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Effect of nonionic surfactant micellar (Triton X-100) on the spectrophotometric determination of Lead (II) with isonitriso-*p*-isopropyl acetophenone phenyl hydrazone

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ABSTRACT

The effect of nonionic surfactant micellar (Triton X-100) on the sensitive, simple and inexpensive, spectrophotometric method was developed for the determination of lead (II) with isonitriso *p*-isopropyl acetophenone phenyl hydrazone (HIPAPH). Lead was complexed with isonitriso *p*-isopropyl acetophenone phenyl hydrazone (HIPAPH) in presence of non ionic surfactant micellar (Triton X-100). Absorption spectrum of Pb- HIPAPH complex in the presence of non ionic surfactant¹ medium shows a peak at 390 nm. The molar absorptivity of the complex was $5.6734 \times 10^4 \text{ dm}^3 \cdot \text{mole}^{-1} \cdot \text{cm}^{-1}$ and Sandell's sensitivity was $2.2367 \times 10^{-3} \text{ mg} \cdot \text{cm}^{-2}$. The system obeys Beer's law in the concentration range of 0.25 to 15.0ppm. 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, isonitriso *p*-isopropyl Acetophenone Phenyl Hydrazone.

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 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 (I). So that it cannot proceed further to form (II) porphobilinogen. The over all 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 0.3ppm. At higher levels of lead in the blood there will 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 ²⁻¹⁵ 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 SECTION

Schimadzu, PR1 UV-visible – recording spectrophotometer, UV-240 and Elico digital pH meter LI-120 were used in the present investigation.. All the reagents and neutral surfactant Triton X-100 are of analytical grade and all the solutions are prepared in double distilled water. 1.598g of lead nitrate was dissolved in deionized double distilled water. 1mL concentrated nitric acid was added and the solution diluted with deionized double distilled water to 1000mL. Buffer of different pH values were prepared by standard procedures. hydrochloric acid – potassium chloride buffer (pH 2.0 – 4.0), acetic acid – sodium acetate buffer (pH 4.0 – 7.0) and ammonium chloride and ammonium hydroxide buffer (pH 8.0 – 12.0) were prepared. Isonitroso p-isopropyl acetophenone Phenyl Hydrazone was prepared by refluxing equimolar amount of isonitroso p-isopropyl acetophenone with phenyl hydrazine for 4 hours. On cooling the reaction mixture, a yellow colored product separated out which was collected by filtration and washed with ethanol. The resulting HIPAPH was recrystallized using aqueous ethanol as the procedure recommended by Vogel ¹⁶. 0.2% solution of HIPAPH prepared in DMF.

General Procedure

An aliquot of the solution contains 0.25 to 10.0 μ g of lead (II) was taken in a 10.0mL volumetric flask, followed by the addition of sodium acetate – acetic acid buffer solution of pH 6.0, 1.0mL of 0.2% HIPAPH solution and 2.0mL of 0.001N Triton X – 100 solutions. The total volume was diluted to 10.0mL of double distilled water. The absorbance values of the Pb (II) - HIPAPH complex was measured at 390 nm against reagent blank after shaking for few minutes.

RESULTS AND DISCUSSION

Absorption spectrum of Pb-(HIPAPH)₂ complex in the presence of non-ionic surfactant shows a peak at 390nm against water blank. It was shown in Fig.1. In absence of Triton X-100, Pb-(HIPAPH)₂ has a maximum absorbance at 360nm in 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 solubilized

completely and the extraction step is avoided. Hence further studies were carried out at 390nm in the presence of Triton X-100. (No change was observed when the mixture was shaken from 1.5 to 3.0 minutes. Hence 2 minutes of shaking time was enough for the complete formation of complex).

The behavior of Pb-(HIPAPH)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-(HIPAPH)₂ complex formation is 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. It was observed that a ten fold excess of reagent was sufficient to produce maximum intensity of colour.

