REVIEW OF RESEARCH

DEVELOPMENT OF AN EXTRACTIVE AND SPECTROPHOTOMETRIC DETERMINATION OF LEAD(II) WITH N,N''-BIS (O-HYDROXY-ACETOPHENONE) ETHYLENEDIIMINE (HAPED) DERIVATIVE AS AN ANALYTICAL REAGENT

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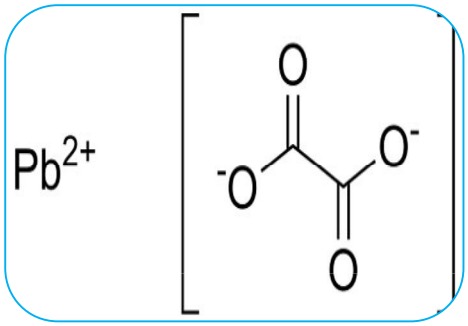

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ABSTRACT:

The Spectrophotometric method is coupled with solvent extraction technique and used for the determination of Pb(II) using N,N''-bis(O-hydroxy -acetophenone) ethylene diimine(HAPED) as an analytical reagent. This reagent is synthesised in the laboratory and characterised by NMR, IR, mass and elemental analysis for its purity. The reagent forms a light yellow-coloured stable complex with manganese metal, which can be quantitatively extracted into chloroform at pH 5.2. This Pb(II)-HAPED complex in chloroform exhibit intense absorption peak at 535nm. Beer's law is obeyed in the range of 1-10 ppm of Lead solution giving linear and reproducible graph. The- stoichiometric ratio of complex studied by Job's continuous variation method, Mole ratio and Slope ratio method. The molar absorptivity and Sandell's sensitivity are also calculated. The molar absorptivity is 1,165.90 L/mol/cm and Sandell sensitivity is 0.0376 µg/cm^2. The proposed method is rapid, sensitive, reproducible, accurate and has been satisfactory applied for determination and separation of Pb(II) in commercial mixtures, pharmaceutical samples and alloys.

KEYWORDS: *HAPED reagent, Lead(II), Sandell'ssensitivity, Molar absorptivity, Spectrophotometric determination.*

INTRODUCTION :

Lead compounds are essential to life. In the present study, solvent extraction methods are proposed for the metals like Pb(II), Ni(II), Fe(II), Co(II), Cu(II), Mn(II), Cr(III)etc[1]. Trace amounts of toxic heavy metals in the honey bee come most probably from contamination during bee's nutrition in polluted area where they fly or from the equipment which employed for processing and storage container. These metals have proved to be of immense importance in various chemicals, biochemical, pharmaceuticals and industrial applications[2-4]. It provides good separation and determination methods. Optimum extraction conditions are evaluated to study several experimental parameters like effect of reagent concentration, different diluents, effect of temperature etc[5]. Diverse ion studies are carried out to study the selectivity for the method. This method is used for the analysis of real sample like various alloys, pharmaceutical samples. Extractive methods are highly sensitive but generally lacks in simplicity. Spectrophotometry is essentially a trace- analytical technique and is one of the most powerful tools in chemical analysis[6]. A wide variety of reagents have been proposed for the spectrophotometric determination of Lead. The extractive spectrophotometric analysis enables to separate desired metal ion, which is to be estimated in presence of other metal from samples. In the present work a novel analytical reagent N,N'bis''(O-hydroxyace - tophenone) ethylenediimine (HAPED), was used for the extractive spectrophotometric

determination of Lead. The developed method can be employed for efficient determination of Lead at microgram level. The results of analysis obtained were compared with those obtained by known methods. The Lead is not necessary for living and considered as very important pollution for vital systems. Kostenko used Chromazural as organic reagent for spectrophotometric determination of Lead in drinking water[7]. Pellerano et al used (5-Br-PADAP) as organic reagent for determination of Lead in surface water[8]. Sonawale et al used TBPO for extraction of Pb(II) and Cu(II) and also applied this method for determination of both metals in casts natural and pharmaceutical samples[9].

EXPERIMENTAL WORK

1. Instruments:- Shimadzu 2100 UV-Visible spectrophotometer with 1.0 cm quartz cell was used for absorbance studies. An Elico LI-120 digital pH-meter was used for pH adjustment.

