June 2010 ISSN : 0974-7419 Volume 9 Issue 2

Trade Science Inc.

Analytical CHEMISTRY
An Indian Journal

Full Paper

ACAIJ, 9(2) 2010 [222-224]

Spectrophotometric assay of lead in water samples

K.Saraswathi[1]*, Ch.Bhargavi[2]
[1]Department of Chemistry, S.V. University, Tirupati - 517 502, A.P., (INDIA)
[2]Department of Chemistry, Andhra Loyola College, Vijayawada - 520 008, A.P., (INDIA)
E-mail : ksaraswathi@gmail.com; bhargavichinta@gmail.com

Received: 23rd February, 2010 ; Accepted: 5th March, 2010

ABSTRACT

A simple and rapid spectrophotometric method is developed for the determination of lead as Pb-(M.D.T.C) complex in the presence of sodium acetate-acetic acid medium at pH = 4.4 and 0.2M $MgSO_4$ as salting out agent. As this complex is having absorption in U.V region, lead is estimated through copper as Cu-(M.D.T.C) complex. This complex shows absorption in visible region at 430nm. The method is sensitive up to 0.2ppm and it is free from interference of many metal ions like Al^{+3},Co^{+2},Zn^{+2},Ni^{+2},Fe^{+2}.Among various anions oxalate, Thiocyanate, iodide & chloride do not interfere up to 100 folds. The developed method is applied for the determination of Pb (II) in various water samples.

KEYWORDS

Spectrophotometry;
Morpholine dithio carbamate;
Complex;
Ligand;
Buffer;
Salting out agent.

INTRODUCTION

Lead is a toxic metal that enters body from lead water pipes, lead based paints and leaded petrol. Hence the determination of trace amount of lead is important in the context of environmental monitoring. Dithio carbamates are half amides of dithiocarbamic acid. They play a major role in analytical chemistry as reagents for metal ions due to their metal binding property & chelating tendency to give high sensitivity. Most of the heavy metal compounds of the dithio carbamates are coloured facilitating spectrophotometric methods in visible or near UV region.

Morpholine dithio carbamate (M.D.T.C) has advantage because of its ability to give stable complexes. M.D.T.C forms a complex with lead extractable in to chloroform shows absorbance in U.V. region. Hence lead is estimated through copper as Cu-(M.D.T.C) complex whose absorbance is with in visible region at 430nm (Figure 1). The quantitative experimental conditions have been developed & the details are shown in TABLE 1.

EXPERIMENTAL

Reagents

All chemicals used were of analytical grade.

Morpholine dithio carbamate

To 100ml of cold methanol, 8.2g w/v of morpholine and 8.0g w/v of pure CS_2 are added & stirred. The white ppt formed is redissolved in 15ml of 8N NaOH. On cooling crystals of M.D.T.C separated out. They are recrystallised by using benzene.

Preparation of Pb (II) solution

The Pb (II) solution is prepared in litre of double distilled water such that its concentration is 0.01M. This solution is further diluted to get 4.915×10^{-4}M concentration.

TABLE : 1 Quantitative experimental conditions

Conditions	Optimum values
pH	4.4
Sodium acetate, M	0.2
$MgSO_4$, M	0.2
Pb(II), ppm	0.2-10
Ligand, M	4.915×10^{-4}

TABLE : 2 Study of interfering ions

Foreign ion	Source of the ion	Tolerance limit, ppm
Al^{+3}	$Al_2(SO_4)_3(NH_4)_2SO_4.2H_2O$	640
Se^{+4}	Na_2SeO_3	640
Co^{+2}	$CoCl_2$	640
Zn^{+2}	$ZnSO_4.7H_2O$	320
Ni^{+2}	$(NH_4)_2SO_4NiSO_4.6H_2O$	320
Fe^{+2}	$Fe(NH_4)_2SO_4(SO_4)_2.6H_2O$	320
Cd^{+2}	$(CH_3COO)_2Cd.2H_2O$	160
Sn^{+2}	Sn(metal) In HCl	128
Te^{+2}	Na_2TeO_3	128
Cr^{+3}	$Cr_2(SO_4)_3K_2SO_4.24H_2O$	128
Mn^{+2}	$MnSO_4$.	128
Cr^{+6}	$K_2CrO_4H_2O$	64
U^{+6}	$UO_2(NO_3)_2.6H_2O$	64
Ce^{+4}	$(NH_4)_4Ce(SO_4)_4.2H_2O$	64
V^{+5}	NH_4VO_3	32
Mo^{+6}	$(NH_4)6Mo_7O_{24}$	6.4

TABLE : 3 Determination of Pb (II) in water samples of Vijayawada town of Andhra Pradesh, India

Sample	Lead ppm/ml	
	Added	found
Krishnalanka	6.11	6.21
Bhavanipuram	6.11	6.25
Chuttugunta	6.11	6.19
Bore-well water	6.11	7.02
Well water	6.11	7.10
Agricultural water	6.11	7.42

