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Effect of Non-ionic Surfactant (Triton X-100) on the Spectrophotometric Determination of Lead(II) with Potassium Benzyl Xanthate

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The effect of non-ionic surfactant (Triton X-100) on the sensitive, simple and inexpensive spectrophotometric method was developed for the determination of lead(II) with potassium benzyl xanthate (KBX). Lead was complexed with potassium benzyl xanthate in presence of non-ionic surfactant (Triton X-100). Absorption spectrum of $Pb(KBX)_2$ complex in the presence of non-ionic surfactant medium shows a peak at 370 nm. The molar absorptivity of the complex was 6.723×10^4 dm^3 mol^{-1} cm^{-1} and Sandell's sensitivity was 3.2478×10^{-3} mg cm^{-2}. The system obeys Beer's law in the concentration range of 0.25 to 10.0 ppm. The composition of the complex (metal:ligand) was 1:2. The method was employed for the determination of lead in industrial effluents and ground water samples.

Key Words: Micellar, Spectrophotometric method, Lead, Potassium benzyl xanthate.

INTRODUCTION

Lead is a relatively abundant metal in nature, occurring in lead minerals. In the atmosphere it is relatively more abundant than other heavy metals. It is one of the metals extensively used in lead storage batteries and as tetra ethyl lead (TEL) in combustion fuels. The major biochemical effect of lead is its interference with hem synthesis, which leads to hematological damage. Lead inhibits several of the key enzymes involved in the overall process of hem synthesis, where by the metabolic intermediate accumulate. Lead inhibits the ACA-dehydrase enzyme[1]. So that it cannot proceed further to form (II) porphobilinogen. The overall affect is the disruption of the synthesis of hemoglobin well as other respiratory pigments, such as cytochromes, which requires hem. Finally lead does not permit utilization of oxygen and glucose for life-sustaining energy production. This interference can be detected at a lead level in the blood of about 0.3 ppm. At higher levels of lead in the blood there will

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be symptoms of anemia due to the deficiency of hemoglobin. Elevated lead levels in blood cause kidney, liver disfunctions. This results in either reduced functioning or complete break down of tissues and finally brain damage.

Literature reveals a number of methods[2-14] to determine lead spectrophotometrically. In the present investigation the author proposed a new spectrophotometric method to determine lead(II) with potassium benzyl xanthate (KBX) in presence of non-ionic micellar medium like Triton X-100.

EXPERIMENTAL

Shimadzu, PR1 UV-visible-recording spectrophotometer, UV-240 and Elico digital pH meter LI-120 were used in the present investigation. Potassium benzyl xanthate (KBX) was prepared by standard method[15]. All the reagents and neutral surfactant Triton X-100 are of analytical grade and all the solutions are prepared in double distilled water. 1.598 g of lead nitrate was dissolved in deionised double distilled water. 1 mL Concentrated nitric acid was added and the solution diluted with deionised double distilled water to 1000 mL. Buffer of different pH values were prepared by standard procedures. Hydrochloric acid-potassium chloride buffer (pH 2-4), acetic acid-sodium acetate buffer (pH 4-7) and ammonium chloride and ammonium hydroxide buffer (pH 8-12) were prepared. A 0.4 % of KBX solution was prepared by dissolving 0.2 g of KBX in 100 mL water.

General procedure: An aliquot of the solution contains 0.25 to 10.0 μg of lead(II) was taken in a 10 mL volumetric flask, followed by the addition of sodium acetate-acetic acid buffer solution of pH 6.0, 2.0 mL of 0.5 % KBX solution and 2.0 mL of 0.001 N Triton X-100 solutions. The total volume was diluted to 10 mL of double distilled water. The absorbance values of the $Pb(KBX)_2$ complex was measured at 370 nm against reagent blank after shaking for few minutes.

RESULTS AND DISCUSSION

Absorption spectrum of $Pb(KBX)_2$ complex in the presence of non-ionic surfactant shows a peak at 370 nm against water blank. It was shown in Fig. 1. In absence of Triton X-100, $Pb(KBX)_2$ has a maximum absorbance at 360 nm in methyl isobutyl ketone (MIBK), since complex was extracted into MIBK. In the absence of Triton X-100, the stability of complex is very low hence it was extracted into MIBK, where in the presence of Triton X-100 the complex was solubulized completely and the extraction step is avoided. Hence further studies were carried out at 370 nm in the presence of Triton X-100. (No change was observed when the mixture was shaken from 1.5 to 3.0 min. Hence 2 min of shaking time was enough for the complete formation of complex).

The behaviour of $Pb(KBX)_2$ complex was studied in the presence of Triton X-100 over a wide range of pH values. The pH studies were carried out varying the pH from 2.0 to 8.0 with 8.0 μg/mL of lead(II). It was observed that the $Pb(KBX)_2$ complex formation increases from pH 2.0 reaches a maximum intensity at pH 6.0

and decrease beyond 6.0. Hence, all further studies were carried out at 6.0 pH. The effect of reagent concentration was studied by measuring the absorbance of solutions containing 8.0 μg/mL metal ion and 0.5 to 3.0 mL of 0.5 % KBX. It was observed that 1.0 mL of 0.5 % of KBX has a maximum absorbance, which was selected and used for further studies. The effect of different Triton X-100 concentrations was studied by measuring absorbance of solutions containing 8.0 μg/mL of metal ion of pH 6.0. The results indicate that 2.0 mL of 0.001 N Triton X-100 give a maximum absorbance which was selected and used for further studies.

