Spectrophotometric Determination for Mercury(II),Lead(II) and Zinc(II) with 2-[(6- Methyl-2-Benzothiazolylazo)]-4-Chloro phenol as Organic Reagent

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Abstract:

A sensitive and selective spectrophotometric method is proposed for the rapid determination of Mercury(II),Lead(II) and Zinc(II) using (6-MeBTAClP), as spectrophotometeric reagent. The (6 – MeBTAClP) reagent gives intense green colour when reacts with these in acetate buffer solution. The maximum absorbance observed at 637 nm,651 nm and 610 nm for Hg(II),Pb(II) and Zn(II) respectively. The molar absorptivity for Mercury(II),Lead(II) and Zinc(II) complexes is (9521.2, 12714.2 and 12748.4) $L.mol^{-1}.cm^{-1}$ respectively. The Mole ratio 1:2 [M:L] for the complexes of Hg(II),Pb(II) and Zn(II). The linear range of(1-35),(1-18) and(1-16) respectively along with limit of detection 0.406, 0.05 and 0.188 μg mL^{-1}. Relative standard deviation for seven replication measurements (2.71% Hg, 0.171% Pb and 0.209% Zn),and the recovery range of (97.43) , 97.65 and (97.95) were obtained for Hg(II), Pb(II) and Zn(II) ions ,and E_{rel}=2.57 , 2.047 , 2.35 for Hg(II),Zn(II) and Pb(II) respectively. . The most important interference were due to Ni^{2+}, Zn^{2+}, Cu^{2+}, WO_4^{2-}, Pb^{2+},MoO_4^{2-} , Hg^{2+}, and CrO_4^{2-} and suitable masking agents were used.

Key words:-Thiazolylazo , Spectrophotomitric determination of Mercury(II),Lead(II) and Zinc(II).

Introduction:

The hetrocyclic azo dyes have been synthesized and proposed as highly sensitive chromogenic reagent for the determination of several metal ions[1].Among the hetrocyclic reagents pyridylazo ,thiazolylazo and benzothiazolylazo dyes are extensively used for the spectrophotometric determination for metal ions[2].Direct determination of trace and toxic heavy metals such as Hg,Pb and Zn in some samples has been important[3]. Heavy metal pollution of the environment, particularl by lead has received a considerable attention as it leads to many fatal diseases including dys function of renal blood and neurological systems.Again, Pb(II) easily deposits in brain, kidney,

reproductive and nervous systems. Acute lead poisoning can result severe anemia, colic shock and irreversible brain damage. Several works on the solid phase extraction of lead by incorporating different chelating groups on the polymer backbone has been reported.Among,2.aminothiophenylSacetica cid,dithiooxamide,oxine,resacetophenone,o-aminophenol,o-aminobenzoicacid,zirconium phosphate,pyrocatechol violet,quinalizari and imidazolylazo anchored/supported polymers are noteworthy[4]. Several techniques such as flame atomicabsorption ,voltammetric and spectrophotometric methods have been used for the determination of the ion in different samples. Among this, uv-visible spectrophotometry is the most commonly

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used techniques for Pb(II)[5]. Zink (Zn) is essential for animale It is often found in food ,drink and grains.It is a representative of the group of microelements, thus in small quantities it is essential for human, animal and plant growth.Zinc influences living processes and displays regulatory functions, as it participates in the content of above 40 enzyme systems. Food zinc deficiency of men has been reported in a number of countries [6],[7]. The strong toxicity is due to the swelling of too large quantities of zinc resins, accidentally or intentionally[8].Mercury is one of the most toxic heavy metal in the earth and it exists in nature at trace and ultratrace amounts in three valence states[1]. Mercury (0, I, II) species and are able to combine with most inorganic and organic ligands to form various complexes,$HgX4^{-2}$(whereX=Cl,Br and I) and methyl mercury[1,2].Mercurycan

Experimental Apparatus:

(FTIR)Spectra(4000-400cm^{-1})in KBr disk were recorded on a SHIMADZU FTIR-8400S fourier. transform. infrared. Spectrophotometer.\(Japan),absorption spectra was measured on aT80 UV-Vis spectrophotometer and absorbance was measured on Apel PD -303UV-visible spectrophotometer using 1 cm quartz cells . A model WTW multi 720 pH – meter was used to adjusted and measure the pH of the solution , Melting point was measured using SMP30 Stuart, UK.

