

## Direct Spectrophotometric Determination of Lead (II) in Industrial Samples using 1, 3-Benzenediamine, N, N'-bis(2-furanylmethylene) in Presence of Surfactant

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### Abstract

Schiff's base are one of the excellent chelating agents that used for the spectrophotometric determination of metal ions. It was observed that 1, 3-Benzenediamine, N,N'-bis(2-furanylmethylene) (BDFM) formed a brown colored complex with lead (II) at 620 nm. Lead (II) was chelated with the Schiff's base (BDFM) in acidic medium at pH = 3.5 using acetate buffer in presence of sodium laureth sulfate (SLS) as a surfactant. The effect of time, temperature and foreign ions were studied. The stoichiometric composition of the formed complex was found to be 1:2 (metal: ligand) by Job's method of continuous variation. Beer's law was obeyed in the range of 10-110  $\mu\text{g mL}^{-1}$  with molar absorptivity  $10.16 \times 10^3 \text{ L mol}^{-1} \text{cm}^{-1}$ . The proposed method was applied for the determination of lead in gasoline, solders, gutters and wastewater.

**Keywords:** Spectrophotometry, lead (II), 1, 3-Benzenediamine, N, N'-bis (2-furanylmethylene) (BDFM), SLS, Beer's law, Industrial samples.

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### Introduction

Schiff's bases have been known since 1864 when Hugo Schiff reported the condensation of primary amines with carbonyl compounds [1].

Schiff's bases are typically formed by the condensation of a primary amine and an aldehyde [2]. Schiff's bases and their metal complexes are known to exhibit anticancer, antioxidant and anti-inflammatory activities [3]. Transition metal Schiff's base complexes are used in various fields, such as medicine,

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agriculture, industries etc. [4]. The Schiff's bases have been effectively used as analytical reagents for extractive spectrophotometric determination of metal ions, as they possess excellent chelating properties [5].

Lead compounds are used in different industries worldwide due to their chemical and physical characteristics [6]. Lead is a cumulative poison that enters the body from lead water pipes, lead-based paints and leaded petrol. Presence of even traces of Pb (II) in environmental samples leads to environmental pollution and many fatal diseases including dysfunction of renal blood and neurological systems [7]. The toxicity of lead has been studied extensively. Lead poisoning in humans causes severe damage in the kidneys, liver, brain, reproductive system, and central nervous system, and sometimes causes death [8].

Lead is used in the preparation of acid storage batteries, ammunition, solder, sheet lead and tern steel. It is also used in the preparation of tetraethyl lead which is used as an antiknock agent in gasoline. Due to relatively low cost and excellent versatility, lead compounds are used as anticorrosive and highway traffic safety paints [9]. Lead determination is very important in the context of environmental monitoring however a large number of spectrometric methods for determination of lead are reported to face interference due to the presence of several metal ions [10].

In waters, lead is usually determined by flame atomic absorption spectrometry (FAAS), electrothermal atomic absorption spectrometry (ETAAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES), usually requiring a pre-concentration step, inductively coupled plasma-mass spectrometry (ICP-MS), and the colorimetric method based on dithizone [11]. Atomic absorption spectrometry was used as the developed method has been applied for the determination of lead (II) appearing as impurities in pharmaceutical substances [12].

A large number of simple sensitive spectrophotometric methods are used for determination of lead by some different reagents such as alizarine yellow [13], dichlorohydroxyl phenyl porphyrin [14], 3-hydroxy-3-*p* tolyl-1-*o* chloro phenyl triezene [15], alizarin red [16], N-(2'-Pyridyl)-4- hydroxybenzamide [17] and 4-(2-Pyridylazo)-Resorcinol [18]. These method offer the advantage of simplicity, rapidity, high sensitivity and direct determination of lead [19].

## **Experimental**

### **Apparatus**

T80 UV/Vis. Double beam spectrophotometer, PG instruments Ltd Company, United Kingdom was used for absorbance measurements and pH adjustment was carried out using (Adwa pH-meter, Model AD 1030, Romania) fitted with a combination glass-calomel electrode.



