The absorbance of each sample flask against its corresponding blank solution was measured, as well as the final pH of each flask was measured. The results indicate that the absorbance was pH depend and maximum absorbance occured at pH 3.4 with maximum wavelength 648 nm.

Effect of surfactance:

The effect of the presence of some surfactant such as cationic cetyltrimethyl ammonium bromide (CTAB), cetylpyridinium chloride (CPC), non-ionic Iso-octylphenoxypolythoxy ethanol, (Triton X-100) and anionic sodium dodecyl sulphate (SDS) on the color intensity of the complex was examined(Table 1).

Table 1: EfEfect of surfactance

Surfactant solution*	Absorbance of 100 µg of bismuth (III)					λmax
	1	3	5	8	10	
CPC 1×10^{-3}	**0.112**	**0.153**	**0.162**	**0.250**	**0.251**	590
CTAB 1×10^{-3}	**0.135**	**0.252**	**0.301**	**0.332**	**0.331**	648
SDS 1×10^{-3}	**0.102**	**0.099**	**0.086**	**0.052**	**0.022**	550
Triton X-100 1%	**0.113**	**0.123**	**0.093**	**0.052**	**0.032**	520
Without surfactant	0.132					550

* ml of surfactant added before reagent.

The results in (Table 1) indicate that CTAB solution causes bathochromic shifted and increasing in the intensity of absorbance, therefore the addition of 8 ml of CTAB gives maximum absorbance and the lowest blank value, so it has been used in the subsequent measurements, show Fig.(1).

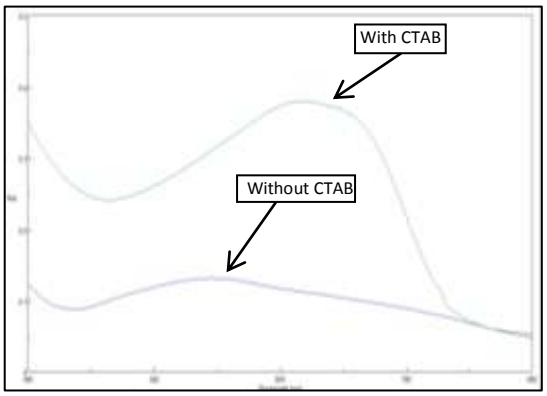


Figure 1.: Effect of CTAB of the absorption spectra for 100 µg Bi(III) /25 ml measured against reagent blank

Effect of order of addition:

This study was examined on the intensity of the color complex, which indicated results were shown in (Table 2) cleared that the (III) order of addition gives maximum absorbance, therefore, this order was adopted in the subsequent work.

Table 2: Study the effect of order of addition

Order addition	Absorbance
M + S + R (I)	0.332
M + R + S (II)	0.314
R + S + M (III)	0.343

* M = Bi(III), R = PCV, S = Surfactant.

Effect of reagent amount:

The effect of different amounts (1.0, 1.5, 2.0) ml of pyrocatecol violet concentration 1×10^{-3} M on the absorbance of solution containing (25, 50, 75, 100, 150 and 200) µg.ml^{-1} Bi(III) was studied, it is evident that using 1 ml of 1×10^{-3} M pyrocatecol violet make the absorbance reached maximum with (R^2 = 0.999775). Therefore, 1 ml 0f 1×10^{-3} M pyrocatecol violet was used in all subsequent experiment.

Study the effect of time:

The stability of the colored complex has been studied. the expiremenal results indicate that the complex was stable for at least 24 hours.

Study the accuracy of the proposed method and precision:

To check the precision as well as the accuracy of the calibration curve, bismuth (III) was studied at (25, 50 and 100) µg. the reported results in Table 3 indicate that the method was satisfactory