Reagents and Solutions:
Preparation of Reagent[12]

mixture of (2.142 gm of para methyl aniline and 3.650 gm of ammonium thiocyanate) in 70 ml glacial acetic acid ,was added drop by drop from burette (1.2 ml Br2 + 15 ml glacial acetic acid) with keep temp. 10 °C.After 15 min alkaline solution was added to precipitate the thiazole derivative . 1.330 gm of thiazole and in 50 ml glacial acetic acid then add (5 ml conc.

accumulate in animals and plants and also enters into human bodythroughthe foodchaincausingdamagetocentralnervoussy stem[9]. Due to the toxicological effects andpotential accumulation of mercury onto human bodies and aquatic organisms, the determination of mercury (II) or organo mercury (II) hasseenanupsurge of interest in the last few years[10]. The toxicity of mercury depends on its chemical state [11]. Some forms of mercury are relatively non-toxic and have been used as medicines,for the treatment of syphilis [11]. In this work a **2-[(6–Methyl–2-Benzothiazolyl) azo] -4-Chlorophenol**, was prepared by Aqeel Mahdi[12] .and used,as an analytical reagent for the micro determination of Pb(II),Hg(II) and Zn(II). The method has been found to be simple, rapid and sensitive for the determination of these metal ions.

HCl +25 ml water) to the solution . After that drop by drop from burette a solution (0.69 gm NaNO2 +50 ml H2O) with stirring at 10 °C to diazonium salt , then (1.300 gm of para chloro phenol +50 ml ethanol) to diazonium salt to the 2-[(6–

Methyl–2-Benzothiazolyl) azo]- 4-Chloro phenol (6-MeBTAClP) Organic reagent.
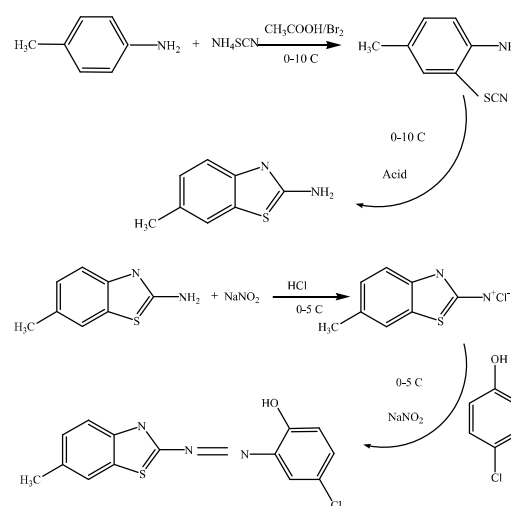
All the chemicals used were of analytical reagent grade, and were used without further

purification. Ethanol were purchased from (GCC, England).

-1x10^{-3}M (6-MeBTAClP) standard solution was prepared by dissolving (0.754)g in 250 ml of absolute ethanol .

-Stock Hg(II) solution ;A solution of Hg(II) (1000 µg.ml^{-1})was prepared by dissolving (0.13532)g of HgCl$_2$ (BDH) in (100 ml) deionized water. Other standard solutions of Hg(II) were prepared by dilution of stock solution with distilled water.

-Stock Pb(II) solution ;A solution of Pb(II) (1000 µg.ml^{-1})was prepared by dissolving (0.15984)g of Pb(NO$_3$)$_2$ (BDH) in (100 ml) deionized water. Other standard solutions of Pb(II) were prepared by dilution of stock solution with distilled water.

-Stock Zn(II) solution ;A solution of Zn(II) (1000 µg.ml^{-1})was prepared by dissolving (0.5210)g of ZnCl$_2$ (Merck) in (100 ml) deionized water. Other standard solutions of Zn(II) were prepared by dilution of stock solution with distilled water.

- The pH of the medium (2-9) were adjusted with ammonium acetate (0.01 mol L^{-1})– ammonia – glacial acetic acid buffer solution .

General procedure:

Into a 10 mL calibrated flask , transfer 1 mL of sample solution containing less than 100 µg.ml^{-1} of Hg(II) and adjust the pH to 7 with ammonium acetate buffer, add 2 ml 1.0×10^{-4}M ethanolic (6-MeBTAClP) solution and diluted to the mark with deionized water. The absorbance of the resultant solution was measured after 10 min at 637 nm at 25 C° against the

corresponding reagent blank prepared under identical conditions but without mercury. The same procedure for zink (II) at pH 4.5 and the absorbance of the result solution after 10 min at 410 nm and for lead (II) at pH 6.5 and the absorbance of the result solution after 15 min at 651 nm.