2. Synthesis of Reagent:- The HAPED reagent was synthesised by O-hydroxyacetophenone and ethylene diamine in methanol in 2:1 molar proportions are mixed in round bottom flask. Shake the flask for 10 to 15 min. immediately light-yellow-colour solid is obtained which is poured in ice-cold water. The solid obtained is separated by filtration and washed with cold water and the product is recrystallised from ethanol. The yield was about 95%. It is then characterised and used for extractive spectrophotometric determination of Pb(II). A stock solution of HAPED reagent with concentration 0.1% was prepared in methanol. The scheme of reaction is shown in Figure 1.

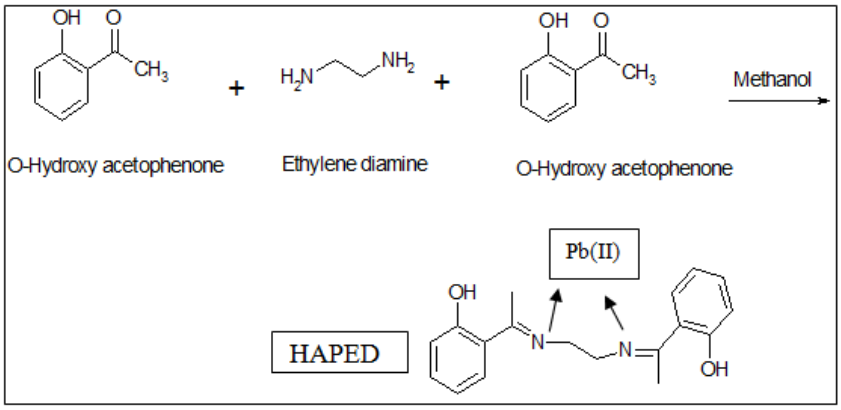
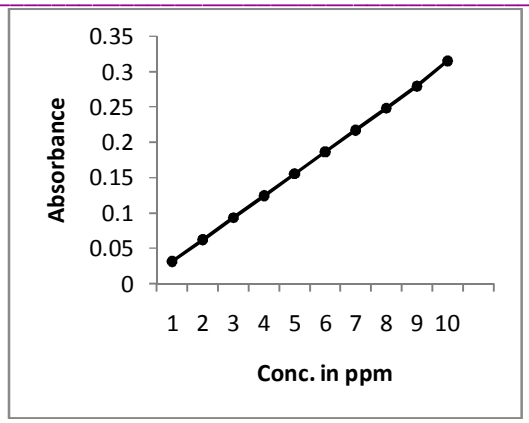


Figure-1:-Synthesis of reagent N,N"-bis (O-hydroxy-acetophenone) ethylene diimine (HAPED)

3. Preparation of stock solution:- A weighed quantity of Lead Chloride was dissolved in double distilled water containing dilute hydrochloric acid and then diluted to desired volume by double distilled water. The solution was then standardised by titrimetric Method.

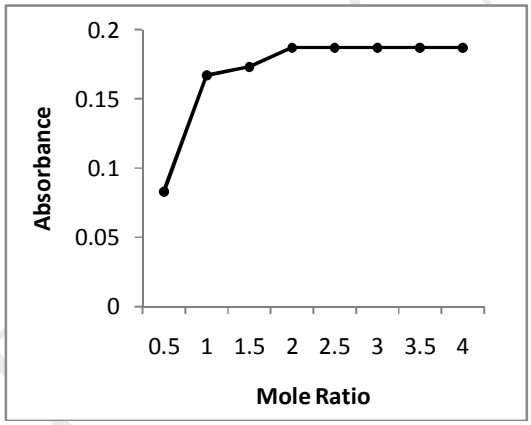
4. Recommended procedure:- Mix 1-cm^3 aqueous solution containing 1-100mg of Lead and 2 cm^3 of 0.1% methanolic solution of HAPED reagent in 25 cm^3 beaker. Adjust the pH of the solution to required value with buffer solution Make the final volume 10cm^3. Transfer the solution into 125 cm^{3}separate funnel and equilibrate for 1min. with 10cm^3 chloroform. Allow the two phases to separate and measure the absorbance of organic phase containing the complex at 535 nm against reagent blank.