1.0ml of concentrated sample is used

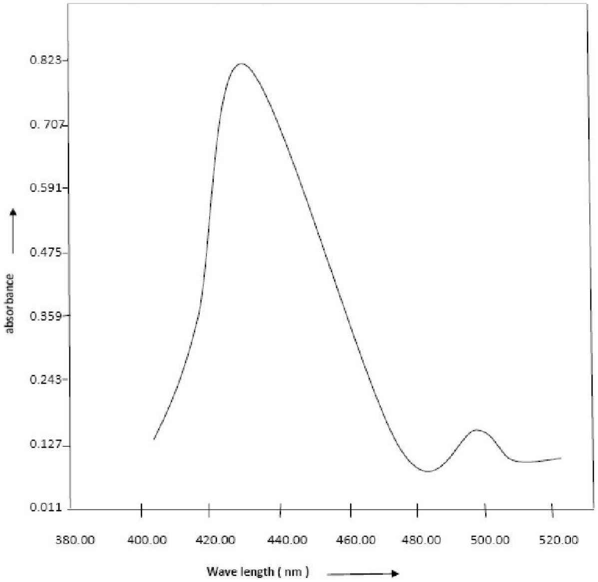

Figure 1 : Absorption spectrum for Lead through Cu (MDTC) Complex

Sodium acetate-acetic acid buffer

Equi molar solutions of sodium acetate and acetic acid are prepared separately & suitable ratios of their solutions are mixed to get desired pH up to 7.0.

Purification of solvents

All the solvents used have been distilled before use. These include chloroform, benzene, methyl alcohol.

Preparation of water samples

One litre of water sample is boiled & pre concentrated by evaporation to 100ml & filtered.

Apparatus

Elico make model BL 198 UV-visible recording Bio spectrophotometer is used for recording absorption spectra of the solutions. This operates over a wave length range of 190-1100nm. The PH measurements are made by using digital PH meter, model 335 systronics.

RESULTS AND DISCUSSION

Effect of PH

Concentration of metal ion 10.18 ppm, ligand 4.915 $\times 10^{-4}$M & sodium acetate-acetic acid 0.2 M are fixed and the pH effect is studied from PH 2 to 7. For pH 8 to 11 ammonium chloride-ammonium hydroxide buffer is used. 0.2M $MgSO_4$ is used as salting out agent. With increase in pH absorption also increases till pH 4.4 then on words decreases. So sodium acetate buffer of pH 4.4 is selected for the experimental study.

Effect of sodium acetate concentration

By keeping all the other factors constant including pH at 4.4, the concentration of buffer solution is varied from 0.1M to 2M & its effect was studied. It is observed at 3.0ml of 0.2M & above concentration of the buffer the maximum absorbance is stable. Since the complex formation & extraction are quantitative in pH range 4.4 of 3.0ml of 0.2M sodium acetate all the experiments are carried.

Effect of reagent concentration

By keeping the concentration of Pb (II), sodium acetate & pH at optimum values as mentioned above a wide range of ligand concentration are added to study the effect of reagent in order to obtain an optimum range of reagent concentration for complete precipitation of the metal ion.

Applicability of Beer's law

To the solution containing different amounts of lead, sodium acetate buffer, ligand (M.D.T.C), salting out agent are added and the contents are extracted with chloroform, organic phase is treated with Cu (II) solution. The absorption is measured at 430nm. Beer's law is applicable to the formed complex.

Study of interfering ions

The extent of interference due to metal ions usually associated with lead along with other cations and various anions are determined by measuring the absorbance of a solution containing 6.4ppm/ml of lead and various amounts of foreign ions. The following foreign ions have been found to give less than 2% error in absorbance value. Al^{+3}, Se^{+4}, Co^{+2}, Zn^{+2}, Ni^{+2}, Fe^{+2}, Cd^{+2}, Sn^{+2}, Te^{+2}, Cr^{+3}, Mn^{+2}, Cr^{+6}, U^{+6}, Ce^{+4}, V^{+5}. Among the various anions nitrate oxalate, thiocyanate, iodide, chloride do not interfere up to 100 folds. EDTA and periodate, citrate and carbonate also do not interfere. Anions iodate and nitrate found to interfere. Fe^{+3} is tolerated in the presence of oxalate and tartrate, and Ag^{+} in thiocyanate or iodide masking agents (TABLE 2).

Determination of formula and instability constant

The composition of the complex is determined by Job's method & Mole-ratio method. It is found that the metal to ligand ratio is 1:4, the structure of which will be confirmed as crystalline compound in later presentation. The instability constant for the complex formed is determined by Asmus method. It is found to be 1.942 $\times 10^{-23}$.

Applications

Determination of Pb (II) in water samples

The method adopted for the analysis of lead content in water samples is standard addition method.

The results indicate that the water samples of Vijayawada town of Andhra Pradesh contain levels of lead within the tolerance limits.

CONCLUSIONS

The developed method is simple and sensitive for trace concentration of lead. The technique showed the advantage of simplicity and low economical cost compared with other instrumental methods.

ACKNOWLEDGEMENTS

The authors are thankful to the management of Andhra Loyola college, Vijayawada for supporting this work by granting minor research project.

REFERENCES

[1] M.L.Nollet; Hand Book of 'Water Analysis', Marcel Dekker (Ed.), 44 **(2000)**.

[2] Hg.Seiler, A.Sigel, H.Sigel; Hand Book of 'Toxicity of Inorganic Compounds', Marcel Dekker New York, **(1998)**.

[3] E.Pichard, G.M.Mackay, J.Points; 'Trace Analysis; A Structure Approach to Obtaining Reliable Results', The Royal Society of Chemistry, U.K., 38 **(1996)**.

[4] R.Renner; Envorn.Sci.Technol., **29**, 256 **(1995)**.

[5] A.D.Kakal, F.E.Hahn, K.N.Raymond; J.Am.Chem. Soc., **112**, 1519 **(1990)**.