SPECTROPHOTOMETRIC DETERMINATION OF Pb(II) USING DMHBIH

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ABSTRACT:

Spectrometric methods are not documented in literature for the measurement of metal ions. Derivatives spectrometric are also a great background reduction method that allows accurate calculation and selectivity. Spectrophotometry derivatives may be a very helpful way of solving complex analytical issues. This study evaluated the spectrophotometric determination of Pb(II) using DMHBIH.

Keywords: Spectrophotometry, Pb (II), DMHBIH

INTRODUCTION:

Although many techniques of spectrophotometry are necessary to get efficient and acceptable results, precise processes are needed. Since the spectrophotometric colour development involves several types of reactions, numerous criteria have to be verified before beginning the process [1-3].

METHODS, RESULTS AND DISCUSSION [4-10]:

A Triton X-100 metal complex was surfaced by the 3,5 dimethoxy-4-hydroxy (DMHBIH) benzaldehyde isonicotine hydrazone (DMHBIH). The surfactant Lead (II) in pH 9.0 was replaced. The metal complex produced was studied and a null-order spectrophotometric method was developed for the assessment of lead (II) in aqueous environments. This wavelength was further chosen for the tests with a maximal radiation concentration of 430 nm, the Pb (II) -DMHBIH Complex.

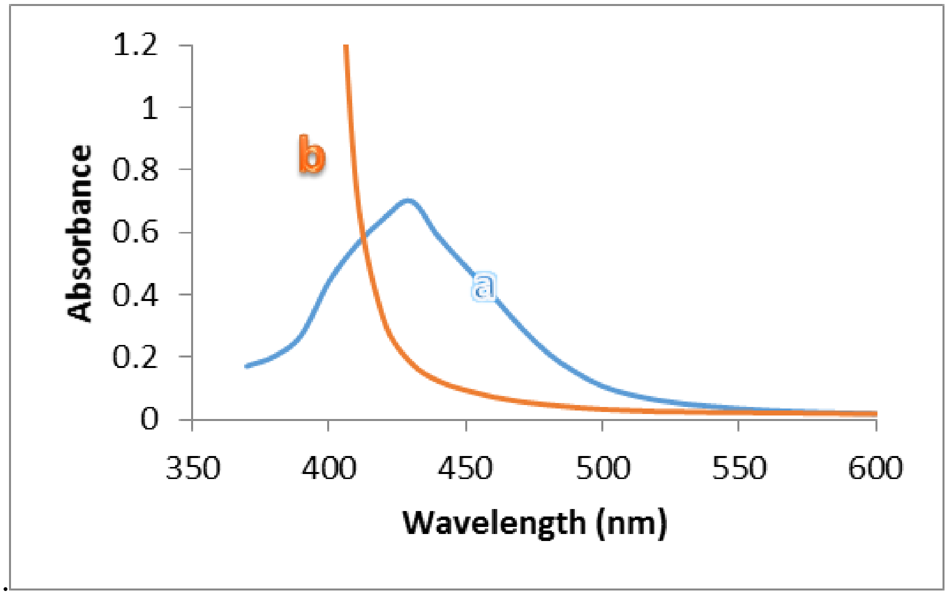

Figure 1. Absorption Maxima of Pb (II)- DMHBIH System Absorption spectra

a) [Lead(II)- DMHBIH] complex vs reagent blank

b) DMHBIH vs buffer blank

[Pb(II)] = 2.5×10^{-5}M

[DMHBIH] = 2.5×10^{-4} M

pH = 9.0

Triton X-100(5%) = 0.5 ml

In the case of a buffer solution (Phosphate Buffer) pH 9.0 the results of the pH, reagent and superfactant research (Triton X-100) indicate a high and consistent intake. The findings shown in Figure. The experimental findings of the reagent effect in Table suggest that a 10 times more complicated reagent is adequate. In presence of Triton X-100 (5 percent) Solution, stability and solubility of the complex were observed. Table of experimental values Table.