Results and Discussion:

FTIR Spectra of the Reagent(6-MeBTAClP) and its complexes:-

FTIR spectral data of the reagent **(6-MeBTAClP)** and its complexes are summarized in Table (1) .The spectra are complicated owing to the extensive overlap of a number of bands arising from O-H , C=N and N=N groups in addition of other bands originated from phenyl and heterocyclic thiazole ring which appeared in the region 400-4000 cm^{-1}.The shifts in the positions of these bands compared with those absorption bands due to the free ligand suggest the probalble modes of bonding in the complexes . Some of these main shifts along with conclusions are given below:-

1- The FTIR spectrum of reagent (6-**MeBTAClP**) gives characteristic broad band at(3200) cm^{-1} assigned to the phenolic υ(O—H). This suggests a strong intermolecular hydrogen bonding [13]. In the spectrum of Pb(п) and Zn(п) complexes show broad and weak bands around (3400-3059)cm^{-1} indicate the presence of water molecule in these complexes.

2- This weak band which has been observed at (3100) cm^{-1} in the spectrum of the free reagent was due to υ(C—H) aromatic. The positions of these bands in the spectra of Hg and Zn complexs are shifted to a lower frequencies (3092-3026) cm^{-1} with decreased in intensity , but the Pb complexe are shifted to(3130)cm^{-1}.

3- The spectrum of reagent shows absorption band at (1700) cm^{-1} which is

considered to be υ(C=N) of thiazole ring[14].This band shifts to lower frequency (1600-1592-1600) cm^{-1} with a little change in shape in the metal complexes spectra.

4- The azo group intens bands at(1483)cm^{-1} due to the —N=N— group .The band appearing at(1491)cm^{-1} to Hg complex and(1488)cm^{-1} to Pb complex and(1452)cm^{-1} to Zn complex respectively with some decrease in intensity of metal complexes spectra. All bands shift and reduction in intensity may indicate that azo group is coordinated to the metal ions to the formation complexes[15].

5- The υ(C—S) of thiazole ring appears (854) cm^{-1} in the spectrum of free reagent. this band unchanged in all complexes means that the sulfur atom of hetero cyclic ring does not participate in coordination[2] .

6- New weak bands in the region (420-563)cm^{-1} which are never been observed in the free reagent spectrum,this may be attributed to υ(M—O) , υ(M—N) , υ(M—Cl) bands[16].
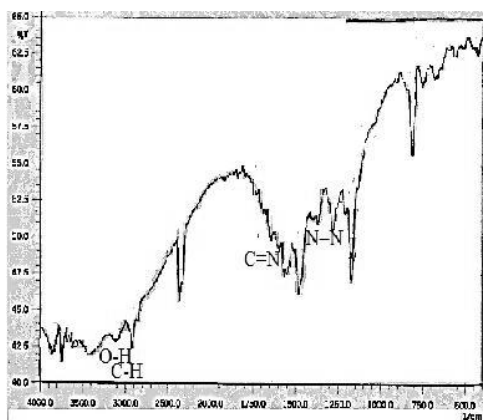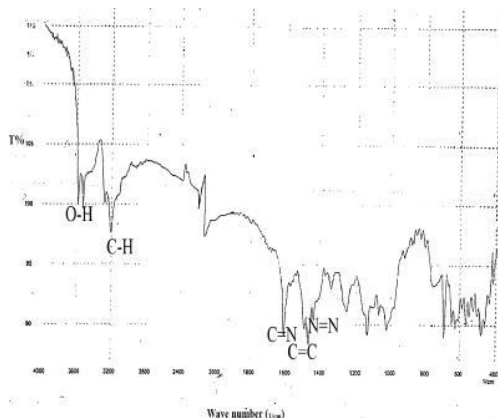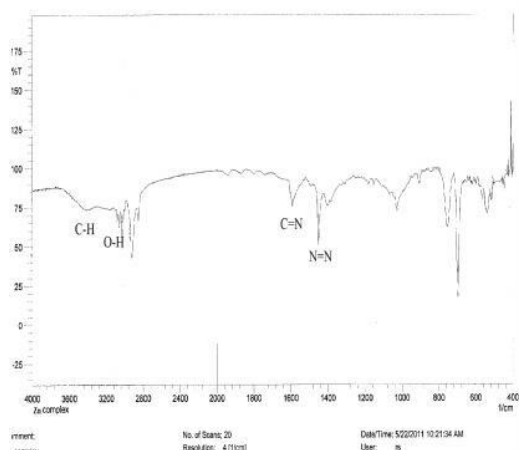