## Chemicals

All chemicals used were of analytical reagent grade (AR), and of highest purity available. They included:

Chemical	Company	Chemical	Company
Furan-2-carbaldehyde	Merk	Sodium acetate	Sigma
Benzene-1,3-amine	Sigma	Borax	Prolabo
Lead acetate	Merk	Boric acid	Merk
Acetic acid	Sigma	SLS	Prolabo
CTAB	Sigma	Methanol (90%)	Prolabo
Triton x100	Sigma	Ethylene glycol.	Prolabo
Ethyl alcohol (90%)	Prolabo	Glycerine	Prolabo
Dimethylsulfoxide	Prolabo	Acetone	Prolabo

Deionized water was usually used in all preparations.

**Ligand reagen:** Stock solution (100 mL) of ligand reagent of concentration ( $1 \times 10^{-2}$  M) was prepared by dissolving the appropriate amount (0.264 g) of ligand reagent in least amount of DMF as a solvent and complete to 100 mL with deionized water.

**Standard lead solution:** lead (II) stock solution of 0.001M was prepared by dissolving 0.0325 g of  $[\text{Pb}(\text{CH}_3\text{COO})_2]$  in 100 mL of deionized water and solutions of lower concentrations were prepared from the stock solution by dilution and the solution was standardized by EDTA [20].

**Surfactant solutions:** The surfactants, Sodium laureth sulfate (SLS), Triton X 100 and Cetyl trimethyl ammonium bromide (CTAB) were prepared at concentration ( $1 \times 10^{-2}$  M) by dissolving the calculated amounts in deionized water and used without further purification.

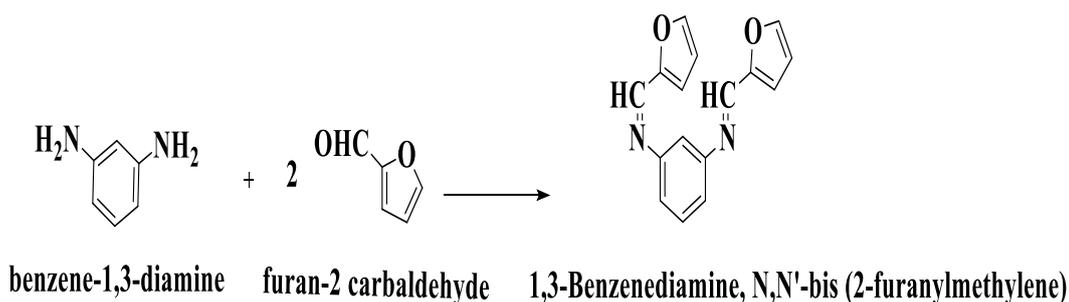
### Buffer solutions

For the preparation of buffer solutions, different types of buffers including acetate buffer in the range of (3.5-6), universal buffer (1.81-11.98) [21], borate buffer (7.6-10) [22], citrate buffer (3-6), phosphate buffer (7-11) and acetate buffer (3.5-6) [23] were prepared.

## Procedures

### Preparation of Schiff's base

A mixture of substituted benzene-1,3-amine (10 mmol) (1.08g) and furan-2-carbaldehyde (20 mmol) (1.65ml) ( $d=1.16$ ) were dissolved in 10 mL absolute ethanol. The mixture was refluxed for 5 hours. It was then cooled and diluted with ice cold water. The resulting solid was recrystallized from ethanol to the proposed Schiff bases.



Chemical Formula	C <sub>16</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub>
Exact Mass	264.09 g/mol
Systematic name	1, 3-Benzenediamine, N,N'-bis(2-furanylmethylene)
Molecular Weight	264.28 g/mol
m/z	264.09 (100.0%), 265.09 (18.1%), 266.10 (1.4%)
Elemental Analysis	C, 72.72; H, 4.58; N, 10.60; O, 12.11

### Preparation of solid complex

The complex was prepared by addition of 10 mL hot solution (0.1M) lead acetate [(Pb(CH<sub>3</sub>COO)<sub>2</sub>)] to 20 mL hot solution (40°C) of the Schiff's base BDFM (0.1M) with 3.00 mL of acetate buffer pH=3.5. The resulting mixture was stirred under reflux for 1.0 hour where up on the complex precipitated. This was collected by filtration, washed with 10 ml a 1:1 ethanol-water mixture and finally by 10 ml diethyl ether.