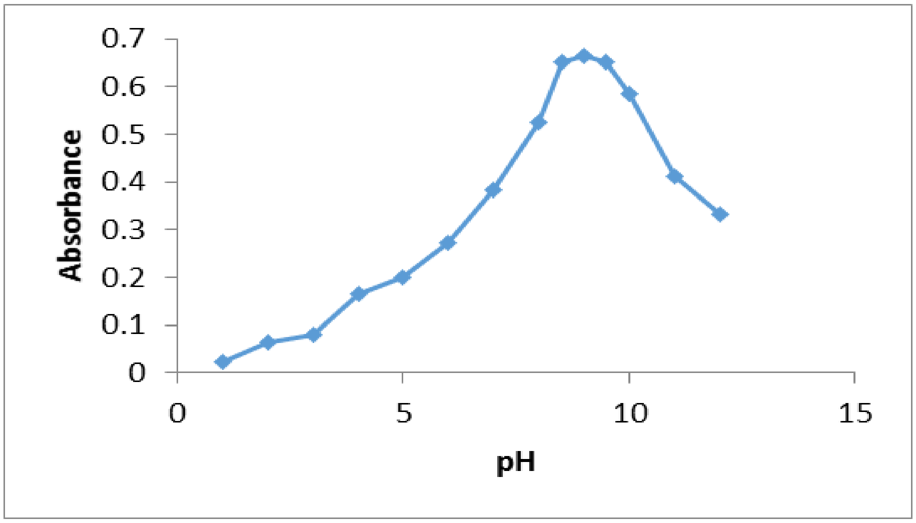

Figure 2. Effect of p^H on absorbance of Pb (II) –DMHBIH Complex

[Pb(II)] = 2.5×10^{-5} M

[DMHBIH] = 2.5×10^{-4} M

Wavelength = 430 nm

Table 1: Study of DMHBIH on the absorbance of metal complex

Pb(II) : DMHBIH	**Absorbance**
1:5	0.433
1:10	0.436
1:15	0.439
1:20	0.436
1:25	0.433
1:30	0.434
1:40	0.429

[Pb(II)] = 5×10^{-5} M

pH = 9.0

Wavelength = 389 nm

Table 2 : Effect of Triton X-100 on the absorbance of Lead (II) – DMHBIH complex

Triton X-100 in ml	Absorbance
0.5	0.55
1.0	0.547
1.5	0.540
2.0	0.543
2.5	0.538
3.0	0.535
4.0	0.534

[Pb(II)] = 5 x 10^{-5} M

[DMHBIH] = 5 x 10^{-4} M

pH = 9.0

Wavelength = 430 nm

Triton X-100(5%) = 0.5ml

The stability of complex with respect to time was studied in the presence of Triton X-100(5%) solution at various time intervals and the complex was found to be stable for six hours. The results were presented in Table.

Table 3: Time effect on stability of Complex

Time (In Minutes)	Absorbance
0	0.550
30	0.551
60	0.549
90	0.547
120	0.548
150	0.545
180	0.548
210	0.546
240	0.549
270	0.550
300	0.551

330	0.550
360	0.548
390	0.541

Studies also revealed that a change in the addition order of buffer, metal and surfactantdoes not show any adverse effect on the optical density of reaction mixture.

Applicability of Beer's Law:

The system was developed to control the law of Beer and it was found to comply with the law of Beer between 0.414 and 10.360µg/mL. Concentrations of 0.83 to 9.32 µg/mL were optimum. Sandell has 1.82 x 10^4 L. mol-1,cm^{-1} and 0.01302 A. cm^{-2} molar removal coefficient and Sandell sensitivity. Theplot was depicted in the Figure.