5. Preparation of calibration plot:- The calibration curve is prepared by taking known amount of Lead which is described in the procedure. A graph of absorbance against concentration is shown in Figure 2. The concentration of the unknown Lead solutions is determined from the calibration plot.



Figure,.2: Calibration plot for extractive spectrophotometric determination of Lead(II) with chloroform.

6. Composition of the extracted species :- The composition of the extracted species was determined by using the Job's continuous variation method and verified by mole ratio method and slope ratio method. These methods show that the composition of Lead(II)- HAPED reagent is 1: 2 which is represented in Figure 3.



Figure,3: Composition of the Extracted Lead(II) - HAPED species by Mole ratio method

7. Effect of foreign ions:- Various cations and anions were investigated to find the tolerance limit of these foreign ions in the extraction of Lead (II) presented in Table 2. The effect of diverse ions on the Lead(II) determination was studied, in presence of a definite amount of a foreign ion. The tolerance limit of the foreign ion was taken as the amount required causing an error of not more than 2% in recovery of Lead(II). The ions which interfere in the spectrophotometric determination of Lead were masked by using appropriate masking agents presented in Table 3.

Table:1

Sr.No	Different parameters Studied	Observation
1	Solvent	Chloroform
2	pH	5.2
3	Equilibrium time	1 min.
4	Stoichiometry M:L	1:2
5	95% confidence limit	±0.2560

6	Reagent Conc.	0.1%
7	Volume of Rgt.	2cm^3
8	Average of 7 determination	9.50
9	Stability of the complex	33 h.
10	Sandellsensitivity	0.0376-µg/cm^2
11	Molar absorptivity	1,165.90 L/mol/cm

Table :2 Effect of foreign ions

Sr. No.	**Interfering ions**	**Tolerance limit**
1	Tartrate, acetate, BrO_3^-, Br^-, NO_3^-, IO_3^-, SO_4^-, SO_3^-, CN^-,	10
2	Co(II), Fe(II), Cu(II), Ni(II), Mn(II) EDTA	Interfere strongly
3	Oxalate , phosphate,	06
4	Al(III), Mg(II),Mo(VI), Cd(II), W(VI),	08
5	Al(III), Bi(III), Ce(IV),Ca(II),	12
6	Na^+, Ag^+,K^+	06

Table-3: Effect of masking agent

Sr. No.	Interfering Ions	Masking Agents
1	Pd(II)	Thiourea
2	Ce(IV), Fe(III), Co(II)	Sodium fluoride
3	Cr(III)	Ammonium acetate
4	Ni(II)	DMG
5	EDTA, Cynide ion	Boiled with conc.HNO_3

8. Comparison between reagents : Various reagents were investigated by the earlier researchers for removal of Pb(II). The proposed reagent (HAPED) is found more superior as that of reported regents and are presented in Table 4.

Table 4: Comparison between regents

Sr./ No.	**Reagent**	**Remark**
1	Diantipyryl-(p-chloro)-phenylmethane	Beer's range0-400 µg/25 cm^3
2	Hydroxamic acid	Sandell Sensitivity is poor
2	Piconaldehyde nicotinoylhydrazone	Beer's Range 0.02-1.5ppm yellow-coloured complex with M:L ratio as 1:2
3	N,N'-diethylaniline	Require heating At 100ºC
4	Methylene green	Beer's range0.2-30 cm^3
5	Ethylenebis(triphenyl Phosphonium cation	Mn^{+2i}nterferes

9. Applications : The present method was applied for determination of amount of Lead(II) in various samples of alloys, commercial mixtures, Honey sample, water sample etc. The results obtained were in well agreement with the standard methods shown in Table -5. Every result is the average of independent determinations.

