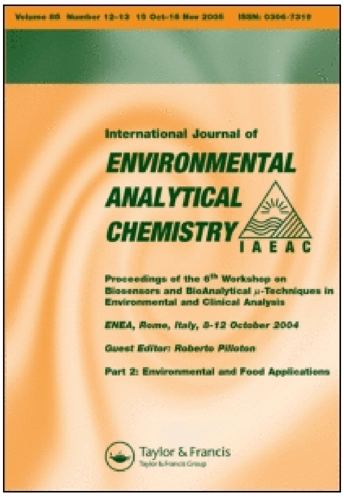

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Extraction and Spectrophotometric Determination of Lead in the Environment

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A rapid method for the extraction and spectrophotometric determination of lead in the environment is described. The lead forms a yellow complex with PBHA at pH 9.5 which is extracted from chloroform. The molar absorptivity was found $4.0 \times 10^2 \, l \, mol^{-1} \, cm^{-1}$ at 390 nm. The phenylfluorone is used as a synergestic. Pb-PBHA complex is back extracted with 0.01 M acetic acid and after raising to pH 9.5 lead is estimated by means of phenylfluorone which gives a yellow-red coloured complex. The molar absorptivity is found to be $1.9 \times 10^3 \, l \, mol^{-1} \, cm^{-1}$ at 510 nm. The effect of pH, electrolytes, reagent concentration and various ions is discussed. The method is applied for the trace determination of lead in industrial effluents and natural resources.

INTRODUCTION

Lead is used as an industrial raw material for the storage battery manufacture, printing, pigments, fuels and photographic materials. The effluents of these industries can contain lead concentrations in the range of 0.5 to 200 ppm.[1] The maximum permissible allowance as recommended by World Health Organization in the water is 100 $\mu g \, dm^{-3}$ and in the food 2 ppm.[2] Lead is a toxic metal and higher concentration results in damage of liver and blood coagulation[3] of vertebrates particularly in children. Various reagents viz dithizone, diethyldithiocarbamate etc. were used for the extraction and spectrophotometric determination of lead (4–7) but common metals Cu, Fe, Ca, Mg etc. interfere. The hydroxamic acids are the potential analytical reagents and are used for the spectrophotometric determination of several metal ions.[8,9] The N-phenylbenzohydroxamic acid (PBHA) has been used for gravimetric determination of lead.[10] In the present investigation the PBHA has been used for the extraction and spectrophotometric determination of lead in presence of phenylfluorone. The lead can be estimated in presence of common metals without any difficulty.

EXPERIMENTAL

Apparatus

The spectral measurements were made on VSU2-P (C. Z. Jena) spectrophotometer. The pH adjustment were made with a Systronics digital pH meter equipped with a glass and a calomel electrode.

Reagents and Chemicals:-

All the chemicals used were of AnalaR or GR grades of B.D.H. and E. Merck respectively unless otherwise specified.

The N-phenylbenzohydroxamic acid (PBHA) was synthesised as described elsewhere.[11] Its purity was checked by TLC, IR and UV spectra. A 0.05 M solution of the PBHA was prepared in chloroform.

0.03% of phenylfluorone solution was prepared by dissolving 30 mg of phenylfluorone in 100 ml of acetone.

A standard lead solution was prepared by dissolving a known amount of lead acetate in acetic acid and distilled water. Its final concentration was determined volumetrically.[12]

Procedure:-

Take 2 ml stock lead solution (1.01×10^{-2} M) in 100 ml separatory funnel and adjust the pH about 9.5 with buffer solution. Then add 10 ml of 0.05 M reagent solution. Allow the phase to separate. Extract the chloroform layer of yellow complex formed and dry over anhydrous sodium sulphate. Wash Na_2SO_4 twice with 2 ml of chloroform. Back extract the lead from the organic layer with 20 ml of 0.01 M acetic acid. To the aqueous layer add 1 ml of 0.03% phenylfluorone and dilute to 25 ml with distilled water. Measure the absorbance at 510 nm against reagent as blank.

Calibration curve

The molar absorptivity of lead phenylfluorone complex is $1.90 \times 10^{+3}$ $l\,mol^{-1}\,cm^{-1}$. The calibration curve is plotted by taking lead concentrations between 0.5 and 100 ppm.