Fig (1):-FTIR Spectrum of the reagent (6-MeBTAClP)

Fig(2):-FTIR Spectrum of the Hg complex

Fig(3):-FTIR Spectrum of the Pb complex

compound	OH,H2O	C-H Aromatic	C=N	C=C	N=N	C-H Aliphatic	C-S
(6MeBTAClP)	3200	3100	1700	1510	1483	2865	854
Hg(II)- (6-MeBTAClP)	3510-3589	3092	1600	1510	1491	3200	855
Pb(II)- (6-MeBTAClP)	3400	3130	1592	1660	1488	2880	857
Zn(II)- (6-MeBTAClP)	3059-3082	3026	1600	-----	1452	2850	851

Table(1)Selected I.R. bands of reagent (6-MeBTAClP) and its complexes

Absorption spectra and characteristics of the complex:

The UV- Vis spectrum of an ethanolic solution of the reagent (**6-MeBTAClP**) (1×10^{-3} M) showed peak (λ max) was observed at the (430 nm) due to the (π-π^*) from aromatic ring through the azo group (charge transfer) was referred to the n- π^* transition of intermolecular charge- transfer taken place from benzene through the azo group(-N=N) Interaction of the metal ions Zn(II),Hg(II) and Pb(II) with the reagent has been studied in aqueous ethanolic solution . A bathchromic shift of Zn(II),Hg(II) and Pb(II) complexes show the absorption maxima of 610,637 and 651 nm referred to (charge transfer) with molar absorptivities

(ε) of 1.27142×10^{-4} L.mol^{-1}.cm^{-1} , 9.5212×10^{-3}L.mol^{-1}.cm^{-1}and 1.27484×10^{-4} L.mol^{-1}.cm^{-1} obtained respectively while the reagent gave the absorption maxima of 430 nm as depicted in Fig. 5. The wave length different(Δ λ_{max}) is (180-207-221 nm) ,a great bathochromic shift in the visible region has been detected in the complex solutions spectra with respect to that of the free reagent. The high shift in the (λ_{max}) gave a good indication for complex formation.
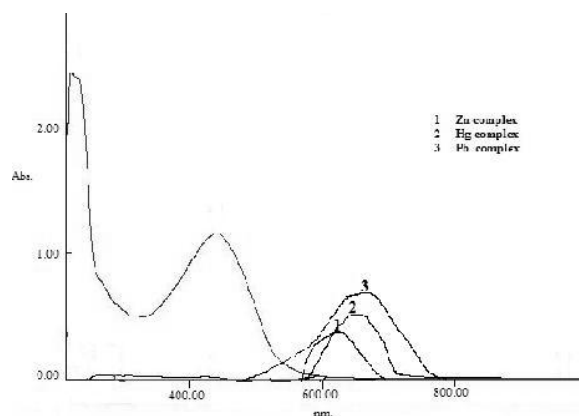

Fig.(5): Absorption spectra   Reagent (6-MeBTAClP) = 1.0 x 10^{-3} M   (1) Zn(II)-(6-MeBTAClP) complex{[10 ppm] in pH=4.5}   ,(2) Hg(II)-(6- MeBTAClP) complex{[10 ppm] in pH=7} and (3)Pb(II)-(6-MeBTAClP) complex{[10 ppm] in pH=6.5}

Method Validation:

Under the optimized conditions, the calibration graphs were constructed by plotting the absorbance signal against the concentrations of each analyte subjected according to the general procedure. The solutions were transferred into the optical

cell of 10-mm for the measurement of each metal ion spectrophotometrically at the respective absorption maxima against a reagent blank prepared under similar conditions .The calibration data and some analytical parameters are summarized in Table(2).
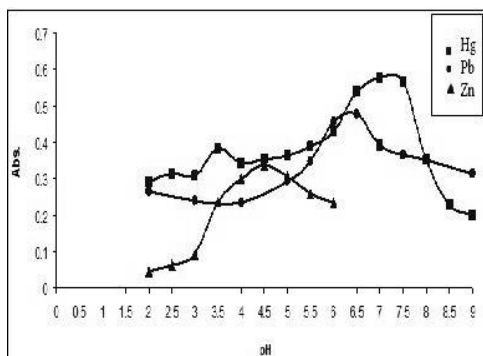
Table (2): Analytical data and some analytical parameters of the proposed method for determenation of Hg(II),Pb(II) and Zn(II)

(N=no. of determination)