### Absorptioncurve

Transfer a 1.00 mL of Pb (II) solution (1×10<sup>-3</sup>M), 2.00 mL of reagent solution (1×10<sup>-2</sup>M) and 3.00 mL acetate buffer pH=3.5 into 10 mL measuring flask and complete to the mark with deionized water. At once, the maximum absorbance was measured by scanning wavelength from 200 to 800 nm against the reagent blank.

### In presence of surfactant

In presence of 1.00 mL surfactant SLS solution (1×10<sup>-2</sup>M) take 1.00 mL of Pb (II) (1×10<sup>-3</sup>M), 2.00 mL of reagent solution (1×10<sup>-2</sup>M) and 3.00 mL acetate buffer pH=3.5 were mixed in 10 mL measuring flask and complete to the mark with 3.00 mL deionized water. At once, the absorbance for the sample was measured by scanning wavelength from 200 to 800 nm against the reagent blank.

### Beer's law

A series of solutions containing a known volume of ligand reagent and different concentration of Pb (II) ranging from 10-110 µg mL<sup>-1</sup>, were mixed in 10 mL voltametric flask, with known volume of acetate buffer pH=3.5 and 1.00 mL SLS at once at 30°C, the absorbance was measured at 620 nm against blank solution for Pb (II).

## Applications

### A-Determination of Pb (II) in solders

A weighed sample (0.5 g) was dissolved in 15 mL hydrochloric acid (1:1) with gently heating for few minutes. The solution was cooled to room temperature and filtered. The solution was diluted to 100 mL. The above solution was mixed with ligand reagent, mask copper by using sodium thio sulfate [20] and measure absorbance against blank.

### B-Determination of Pb (II) in gutters

A 1.5 m of gutters was dissolved in 15 mL concentrated hydrochloric acid with gently heating for few minutes. The solution was cooled to room temperature and filtered. The solution was diluted to 100 mL. The above solution was mixed with ligand reagent according to the above.

### C- Determination of Pb (II) in gasoline<sup>80</sup>

A 2.00 mL of the gasoline solution was mixed with ligand reagent and the absorbance was measured at 620 nm against the blank according to the above procedures.

### D- Determination of Pb (II) in wastewater sample

1.00 mL of the wastewater sample was mixed with ligand reagent and the absorbance was measured at 620 nm against the blank according to the above procedures.

## Results and discussion

### Absorption curve

BDFM ligand reagent was used as a new spectrophotometric reagent for the determination of lead (II) by forming brown coloured stable complex at 620 nm for Pb (II), as shown in Fig. (3), while Fig (1), Fig (2) represented the IR spectra of ligand and the complex respectively.

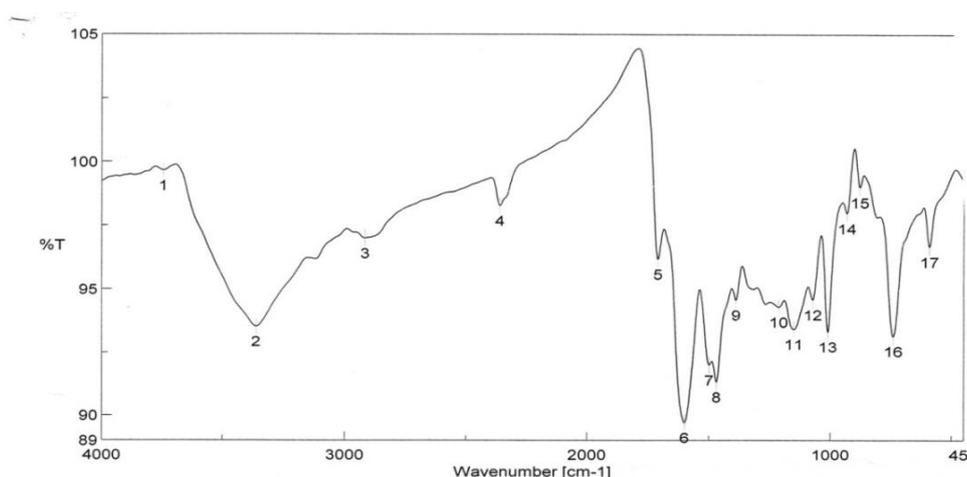


Fig (1): IR spectra of ligand (BDFM)