With the optimum conditions developed the calibration curve was constructed. It was observed that Beer's law was obeyed in the concentration range of 0.25 μg to 10.0 μg of lead(II) (Fig. 2). It was observed that in the pressure of Triton X-100 the sensitivity of the complex is increasing tremendously. Sandell's sensitivity of the reaction obtained from Beer's law is 3.2478×10^{-3} μg cm^{-2} and the molar absorptivity of the complex is calculated as 6.723×10^{4} dm^3 mol^{-1} cm^{-1}. Aliquots containing 8.0 μg/mL of lead(II) gave a standard deviation of 0.4407×10^{-3} and coefficient of variation is 0.1447 %.

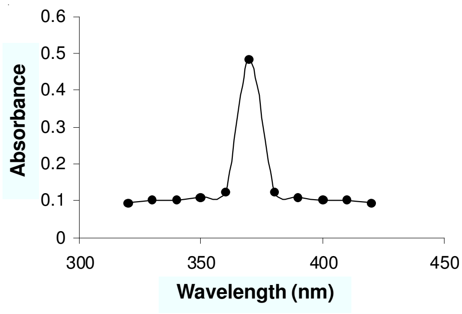

Fig. 1. Absorption spectra of $Pb(KBX)_2$ complex in presence of Triton X-100

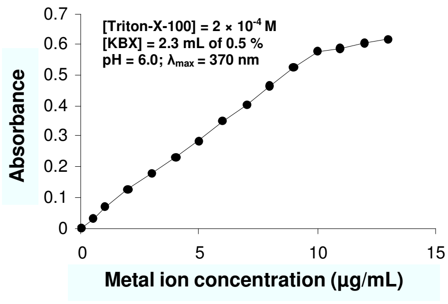

Fig. 2. Plot of $[Pb^{2+}]$ *vs.* absorbance

The composition of lead(II) complex was found to be 1:2 according to Job's method, Molar ratio method and Asma's method. The instability constant of the complex was found to be 2.051×10^{-8} by Edmond's and Birnbaum's method.

In order to assess the possible analytical applications of this method, the effect of diverse ions on spectrophotometric determination of lead(II) were studied in the presence of Triton X-100. A known amount of the ion in question was added to a solution containing 15 μg/mL of lead(II). The tolerance limit was set at the amount required, so as not to cause beyond ± 2 % error in lead recovery. The results are presented in Table-1.

The results revealed that, cations like Ba(II), Sr(II) and Mg(II) do not have any effect when present up to 5000 μg in the extraction of $Pb(KBX)_2$ complex. Zn(II), Sn(II) and Cd(II) can be tolerated up to 2000, 4000 and 1000 μg, respectively. Mn(II) was not interfere even when present up to 3000 μg. Fe(III), Cu(II) and Co(II)

TABLE-1
EFFECT OF FOREIGN IONS ON THE EXTRACTION OF $Pb(KBX)_2$ COMPLEX

Foreign ion	Sources of the ion	Tolerance limit (μg)
Ba(II)	$BaCl_2 \cdot 2H_2O$	5000
Sr(II)	$Sr(NO_3)_2$	5000
Cd(II)	$(CH_3COO)_2Cd \cdot H_2O$	1000
Zn(II)	$ZnSO_4 \cdot 7H_2O$	2000
Sn(II)	$SnCl_2$	4000
Mg(II)	$MgSO_4 \cdot 6H_2O$	5000
Mn(II)	$MnSO_4 \cdot H_2O$	5000
Fe(III)*	$FeCl_3$	-
Cu(II)*	$CuCl_2$	-
Co(II)*	$CoCl_2 \cdot 6H_2O$	-
Fluoride	NaF	5000
Chloride	KCl	5000
Acetate	CH_3COONH_4	3500
Tartrate	COOK, CHOH, CHOH, $COONa \cdot 4H_2O$	3500
Sulphate	Na_2SO_4	2000
Thiocyanate	NH_4SCN	1000

*Masked by using 1.0 mL of 0.4 % citrate solution; [Triton-X-100] ÷ 2.0 mL of 0.001 N, $[Pb^{2+}]$- 8.0 μg/mL, [KBX]- 2.0 mL of 0.4 %; pH = 6.0, λ_{max} = 370 nm

interfere severely with the determination of Pb(II). The interference due to Fe(III), Cu(II) and Co(II) can be suppressed by using 1.0 mL of 0.4 % citrate solution.

Anions like fluoride and chloride up to 5000 μg, acetate and tartrate up to 3500 μg, sulphate up to 2000 μg and thiocyanate up to 1000 μg do not interfere in the determination of Pb(II).

Application of the developed method: The developed spectrophotometric method for lead(II) in presence of non-ionic micellar medium was applied successfully for its determination of industrial effluents and ground water samples.