Table 5: Applications

Sr. No.	Sample	Amount of Pb(II) predicted from **Standard method**	Amount of Pb(II) predicted from **Present method**
1	Solder Alloy	88.10%	88.06%
2	Galena	10.55%	10.45%
4	Pb(5) + Zn(5)	4.95ppm	4.90ppm
5	Pb(50) + Cd(50) + Ni(50)	50ppm	49.95ppm

RESULT AND DISCUSSION:

In this section, experimental results of solvent extraction for removal of Pb(II) by using HAPED as organic reagent are presented. The stability of Lead complex is 33h. Represented as in figure:3. It is observed from this figure that a linear calibration curve was obtained in the range of 1-10 ppm Lead. Effect of various parameters like pH, absorbance, wavelength and validity of Beer's and Lambert's law. The absorption is observed maximum at wavelength 535 nm. The equilibrium is attained within 1 min. The best results of solvent extraction were obtained in aqueous phase at pH 7.6 whereas organic phase containing Chloroform as solvent.

1. Effect of pH and absorbance

Chloroform is found to be the most suitable solvent which is carried maximum extraction which is shown in figure 5. 1 cm^3 aqueous solution contain 100 ppm Pb(II) at different pH shaking with 2 cm^3 of 0.1% HAPED in chloroform, after separated a two layers measure the absorbance of organic phase at wavelength of 535nm and pH of 5.2 respectively which is represented in figure: 6.

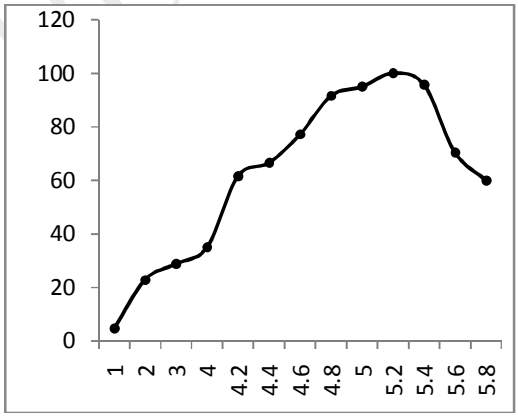


Figure :6- Effect of pH on the extraction of Pb(II):HAPED complex

2. Selection of The Solvent

Various solvents were tried to determine the maximum extraction of Chromium. Chloroform was found to be most suitable solvent as it showed the maximum extraction. The extraction of Chromium varied from maximum to minimum for the solvent in the order of chloroform>ethyl Acetate

> n-butanol>xylene>cyclohexanone> diethyl ether >toluene>carbon Tetrachloride > n-Hexane >nitrobenzene which is shown in figure:7.

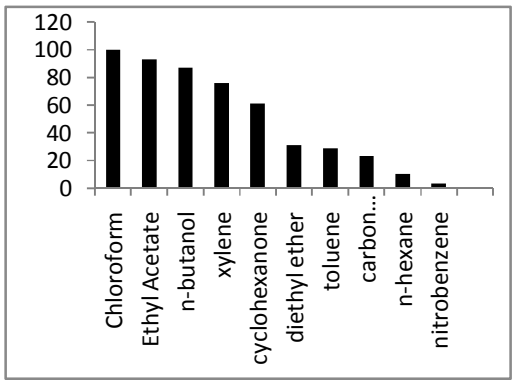


Figure :7 Effect of various solvents on Lead(II) : HAPED complex

3. Shaking time effect

1-cm^3 aqueous solution contain 100 ppm Pb(III) at pH 5.2 after added 2 cm^3 of 0.1%HAPED in chloroform, shaking for different times (0-60) min. after separating the layers, measuring the absorbance of organic phase at wavelength of 535nm.

4 Mole ratio method

Solution of 0.01M HAPED in chloroform used to extract 0.01M Pb(II) from aqueous solution at optimum conditions, also determine absorbance of organic phase at wavelength of 535nm against chloroform, figure : 3 indicates that the ratio of Pb(II) to complex was 1:2[Pb^{+2}:(HAPED)].

4. CONCLUSION

The proposed novel reagent is found to be more effective over reagents reported by earlier investigators. The proposed method is simple, more highly sensitive and selective than the reported methods for the extractive Spectrophotometric determination of microgram amounts of Lead. It has been successfully applied to the determination of Lead at trace level in synthetic mixtures and alloys . It offers advantages like reliability and reproducibility in addition to its simplicity, instant colour development and suffers from less interference.This method iseasily employed anywhere as does not require sophisticated instruments.

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