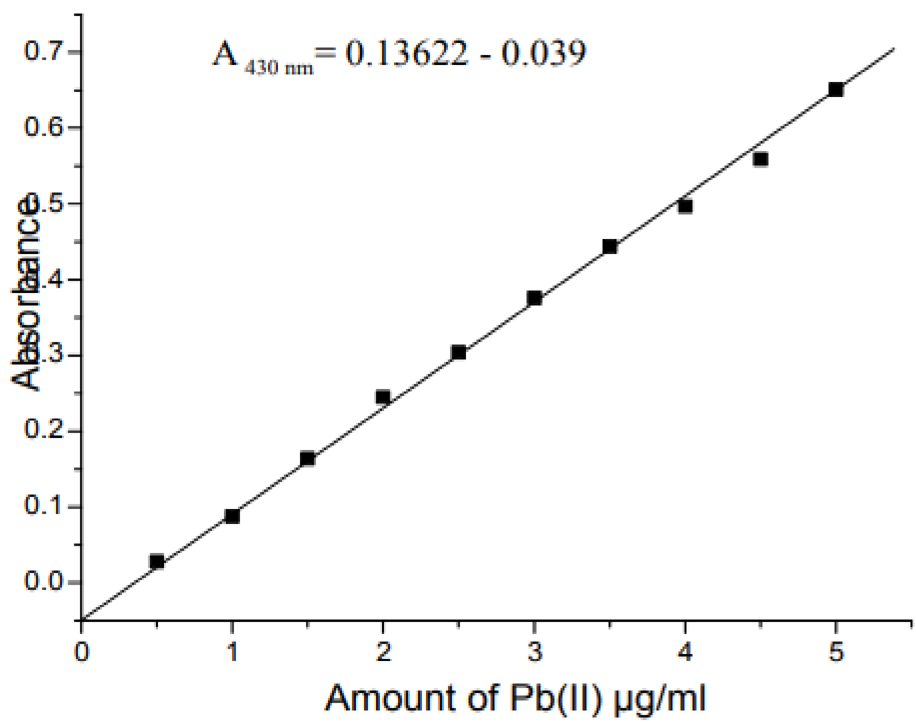

Figure 3: Linearity Plot of Pb (II) –DMHBIH Complex

[DMHBIH] = 2.5 x 10^{-4} M

pH = 9.0

Wavelength = 430 nm

Triton X-100 (5%) = 0.5 ml

Effect of foreign ions

Foreign ions interference was studied with 5.18µg/ml of Lead (II) and it was found that many anions and cations do not interfere in the determination of Pb(II) using DMHBIH reagent. The values are tabulated in Table.

Table 4: Tolerance limit of foreign ions in the estimation of 5.18 µg/ml of Pb(II)

Ion added	Tolerance limit µg/Ml	Ion added	Tolerance limit µg/mL
Bromide	3196.16	Hg^{2+}	100.3
Iodide	2538	Co^{2+}	88.4
Urea	1500	Zn^{2+}	65.39
Chloride	1064	Ca^{2+}	60.15
Tetraborate	970	Sn^{2+}	59.35
Sulphate	940	Zr^{4+}	45.61
Oxalate	880	Ni^{2+}	29.34
Nitrate	620	U^{6+}	12.0
Acetate	590	Ag^{+}	11.0
Thiocyanide	581	Mo^{6+}	9.59
Phosphate	475	As^{3+}	7.5
Ascorbic acid	176.13	Sb^{3+}	6.08
Tartarate	148	Pd^{2+}	5.32
Thiourea	115	Ru^{3+}	5.05
Fluoride	95	Al^{3+}	2.69
Ba^{2+}	206	Cr^{3+}	2.59
W^{6+}	184	V^{5+}	2.54
Sr^{2+}	175.2	Cu^{2+}	0.63 1.27†
Mn^{2+}	110	Fe^{3+}	2.8,3.8*
Bi^{3+}	104.5		

* Masked with 5.0 µg/ml of Phosphate

†Masked with 762 µg/ml of Thiourea

Applications

The Pb(II) was estimated in Bulked Food & Soil samples[245] by the method developed and the results of the present method was compared with certified results and incorporated in Tables.