Analytical Parameter	Zn (II) / ppm	Pb (II) / ppm	Hg (II) / ppm
λ_{max} (nm)	410	651	637
Regression equation	Y=0.0355X-0.0032	Y=0.0255X+0.2177	Y=0.0193X+0.0067
Molar absorptivity(L.mol^{-1}.cm^{-1})	12714.2	12748.4	9521.2
Sandell Sensitivity(μg.cm^{-2})	0.0282	0.0392	0.0518
Correlation coefficient(r)	0.997	0.9979	0.9992
Detection Limit(D.L)	0.05	0.188	0.406
Linear dynamic range	1-16	1-18	1-35
Standerd deviation	0.00058	0.001	0.00876
Relative . Standard . Deviation %	0.171 (N=7)	0.209 (N=7)	2.71 (N=7)
Percent Relative error	2.047	2.35	2.57
Percent Recovery	97.95	97.65	97.43
Composition of complex (M: L)	1:2	1:2	1:2

These results indicating that this method is highly precise and suitable for the determination of Hg(II) ,Pb(II) and Zn(II) spectrophotometrically
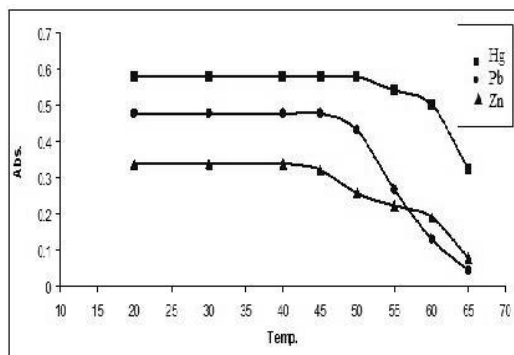
Optimization of Procedure:

Effect of pH

The effect of pH on formation of the (Hg(II) ,Pb(II),Zn(II))- (**6-MeBTAClP**) complexes was determined by recording their absorbance signals at λ_{max} ,over the range of 2-9 ,using different pH buffer solution (NH$_3$+ CH$_3$COOH). The results are shown in Fig.6.

Fig.(6): Effect of pH on the formation of(6-MeBTAClP)-Mcomplexes formed with Hg (II),Pb(II) and Zn(II)).

As can be seen in Fig.(6), the absorbance first increased with increasing pH and reached a maximum at pH 4.5 for Zn(II) and 6.5 for Pb(II) and 7 for Hg(II) complexes ,respectively. The absorbance gradually decreased may be deu to

formation another chromophoric formala of reagent at higher pH . Therefore, pH4.5 , 6.5 and 7 were selected as the optimum pH's for complete formation of for Zn(II) Pb(II) and Hg(II) complexes, respectively .

Effect of Temperature and Time

The effects of temperature and time were examined due to their importance for the reaction completion . Consequently, a study was carried out to choose the range of temperature that enhances higher absorbance

signals for Hg(II) ,Pb(II) and Zn(II) ions. The temperature was varied from 10 $^{\circ}$C to 80 $^{\circ}$C in a search of optimum value. It can be seen from Fig. 7 that the highest absorbance signals were achieved when the temperature at approximatly (20-40) $^{\circ}$C.

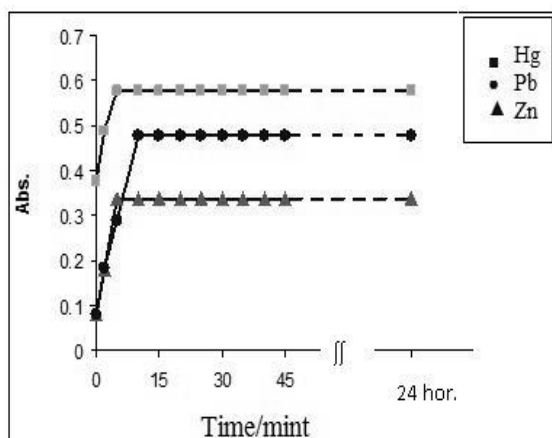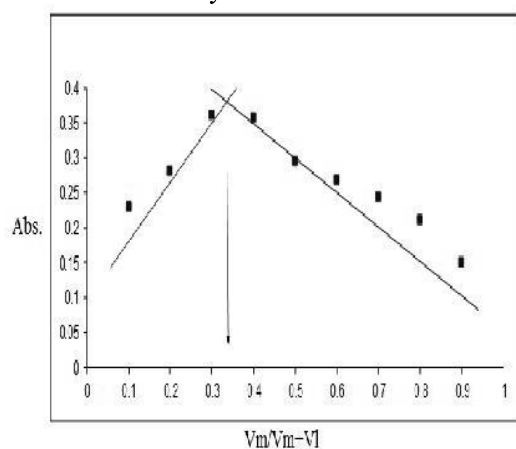**Fig.(7): Effect of the temperature on the absorbance for (6-MeBTAClP)- M complexes formed with Hg (II),Pb(II) and Zn (II)).**

It was also observed that the incubation time of 15 min for Pb(II) and 10 min are sufficient for the maximum absorbance of Hg(II) and Zn(II) ,respectively.