Determination of lead(II) in industrial effluents: The proposed method was applied for the determination of lead(II) in industrial effluents of Hindustan Zinc Limited, Visakhapatnam, India. The pretreatment of the industrial effluents were carried out according to the standard procedure. After pretreatment 200 mL of the sample was evaporated to 30 mL and then diluted to 50 mL with double distilled water. Form this aliquots of the sample were taken for the analysis and the results of investigation are presented in Table-2 of the six samples analyzed. All the samples containing lead(II) are found in the concentration range of 3.70 to 7.01 ppm. All these values are good agreement with dithiazone method.

Determination of lead(II) in ground water samples of Chirala Town, Prakasam District: Chirala is a major industrial town in Prakasam district, India and large numbers of textile processing units are present in the town. The data pertaining to the analysis of ground water sample of Chirala town present in Table-3, which shows that the results obtained by the present method and the results obtained

TABLE-2
LEAD(II) LEVELS FOUND IN INDUSTRIAL EFFLUENTS OF
HINDUSTAN ZINC LIMITED, VISAKHAPATNAM, ANDHRA PRADESH, INDIA

Sampling station	Lead found (ppm)		Recovery (%)
	Dithiazone method	Present method*	
Sample-1	5.68	5.60	98.6
Sample-2	4.32	4.29	99.3
Sample-3	3.93	3.82	97.2
Sample-4	6.27	6.19	98.7
Sample-5	7.10	7.06	99.4
Sample-6	5.08	5.03	99.0

*Average value of three determinations; [KBX]- 2.0 mL of 0.4 %, pH- 6.0, Shaking time- 3.0 min; λ_{max} – 370 nm, Triton-X–100- 2.0 mL of 0.001 N

TABLE-3
LEAD(II) LEVELS IN GROUND WATER SAMPLES OF
CHIRALA TOWN, PRAKASAM DISTRICT, ANDHRA PRADESH, INDIA

Sampling station	Lead found (μg/mL)		Recovery (%)
	Thiocyanate method	Present method*	
Ujilipet	ND	ND	–
Paparaju thota	1.10	1.09	99.0
Anandapet	4.75	4.70	98.9
Perala Center	ND	ND	–
Kotta Pet	ND	ND	–
ILTD Company	8.14	8.09	99.3
Wood Nagar	ND	ND	–
Bodi Palem	3.44	8.39	98.5
Mutyala Pet	ND	ND	–
Veera Ragahava Pet	4.15	4.10	98.8
NTR Nagar	ND	ND	–
Church Bazar	2.70	2.68	99.2
Srinivasa Nagar	ND	ND	–

*Average value of three determinations; [KBX]- 2.0 mL of 0.4 %, pH- 6.0, Shaking time- 3.0 min, λ_{max} – 370 nm, Triton-X–100- 2.0 mL of 0.001 N

by the standard method are in good agreement. Of the 13 water samples analyzed, only 6 samples contained lead(II). The maximum metal content analyzed was 8.02 ng/mL of lead(II). The metal ion concentrations determined were below the permissible levels given by WHO.

REFERENCES

1. G.L. McIntire, *Crit. Rev. Anal. Chem.*, **21**, 257 (1990).
2. Magda Ali Akl, *Anal. Sci.*, **22**, 1227 (2006).
3. M.O. Luconi, R.A. Olsina, L.P. Fernandez and M.F. Silva, *J. Hazard. Mater.*, **128**, 240 (2006).
4. N. Rajesh and S. Manikandan, *Spectrochim. Acta*, **70A**, 754 (2007).
5. B. Jankiewicz, B. Ptaszyñski and M. Wieczorek, *Polish J. Environ. Stud.*, **10**, 123 (2001).

6. R.B.R. Mesquita, S.M.V. Fernandes and A.O.S.S. Rangel, *Talanta*, **62**, 395 (2004).
7. A.A. Ramirez, D. Gazquez, M. De la Rosa and F. Moreno, *Anal. Lett.*, **27**, 1595 (1994).
8. G. Fang, S. Meng, G. Zhang and J. Pan, *Talanta*, **54**, 585 (2001).
9. S.B. Savvin, T.V. Petrova, T.G. Dzherayan and M.M. Reichstat, *Fresenins J. Anal. Chem.*, **340**, 217 (1991).
10. A. Niazi, T. Momeni-Isfahani and Z. Ahmari, *J. Hazard. Mater.*, **165**, 1200 (2009).
11. W. Liu, W.-J. Zhao, J.-B. Chen and M.-M. Yang, *Anal. Chim. Acta*, **605**, 41 (2007).
12. A. Santalad, S. Srijaranai, R. Burakham, T. Sakai and R.L. Deming, *Microchem. J.*, **90**, 50 (2008).
13. Z. Wang, J.-H. Xu, W. Zhang, B. Zhuang and H. Qi, *Colloids Surfaces B*, **61**, 118 (2008).
14. H. Akbas and Ç. Batigoç, *Fluid Phase Equilib.*, **279**, 115 (2009).
15. A.I. Vogel, A Text Book of Practical Organic Chemistry, Longmann, London, edn. 3 (1968 and 1969).

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