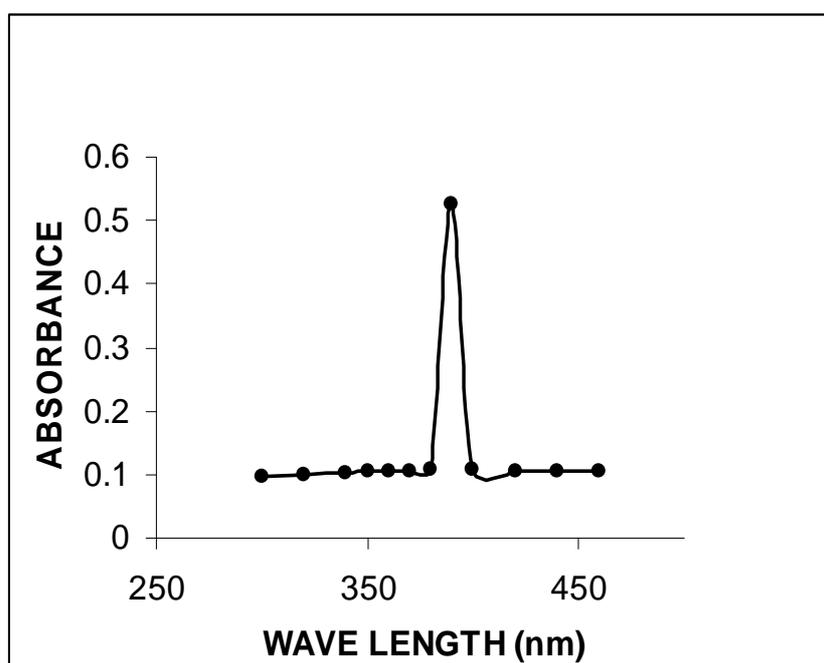
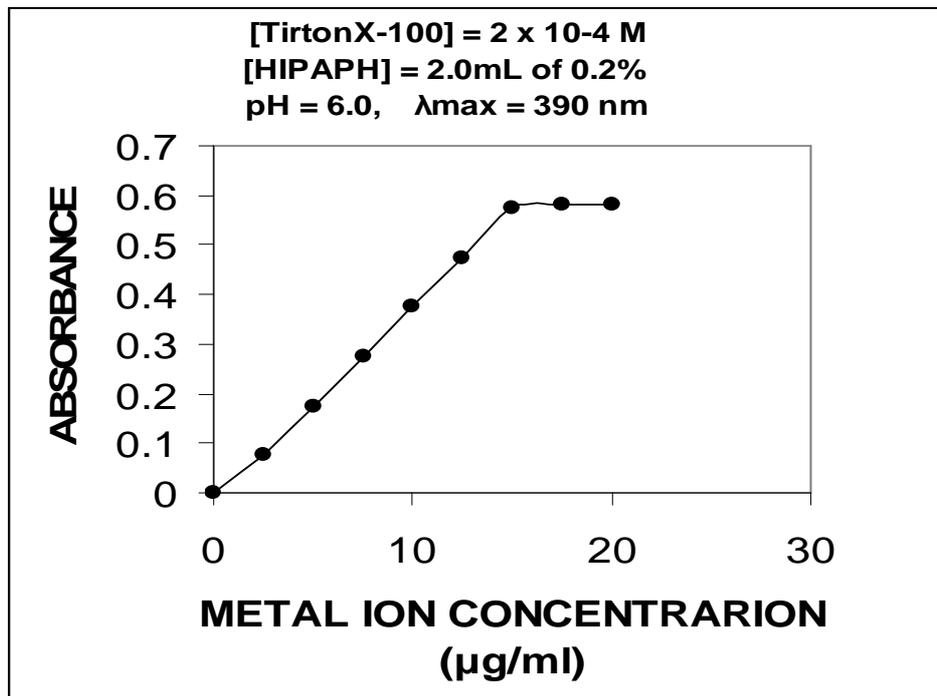


FIG: 1 Absorption spectra of Pb (HIPAPH)₂ complex in presence of Triton X-100

This is equal to 1.0mL of 0.2% HIPAPH solution. 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 2.0mL of 0.001N 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 15.0 µg of lead (II) (Fig-2). It was observed that in the presence of Triton X-100 the sensitivity of the complex is increasing tremendously. Sandell's sensitivity of the reaction obtained from Beer's law is $2.2367 \times 10^{-3} \text{ mg.cm}^{-2}$ and the molar absorptivity of the complex is calculated as $5.6734 \times 10^4 \text{ dm}^3.\text{mole}^{-1}.\text{cm}^{-1}$. Aliquots containing 8.0 µg/mL of lead (II) gave a standard deviation of 0.4518×10^{-3} and coefficient of variation is 0.1336 per cent.