Composition of the complex:

The composition and the stability constant were evaluated by both of the continuous.

variation and the mole ratio methods (fig.9-14). Both methods showed that the molar ratio of Hg(II),Pb(II) and Zn(II) ions to reagent (**6-MeBTAClP**) is 1:2, (metal : ligand). The stability constant is found to be 6.06×10^9 L.mol^{-1} ,1.05×10^9 L.mol^{-1} and 4.93×10^9 L.mol^{-1} for Hg(II)Pb(II) and Zn(II) respectively.
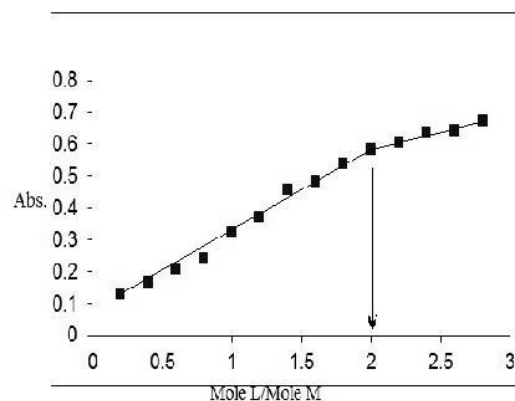

Fig (10):- Mole ratio method for Hg complex with (6-MeBTAClP)at pH=7

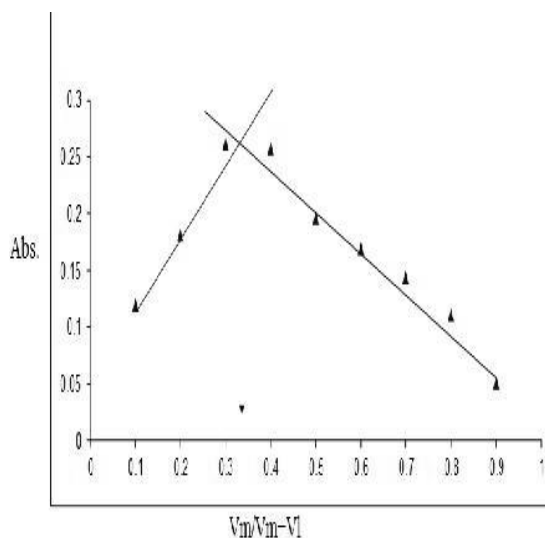Fig(9):-Cotinuous variation method for Hg complex with (6-MeBTAClP)at pH=7

Fig (11):- Cotinuous variation for Zn complex with (6-MeBTAClP)at pH=4.5
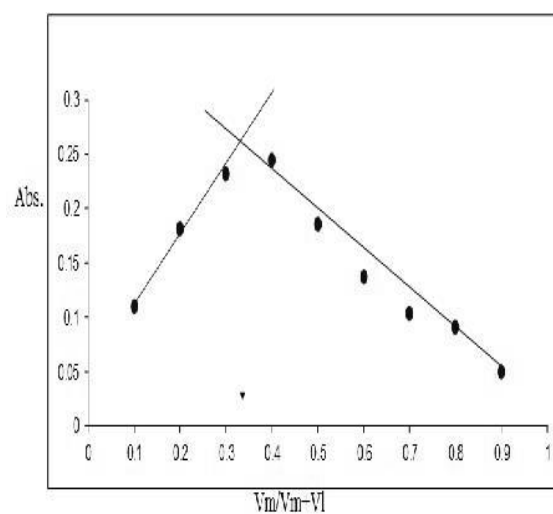