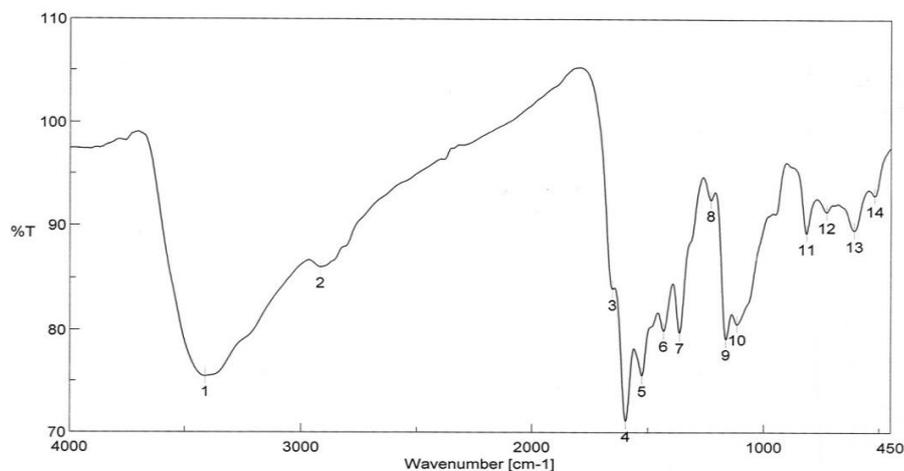


Fig (2): IR spectra of lead solid complex

### Effect of pH and buffer solution

The study of the effect of pH on the colour intensity of the complexes showed that the appropriate pH range was from 1 to 5 with maximum value at pH=3.5 for Pb-BDFM complex as shown in Fig. (4). Studying the effect of different buffer types in the range from (3-5) indicated that the maximum absorbance values were obtained using acetate buffer at pH=3.5 for complex as shown in Fig. (5) and Fig (6).

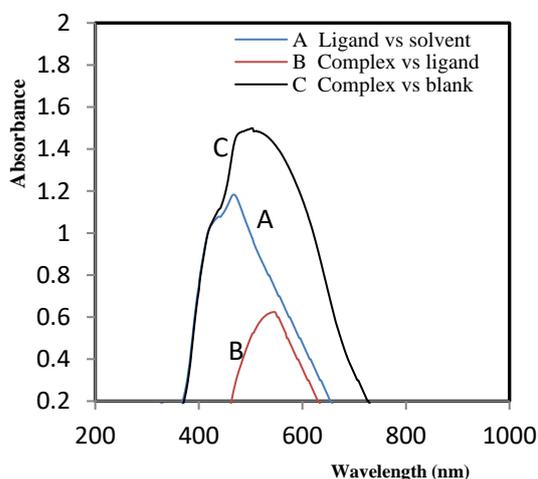


Fig (3): Absorption spectra for Pb (II) With BDFM complex.