Table 5: Estimation of Pb(II) in Bulked Food & Soil Sample

Sample	**Certified values(µg g-1)**	**Pb (II) (µg g-1) (Present)**	**Error (%)**	**Recovery (%)**
Bulked Foodsample	1.30	1.34	-3.07	98
Soil sample	1.62	1.60	+1.23	101

*Average of best three determinations among five determinations

Table 6: Estimation of Lead(II) in Biological sample, Soil & Vehicle Exhaust

Sample	**Concentration (µg g^{-1})**		**Error %**
	Certified value	**Found***	
NIES, No.1 Tea Leaves	0.8	0.78±0.04	+2.50
NIES, No.2 Human Hair	6.0	5.8±0.1	+3.33
NIES, No.3 Pond Sediment	105	103±1	+1.90
NIES, No.4 Vehicle Exhaust Particulates	219	218±2	+0.46

A study of Job's continuous variation and Molar ratio method revealed the stabilityconstant and the optimal ratio of Metal to Ligand ratio was found to be8.99 x 10^6 and 1:1 respectively. The plots were given in Figures.

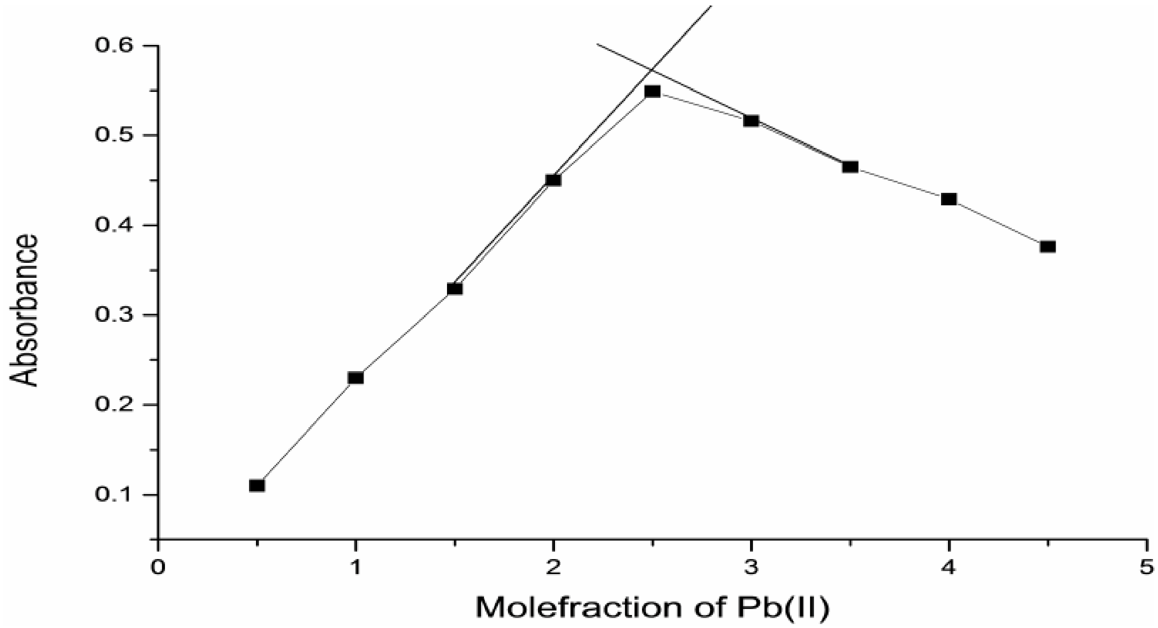

Figure 4: Job's curve of [Pb(II)-DMHBIH] complex

[Pb(II)-DMHBIH]	=	5 x 10^{-4} M
pH	=	9.0
Wavelength	=	430 nm
Triton X-100 (5%)	=	0.5 ml