Table 3: Study the accuracy and precision

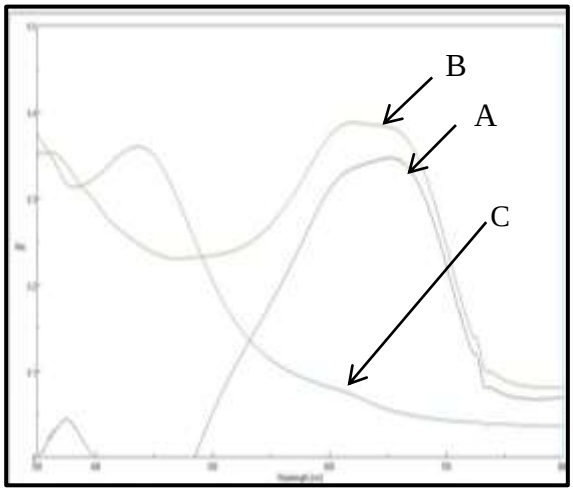
Amount of bismuth taken, µg	Amount of bismuth found, µg	Recovery, *(%)	RSD %
25	23.8	99.5	± 0.00047
50	49.0	98.1	± 0.013
100	99.4	100.0	± 0.00024

* Average of five determinations.

Final absorption spectrum:

Under the established conditions which recorded as above, absorption spectra of a blue colored complex of Bi(III)-PCV-CTAB and of its reagent blank were recorded and were shown in Fig.(2).The colored complex exhibiting maximum absorbance at 648 nm contrast with the reagent blank which shows maximum absorbance at 520 nm.

Figure 2.: Absorption spectra for 100 µg Bi(III) complex /25 ml measured (A) against reagent blank,



(B) against distilled water, (C) blank measured against distilled water

Recomended procedure and calibration graph:

Sample aliquots containing 5-200 µg of bismuth solution were placed into 25 ml volumetric flask. To each flask 8 ml of CTAB and 1 ml of pyrocatecol violet (1×10^{-3}) M were added(in order III of addition). All flasks' solution were mixed well and diluted with distilled water to the mark, the absorbance of each colored solution was measured at 648 nm against the reagent blank, The range of Beer's law is (5.0-200.0) µg of Bi(III) in 25 ml as a final volume, (i.e. 0.2-8.0) ppm, the values of molar absorptivity and factor Sandell's sensitivity index were 1.72×10^{4} l.mol^{-1}.cm^{-1} and 0.01215 µg.cm^{-2} respectively Fig.(3).

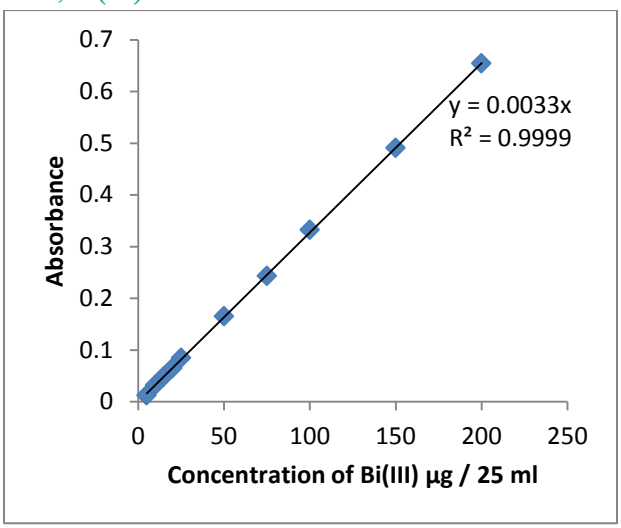


Figure 3: Calibration curve for Bi(III)complex determination via proposed method

Nature of the coloured complex:

The stoichiometry of the coloured complex has been studied by containous variations method (Job's method)[19]. The results reveal that the combination ratio of bismuth (III) with pyrocatecol violet was (1:2).