Fig (13):- Cotinuous variation method for Pb complex with (6-MeBTAClP)at pH=6.5

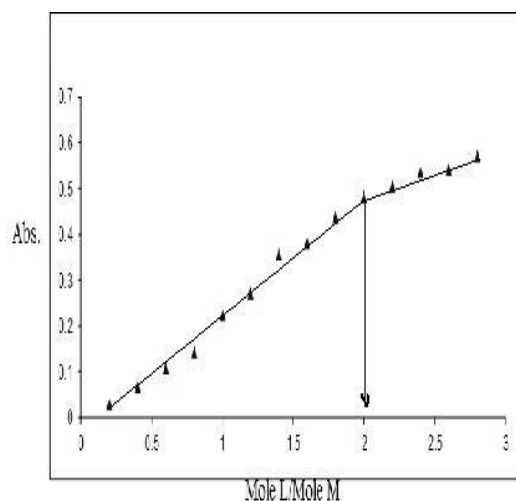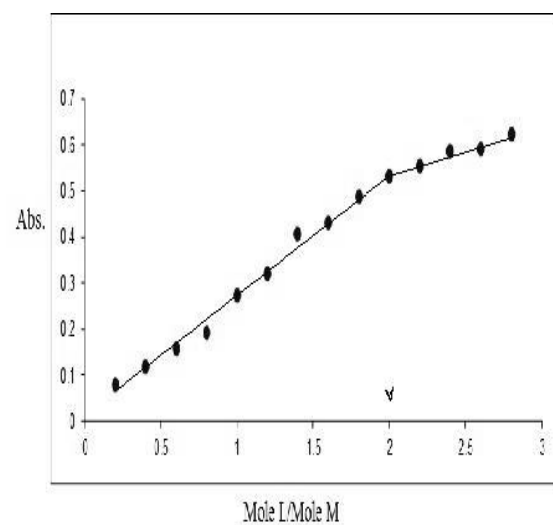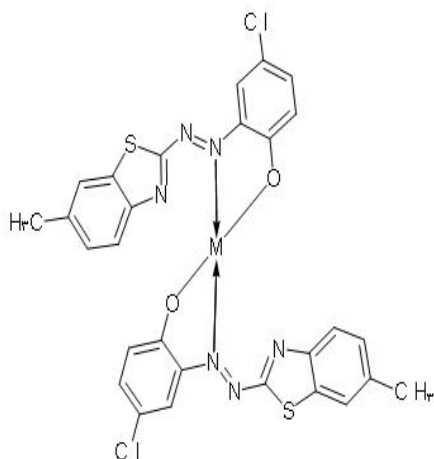**Fig (12):- Mole ratio method for Zn complex with (6-MeBTAClP)at pH=4.5**

Fig (14):- Mole ratio method for Pb complex with (6-MeBTAClP)at pH=6.5

Suggestion of structural formula of complexes:

From the obtained results of metal to reagent ratio ,FTIR-Spectrum ,and depending on thiazolylazo compounds properties; the following structure can be suggested :

Where M=Hg,Pb,Zn

Interference studies Mercury(II),Lead(II) and Zinc(II) complexes:

The effect of the interference of ions which form complexes with the reagent (**6-MeBTAClP**) during its reaction with {(Mercury,Lead and Zinc)10 ppm} were studied. The selectivity of various masking agents are examined for eliminating the effect of the interfering eight ions for each one . These are 5-Sulfosalicylic acid , NaNO2,Sodium acetate, Ascorbic acid, Tartaric acid, $Na_2S_2O_3$,KCl and KI. The results are shown in tables (3-5).

Table(3): Effect of foreign ions on the determination of Hg(II) in concentration (10)ppm and suitable masking agents .

Foreign ion 100 ppm	[0.01]M,Masking agent(1.0)ml	Error%
Cu^{2+}	5-Sulfosalicylic(0.5)[0.01]	0.002
Ni^{2+}	$NaNO_2$(0.2)[0.1]	0.003
CrO_4^{2-}	$NaNO_2$(0.4)[0.1]	0.002
Zn^{2+}	Sodium acetate (0.1) [0.01]	-0.003
Pb^{2+}	Ascorbic acid (1.0)[0.01]	-0.003
WO_4^{2-}	Tartaric acid (1.75) [0.01]	0.0
Cd^{2+}	$NaNO_2$(0.1)[0.1]	-0.002
MoO_4^{2-}	Tartaric acid (0.2) [0.4]	0.004

Table(4): Effect of foreign ions on the determination of Pb(II) in concentration (10)ppm and suitable masking agents .