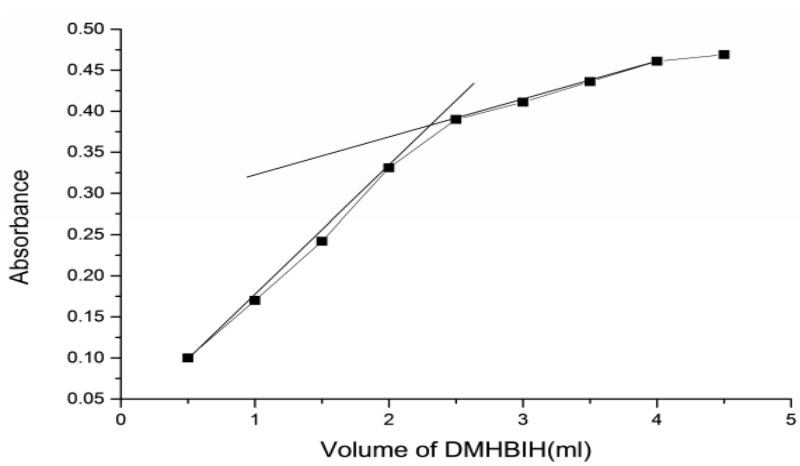

Figure 5: Molar ratio plot of [Pb(II)-DMHBIH] complex

[Pb(II)-DMHBIH]	=	5 x 10^{-4} M
pH	=	9.0
Wavelength	=	430 nm
Triton X-100(5%)	=	0.5 ml

Derivative Spectrophotometry:

The developed Zero order Pb (II)-DMHBIH system was employed for taking absorption spectrum of First and Second order spectrum and were shown (Figures) maximum amplitude at 470 and 539 nm respectively.

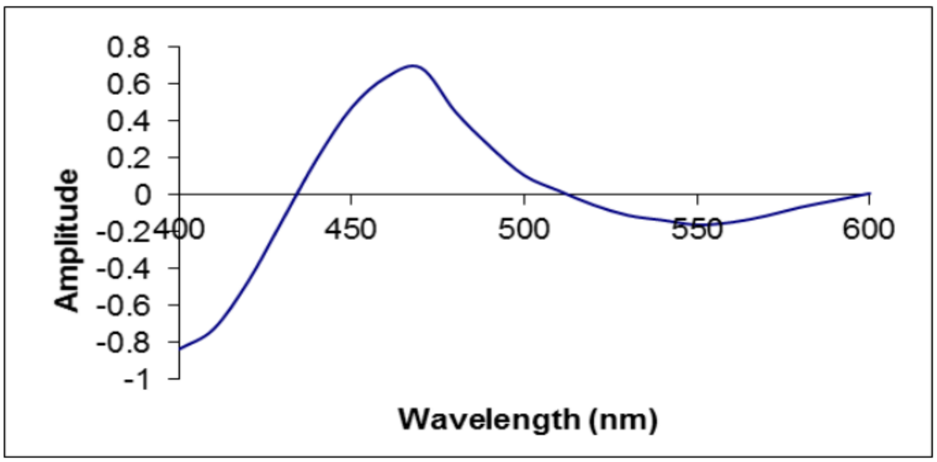

Figure 6: First derivative spectrum of [Pb(II)-DMHBIH] vs reagent

Pb(II)	=	2.5 x 10^{-5} M
[DMHBIH]	=	2.5 x 10^{-4} M
pH	=	9.0
Triton X-100 (5%)	=	0.5 ml