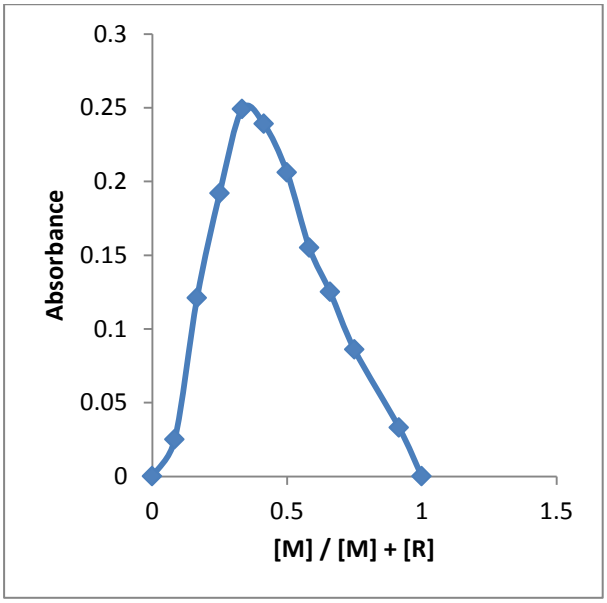
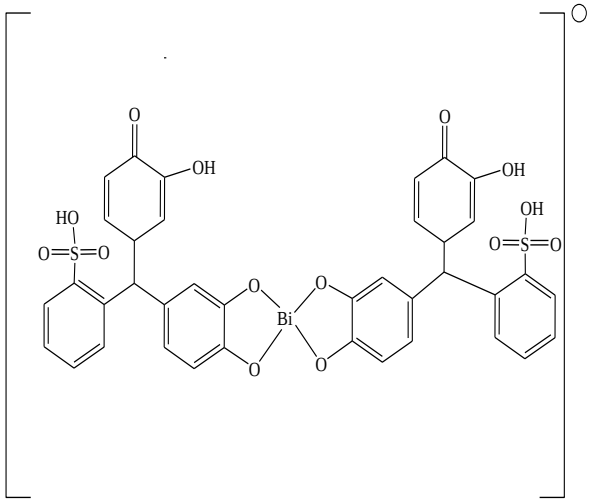


Fig 4. Job's plot for Bi(III) complex – pyrocatecol violet.

The suggested illustrated below



Bi(III)-Pyrocatecol violet complex

Effect of interferences:

The influence of divers ion on the determination of bismuth was examined under the conditions of standerd procedure. The divers ion were added, individually, to solutions containing 100 µg of bismuth(Table 4). The results were sammarised in Table 4, from which it can be concluded that the method seems to be selective except towards Ba^{+2}, Mn^{+2}, Al^{+3}, Fe^{+2}.

Table 4: Effect of interferences

Foreign ion	Form added	Error % µg of interferences added		
		50	100	300
Al^{+3}	$AlCl_3.6H_2O$	-28.3	-33.4	-63.6
Ag^{+}	$AgNO_3$	-2.10	-4.02	-3.31
Ba^{+2}	$BaCl_2.2H_2O$	+0.30	-9.33	-11.74
Be^{+2}	$BeSO_4.2H_2O$	+2.7	+3.9	+3.31
Ca^{+2}	$Ca(NO_3)_2$	-1.5	-3.01	-6.32
Fe^{+2}	$FeSO_4(NH_3)_2SO_4.6H_2O$	-13.25	-31.92	-57.2
Fe^{+3}	$Fe(NO_3)_3.9H_2O$	0.90	-2.10	-3.01
Hg^{+2}	$Hg(NO_3)_2$	0.30	-0.30	+0.90
K^{+}	KBr	+0.301	-2.40	-6.32
Mg^{+2}	$MgCl_2.6H_2O$	-0.60	-0.30	+2.10
Mn^{+2}	$MnCl_2$	-28.9	-41.26	-62.9
Zn^{+2}	$Zn(Ac)_2.5H_2O$	+2.10	+3.01	+4.21
$S_2O_3^{-2}$	$Na_2S_2O_3.5H_2O$	-2.71	-3.61	-3.91
SO_3^{-2}	Na_2SO_3	+3.91	+4.51	+5.41
SO_4^{-2}	Na_2SO_4	+3.61	+3.01	+4.21
PO_4^{-3}	$Na_2H_2PO_4.2H_2O$	+2.10	+0.30	-3.01
Pb^{+2}	$Pb(NO_3)_2$	-2.71	-3.31	-3.91

Application part:

The present method has been applied to determine Bismuth (III) in various samples of water, the results were shown in (Table 5).