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 2.11×10^{-8} by Edmond's and Birnbaum's method.

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 .2H ₂ O	5000
Sr (II)	Sr (NO ₃) ₂	5000
Cd (II)	(CH ₃ COO) ₂ Cd.H ₂ O	1000
Zn (II)	ZnSO ₄ .7H ₂ O	2000
Sn (II)	SnCl ₂	4000
Mg (II)	MgSO ₄ .6H ₂ O	5000
Mn(II)	MnSO ₄ .H ₂ O	5000
Fe (III)*	FeCl ₃	-
Cu (II)*	CuCl ₂	-
Co (II)*	CoCl ₂ .6H ₂ O	-
Fluoride	NaF	5000
Chloride	KCl	5000
Acetate	CH ₃ COONH ₄	3500
Tartrate	COOK. CHOH. CHOH. COONa.4H ₂ O	3500
Sulphate	Na ₂ SO ₄	2000
Thiocyanate	NH ₄ SCN	1000

* Masked by using 1.0mL of 0.4% citrate solution
 [Triton X-100] ÷ 2.0mL of 0.001N, $[Pb^{+2}]$ - 15.0 µg/mL, [HIPAPH]- 2.0mL of 0.2%,
 pH - 6.0, λ_{max} - 390nm

In order to assess the possible analytic 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 percent 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 5000µg in the extraction of Pb-(KBX)₂ complex. Zn (II), Sn (II) and Cd (II) can be tolerated up to 2000 µg 4000 µg and 1000 µg respectively. Mn (II) was not interfere even when present up to 3000µg. Fe (III), Cu (II) and Co (II) 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 per cent citrate solution.

Anions like Fluoride and Chloride up to 5000µg, acetate and tartrate up to 3500 µg, sulphates 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 of Hindustan Zinc Limited, Visakhapatnam

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. From 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) in concentration range 3.70ppm to 7.01ppm. All these values are in good agreement with Dithiozone method.

Table – 2 Lead (II) levels found in Industrial Effluents of Hindustan Zinc Limited, Visakhapatnam, Andhra Pradesh, India.

S.No	Sampling station	Lead found (ppm)		Recovery %
		Dithiozone method	Present Method*	
1	Sample – 1	5.68	5.60	98.6
2	Sample – 2	4.32	4.29	99.3
3	Sample – 3	3.93	3.82	97.2
4	Sample – 4	6.27	6.19	98.7
5	Sample – 5	7.10	7.06	99.4
6	Sample – 6	5.08	5.03	99.0

* Average value of three determinations

[HIPAPH]- 2.0mL of 0.2%, pH- 6.0, Shaking time- 3.0 minutes,

λ_{max} - 370nm, Triton X-100- 2.0mL of 0.001N

Determination of Lead (II) in Ground Water Samples of Chirala Town, Prakasam District, Andhra Pradesh

Chirala is a major industrial town in Prakasam district, Andhra Pradesh, 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 by the standard method are in good agreement. Of the thirteen water samples analyzed, only six 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.

Table – 3 Lead (II) Levels in Ground Water Samples of Chirala Town, Prakasam District, Andhra Pradesh, India.

S.No	Sampling station	Lead found (µg/mL)		Recovery %
		By thiocyanate method	By Present Method*	
1	Ujilipet	ND	ND	--
2	Paparaju thota	1.10	1.09	99.0
3	Anandapet	4.75	4.70	98.9
4	Perala Center	ND	ND	--
5	Kotta Pet	ND	ND	--
6	ILTD Company	8.14	8.09	99.3
7	Wood Nagar	ND	ND	--
8	Bodi Palem	3.44	8.39	98.5
9	Mutyala Pet	ND	ND	--
10	Veera Ragahava Pet	4.15	4.10	98.8
11	NTR Nagar	ND	ND	--
12	Church Bazar	2.70	2.68	99.2
13	Srinivasa Nagar	ND	ND	--

* Average value of three determinations

[HIPAPH]- 2.0mL of 0.2%, pH- 6.0, Shaking time- 3.0 minutes,

λ_{max} - 370nm, Triton X-100- 2.0mL of 0.001N

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