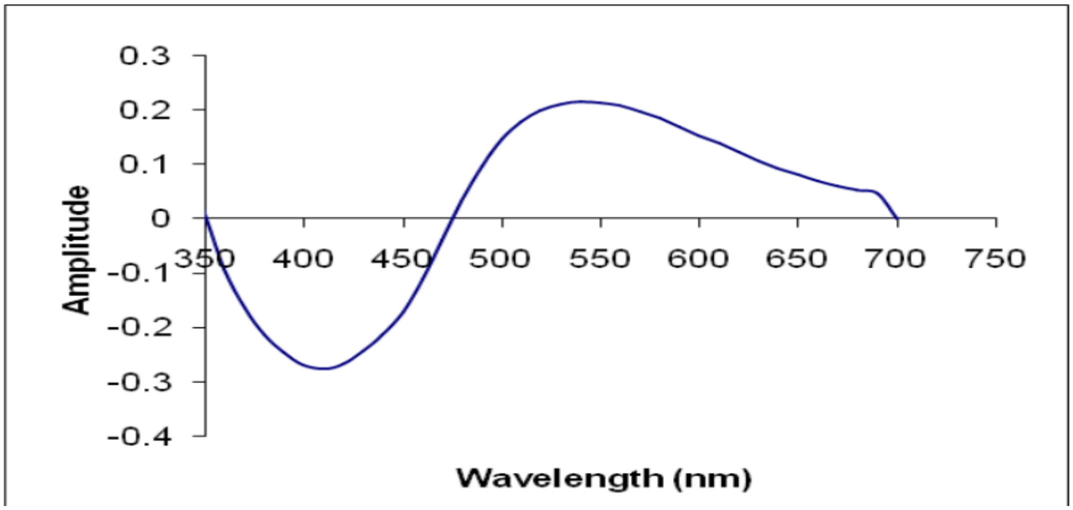

Figure 7: Second derivative spectrum of [Pb(II)-DMHBIH] vs reagent

Pb(II)	=	2.5×10^{-5} M
[DMHBIH]	=	2.5×10^{-4} M
pH	=	9.0
Triton X-100 (5%)	=	0.5 ml

The derivative amplitude was measured at 470nm and 539 nm for First and Second orderand plot were drawn between amplitude and amount of Pb (II) in µg/ml. The derivative system obeyed beer's law as shown in Figures.