Table 5: Estimation of Bismuth (III) in various sample of water

Water sample (ml)	Bi(III) added, µg	Recovery(%) of Bi (III)			
		Natural spring water	Tap water	Well water*	Bekaal fall water**

1	25	98.1	98.1	98.1	101.8
2	25	99.0	99.0	99.0	102.8
3	25	101.8	100.9	101.8	100.9
1	50	101.0	100.0	101.8	98.1
2	50	102.8	99.0	100.9	104.6
3	50	102.1	101.8	101.2	101.8
1	100	96.2	98.1	96.2	103.7
2	100	00.0	99.0	97.1	105.6
3	100	101.2	102.1	101.5	102.1

*Well water from Algubba near Mosul /Iraq.
**Bekaal fall water in Erbil city/ Iraq.

Determination of bismuth in pharmaceutical preparation.

The results were listed in Table 6 indicated that the proposed method has been applied for determining Bi(III) in pharmaceutical preparation.

Table 6: Determination of Bi(III) in pharmaceutical preparation.

Pharmaceutical preparation	Bi(III) Drug µg	Recovery * (%)
Bismuth(262 mg, Tablet) Allegiant Health	25	102.3
	50	98.1
	100	100.1

The above results reveal that the method was suitable for determination of bismuth in the above sample with satisfactory recovery. Both the present method and the literature method [20] have been applied at the same time to t-test Table 7 (confidence level 95%, degrees of freedom=5)[21].

Table 7: The result of t-test analysis.

Drug	Present method		Literature method[20]		t-test	t-tabulated
	µg Bi(III) taken	R%*	µg Bi(III) measured	R%*		
Bismuth (262 mg) Allegiant Health	100	100.1	100	99.9	0.581	5.05

* Average of five determinations.

Comparison of method:

Table 8 shows the analytical variable of the comparison of the present method with those of other literature method for bismuth determination.

Table 8: The comparison of the proposed method with literature methods

Analytical variables	Proposed method	Literature method [20]	Literature method [22]
Reagent	pyrocatechol violet	Bromopyrogallol red	4-(2-pyridylazo) resorcinol)
Surfactant	CTAB	CPC+Triton X-100	CPC
pH	3.4	3.0	5.0
λ_{max} (nm)	648	644	532
Beer's law (ppm)	0.2-8.0	0.12-6.0	0.2-8.0
Molar absorptivity ($l.mol^{-1}.cm^{-1}$)	1.72×10^4	3.3×10^4	3×10^4
Color of the product	Blue	Blue	Red
Application of the method	Pharmaceutical preperation and water samples	Pharmaceutical preperation and water samples	Pharmaceutical preperation and water samples

The results in the table 8 show the propsed method is simple ,sensitive ,rapid ,stable and can be applied to the determination of of bismuth in various water sample and pharmaceutical preparation .

CONCLUSION

The proposed spectrophotometric method is simple, sensitive and low cost, it dosenot involve solvent extraction step, and gives precise and accurate results. The method has been applied successfully to the determination of bismuth in water sample and in a pharmaceutical preperation.

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لتقدير الطيفي للبزموث باستخدام صبغة البايروكاتيكول البنفسجية بوصفها كاشف كروموجيني

ايناس سمير ذنون **خالدة محمد عمر**

قسم الكيمياء، كلية العلوم، جامعة الموصل

الملخص

تم اقتراح طريقة طيفية بسيطة وسريعة ومباشرة لتحديد البزموث (III). تعتمد الطريقة على تفاعل البزموث (III) مع البايركاتيكول البنفسجي في محلول مائي وبوجود سيتايل ثلاثي مثيل امونيوم بروماید CTAB عند الدالة الحامضية ٣.٤ لتكوين معقد ملون له أقصى امتصاص عند الطول الموجي ٦٤٨ نانومتر. كانت حدود قانون بير ضمن مدى التراكيز (٥-٢٠٠) مايكروغرام / ٢٥ مل أي (٠.٢-٨.٠) جزء لكل مليون جزء ومعامل الامتصاص المولاري (١.٢٧×١٠^{٤}) لتر.مول$^{-١}$.سم$^{-١}$ ودلالة حساسية ساندال (0.01215) مايكروغرام.سم$^{-٢}$. طبقت الطريقة لتقدير البزموث بنجاح في نماذج مائية مختلفة وفي المستحضر الصيدلاني(الاقراص الدوائية).

الكلمات المفتاحية: التقدير الطيفي، سيتايل ثلاثي مثيل امونيوم بروماید CTAB ،البزموث (III)، بايروكاتيكول البنفسجي.