Determination of lead in industrial effluents

Into a 250 ml separatory funnel 100 ml of industrial waste was taken and pH 9.5 was adjusted with buffer solution. 10 ml 0.05 M reagent solution was added and the contents were shaken for 10 min. The lead was extracted and the Pb-PBHA complex was back extracted as above. The absorbance of phenylfluorone complex was measured at 510 nm. The concentration of lead was calculated from the calibration curve.

Determination of lead from plants and soil samples

The plants were dried in oven and then heated to ash in silica crucible. Depending upon the concentration of lead in plants or soil samples, 15 to 20 g plants or soil were digested with excess of hydrochloric acid and nitric acid. The filtrate was evaporated and diluted to 100 ml. The lead concentration was determined as above and calculated from the calibration curve.

RESULTS AND DISCUSSION

The yellow coloured complex of Pb-PBHA gives no maxima (λ max) but measurements are reproducible at 390 nm, hence all the measurements are made at this wavelength. The reagent has no absorbance at this wavelength.

The yellowish red coloured lead phenylfluorone complex has a maximum absorbance at 510 nm. The characteristic of coloured complex of lead phenylfluorone system is given in Table I.

The molar absorptivity for lead PBHA complex is $4.0 \times 10^2 \, l \, mol^{-1} \, cm^{-1}$. The maximum colour intensity of the complex was found at pH 9.5.

Effect of solvents

The Pb-PBHA complex was extracted with various solvents viz. chloroform carbon tetrachloride, isobutyl methyl ketone, isoamyl alcohol etc. It was found that chloroform is the most suitable solvent for extraction of lead. These data are given in Table II.

Effect of reagent concentration

Divalent lead was extracted at pH 9.5 with varying volumes and concentrations of the reagent. Results indicate that single extraction with 10 ml of 0.05 M reagent was adequate for quantitative extraction of lead while extraction was incomplete with 0.005 M 0.001 and 0.03 M reagent solution. Large excess of reagent could be used without any difficulty.

TABLE I
Spectral characteristic of lead-phenylfluorone complex system in water.
Colour of the complex: Yellowish-red
Wavelength : 510 nm.

Lead Conc. ppm	pH	Absorbance	Molar absorptivity
20	4.0	0.090	0.9×10^3
20	6.0	0.155	1.6×10^3
20	8.0	0.175	1.8×10^3
20	9.0	0.178	1.8×10^3
20	9.2	0.181	1.9×10^3
20	9.5	0.183	1.9×10^3
20	9.6	0.183	1.9×10^3
20	9.8	0.180	1.8×10^3
20	10.0	0.095	1.0×10^3
20	9.5	0.183	1.9×10^3
40	9.5	0.366	1.9×10^3
60	9.5	0.550	1.9×10^3
80	9.5	0.732	1.9×10^3

TABLE II
Effect of Solvents on the Extraction of Lead phenylfluorone System.

Lead conc = 20 ppm λ_{max} = 510 nm
pH = 9.5 Colour = Yellow

Solvents	%E	Molar absorptivity
Chloroform	100.0	1.9×10^3
Carbon Tetrachloride	84.2	1.6×10^3
Isobutylmethyl ketone	90.7	1.7×10^3
Iso Amyl Alcohol	85.2	1.6×10^3

Effect of various ions

20 ppm of lead is extracted in presence of 50 mg of each Ba^{2+}, Sr^{2+}, Ca^{2+}, Co^{2+}, Ni^{2+}, Bi^{3+}, Be^{2+}, Mg^{2+}, V^{5+}, Mo^{6+}, W^{6+}, Nb^{5+}, Ta^{5+}, Pd^{2+}, Ti^{4+}, Zr^{4+}, and Hf^{4+}, but Cu^{2+}, Fe^{3+} and Ce^{4+} are interfere with the determination of lead. However, the interference of Cu^{2+} and Fe^{3+} could be eliminated by masking them with ascorbic acid.

Determination of lead in standard sample and in the environment

The data on the determination of the concentration of lead in the standard samples, plants, and industrial effluents are given in Table III. These data were also compared with AAS and a good agreement was found.

TABLE III

Concentration of lead in industrial effluents, plants and soils

Sample	Lead Found* (ppm)		Standard deviation
	Present method	ASS	
Soil sample	5–600	5–610	±0.2
Plants	5–100	4.5–110	±0.2
Battery Manufacture	5–50	4.8–48	±0.1
Television Tube Manufacture	200–400	200–400	±1.0
Mining Drainage	0.02 to 2.5	0.01–3.0	±0.01
Tetra Ethyl Lead Manufacture	45	45±48	±0.2

*Average of 20 determinations.

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