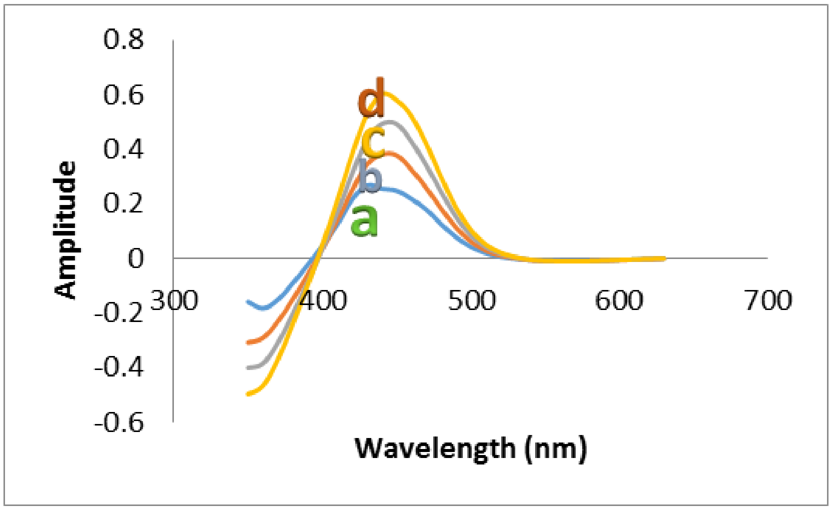

Figure 8: First derivative spectra of [Pb(II)-DMHBIH] vs reagent

Pb(II) (μg/ml) = A) 2.072; B) 3.108; C) 4.144; D) 5.180

DMHBIH] = 2.5 x 10^{-4} M

pH = 9.0

Triton X-100(5%) = 0.5 ml

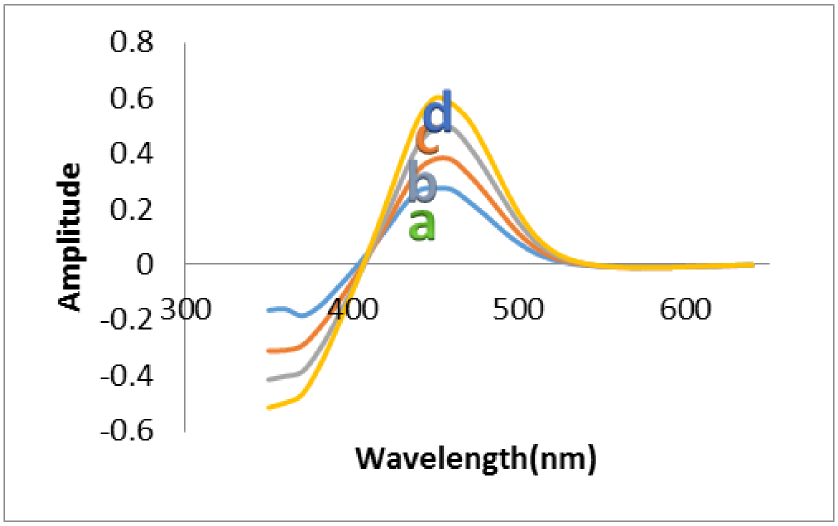

Figure 9: Second derivative spectra of [Pb(II)-DMHBIH] vs reagent

Pb(II) (μg/ml) = A) 2.072; B) 3.108; C) 4.144; D) 5.180.

[DMHBIH] = 2.5 x 10^{-4} M

pH = 9.0

Triton X-100(5%) = 0.5 ml

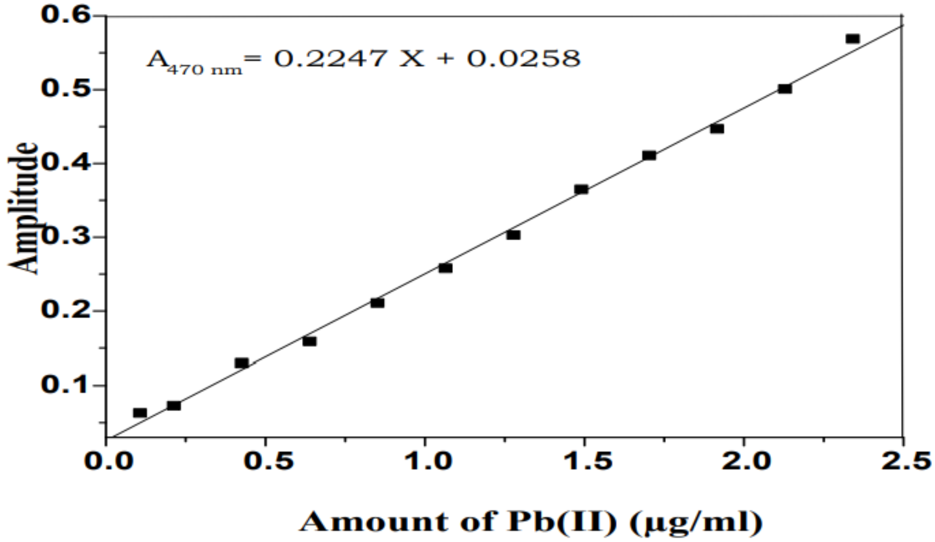

Figure 10: First derivative amplitude vs Amount of Pb(II)