Foreign ion 100 ppm	[0.01]M,Masking agent(1.0)ml	Error%
Cu^{2+}	Ascorbic acid (1.0)[0.03]	0.004
Ni^{2+}	Ascorbic acid (1.0)[0.02]	0.005
CrO_4^{2-}	$Na_2S_2O_3$(0.2)[0.1]	0.002
Zn^{2+}	KCl (0.2) [0.01]	0.01
Pb^{2+}	Sodium acetate (3.0)[0.01]	0.01
WO_4^{2-}	KI (1.0) [0.01]	0.004
Hg^{2+}	$NaNO_2$(0.1)[0.1]	0.0
MoO_4^{2-}	Tartaric acid (0.1) [0.2]	0.0

Table(5): Effect of foreign ions on the determination of Zn(II) in concentration (10)ppm and suitable masking agents .

Foreign ion 100 ppm	[0.01]M,Masking agent(1.0)ml	Error%
Cu^{2+}	Ascorbic acid (1.0)[0.03]	0.004
Ni^{2+}	Ascorbic acid (1.0)[0.02]	0.005
CrO_4^{2-}	$Na_2S_2O_3$(0.2)[0.1]	0.002
Zn^{2+}	KCl (0.2) [0.01]	0.01
Pb^{2+}	Sodium acetate (3.0)[0.01]	0.01
WO_4^{2-}	KI (1.0) [0.01]	0.004
Hg^{2+}	$NaNO_2$(0.1)[0.1]	0.0
MoO_4^{2-}	Tartaric acid (0.1) [0.2]	0.0

Conclusion :

In this work 2-[(6- Methyl-2-Benzothiazolylazo)]-4-Chloro phenol was used to determination of microamounts of Zn(II) , Pb(II) and Hg(II) . The molar absorpinity of this complaxes are $1.2714×10^4$, $1.2748×10^4$, $9.521×10^3$ l.mol.cm^{-1}respectirelly.Most foreign ions donot interfere with ions study when masked using defferent masking agents . Because the (6-MeBTACIP) can rapidly react whith Zn(II), Pb(II) and Hg(II) at room temperature , this method can determine these ions spectrophotometrically whithout heating or extraction . It is sensitive , selective , rapid , easy and convenience method for determined the ions Zn(II) , Pb(II) and Hg(II).

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التقدير الطيفي للزئبق (II) , الرصاص (II) والزنك (II) باستخدام الكاشف العضـــوي 2-(6-مثيل-2- بنزوثيازوليل ازو) -4- كلورو فينول

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الخلاصة:

يتضمن البحث تقدير الزئبق($\prod$) ,الزنك($\prod$) والرصاص ($\prod$) بطريقة طيفيه حساسة وإنتقائيـــة وسـريعة بإسـتخدام كاشف (6-
MeBTACIP) . أن التفاعل بين الكاشف المذكور وأيونات الزئبق($\prod$) ,الزنك($\prod$) والرصاص ($\prod$) يكون سريع وحساس عند الطـول
الموجي للأمتصاص الاعظم (λ_{max}637=nm) و pH=7 ليعطي معقد اخضر زيتوني لمعقد الزئبـق , (λ_{max}610=nm) و pH= 6.5 و
معقد اخضر لمعقد الزنك و(λ_{max}651=nm) و pH=4.5 و معقد اخضر زيتوني لمعقد الرصاص وبنسبة مولية 2:1 (فلــز : ليكانــد)
وإن الامتصاص يكون مستقر من (1-24) ساعة لكل المعقدات السابقة,حيث كانت التراكيز التي تطاوع قانون بير مسـاوية الـى (1-
35),(1-16) و(1-18) مايكروغرام /مل وقيمة معامل الامتصاص المولاري (ϵ)هي (12714.2, 9521.2 و12748.4) لتر.مول⁻
¹ . سم⁻¹ .وحد الكشف (0.406 ,   0.05   و 0.188)مايكروغرام/ مل كما تم تحديد دقة وضبط الطريقة التحليلية حيـث وجـد إن
قيمة الانحراف القياسي (R.S.D%) هي2.71 , 0.171 و 0.209 والاستعادية(Re%) 97.95,97.43 و97.65 والخطـأ النسـبي
(E$_{rel}$) هو2.047,2.57 و2.35 للزئبق ,الزنك والرصاص على التوالي . تم دراسة تأثير الأيونـات المتداخلــة وهـي ⁺Ni², Zn²⁺,
Hg²⁺, Cu²⁺, WO₄²⁻, Pb²⁺,MoO₄²⁻ و CrO₄²⁻ وأستخدم لهاعوامل حجب قياسية .

مفتاح الكلمات:- ثيازوليل ازو , التقدير الطيفي للزئبق, الرصاص والزنك .

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