[DMHBIH]	=	2.5×10^{-4} M
pH	=	9.0
Wavelength	=	470 nm
Triton X-100(5%)	=	0.5 ml

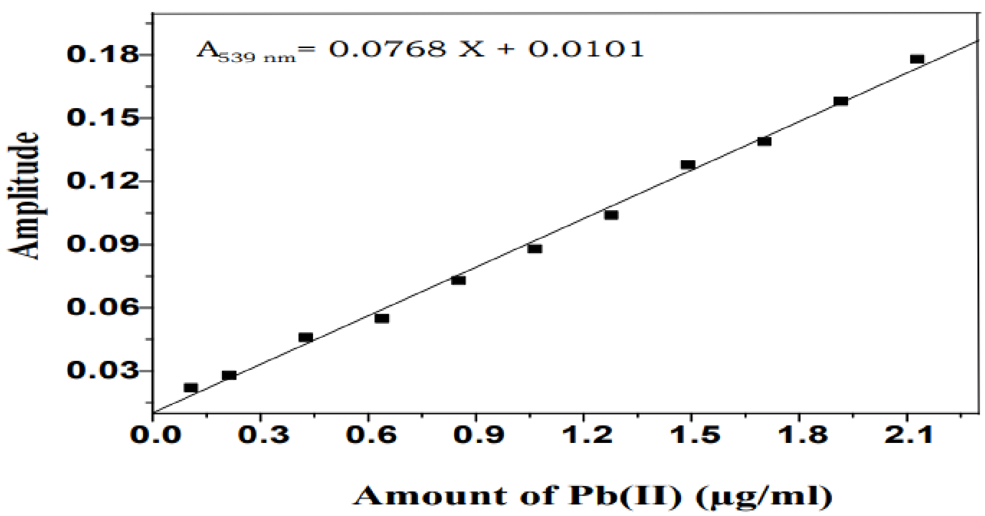

Figure 11: Second derivative amplitude Vs Amount of Pb(II)

[DMHBIH]	=	2.5×10^{-4} M
pH	=	9.0
Wavelength	=	539 nm
Triton X-100(5%)	=	0.5ml

Effect of foreign ions

The interference of anions and cations was observed in derivative method and the values are tabulated in Table. The experimental results shows that the order of interference is as follows i.e Zero order > First Order > Second order.

Table 7: Tolerance limit of foreign ions in the estimation of 5.18 µg/ml Pb(II)

Ion added	Tolerance limit µg/mL	Ion added	Tolerance limit µg/mL
Bromide	3196.16	Hg^{2+}	100.3
Iodide	2538	Co^{2+}	88.4
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Bi^{3+}	104.5		

* Masked with 5.0 µg/ml of Phosphate

†Masked with 762 µg/ml of Thiourea

Applications

Quantitative estimation of Pb(II) in Bulk Food, Soil, Vehicle Exhaust Particulates andBiological samples was done by employing the developed method and the results are presented in Tables.

Table 8: Estimation of Pb(II) in Bulked Food and Soil Samples

Sample	Certified values(µg g-1)	Pb (II) (µg g-1)(Present)	Error (%)	Recovery(%)
Bulked Foodsample	1.30	1.33	-2.30	98
Soil sample	1.62	1.65	-1.85	101

* Average of best three among five determinations.

Table 9: Estimation of Lead(II) in Biological sample, Soil & Vehicle exhaust

Sample	Concentration ($\mu g\ g^{-1}$)		Error %
	Certified value	**Found***	
NIES, No.1 Tea Leaves	0.8	0.77±0.04	+3.75
NIES, No.2 Human Hair	6.0	5.6±0.1	+6.66
NIES, No.3 Pond Sediment	105	103±1	+1.90
NIES, No.4 Vehicle Exhaust Particulates	219	218±2	+0.45

The experimental results were summarized in Table which indicates Analyticalcharacteristics of Pb (II)-DMHBIH system.

Table 10: Analytical Parameters of Pb (II)- DMHBIH system

Characteristics	Results
λ_{max}(nm)	430
Color	Bright yellow
pH	8.0-10.0
Reagent required	10 (folds)
Validity range (µg/ml)	0.414-10.360
Optimum concentration range (µg/ml)	0.83-9.32
Molar absorptivity ($L.mol^{-1}cm^{-1}$)	$1.82x\ 10^4$

Sandal's sensitivity (µg/cm^2)	0.01302
Stability constant of the complex	8.99x10^6
Composition of complex (M: L)	1:1
RSD (%)	2.6

CONCLUSION:

Here, spectrophotometric determination of Pb(II) using DMHBIH has been analyzed and received satisfactory result.

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