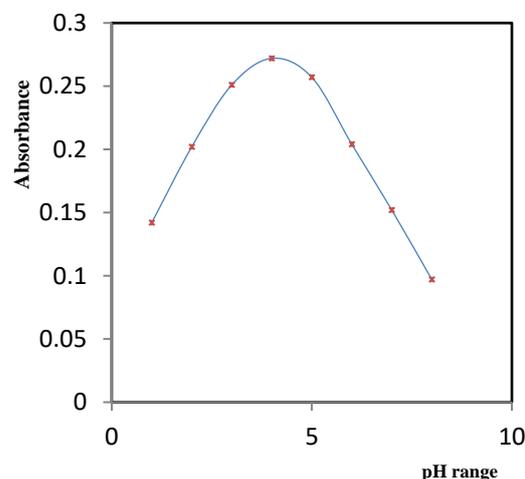


Fig (4): Effect of different pH on the formation for Pb (II) with BDFM

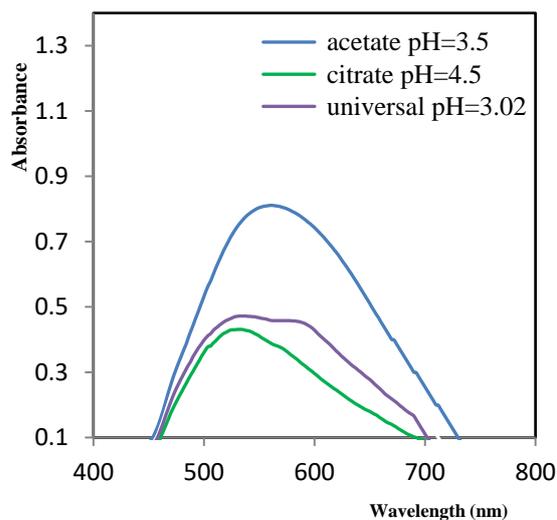


Fig (5): Effect of different buffers on the formation of Pb (II)-BDFM complex

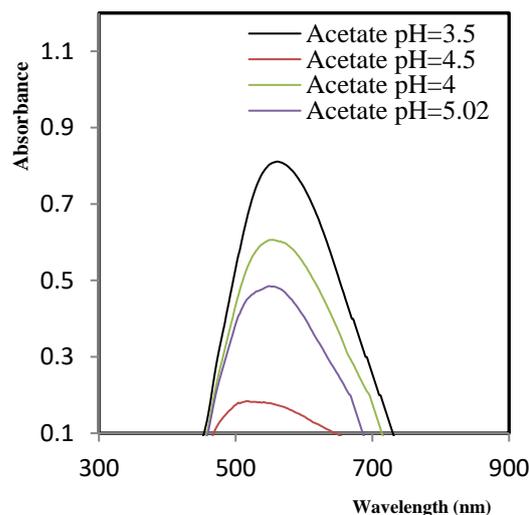


Fig (6): Effect of different acetate buffers on the formation of Pb (II)-BDFM complex.

### Effect of organic solvents

The effect of organic solvents on the absorption spectra of the formed complex was studied in methanol, ethanol, isopropanol, acetone, DMSO, glycerine, ethylene glycol and formaldehyde as solvents and the absorbance was measured in presence of other optimum conditions against the blank solution. The results illustrated that, the value of absorbance was not improved by using any organic solvents as shown in Table (1) and Fig (7):

Table (1): Effect of different solvents of and lead (II).

Effect of solvent	$\lambda_{max}$	Absorbance
Without	560	0.811
Acetone	544	0.561
DMSO	558	0.653
Methanol	572	0.744
Ethanol	582	0.765
Ethylene glycol	550	0.497
Glycerine	566	0.752
Formaldhyde	580	0.449
Iso propyl alcohol	558	0.528

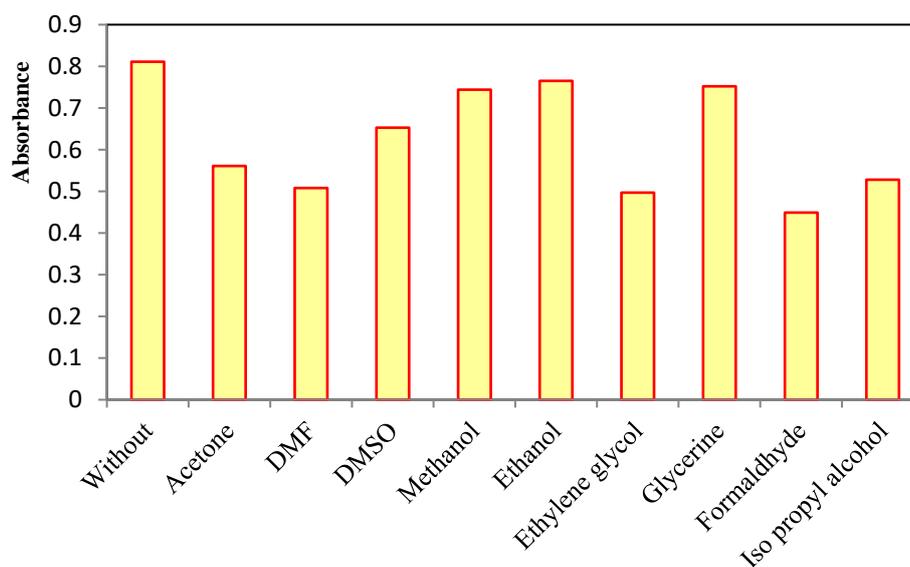


Fig (7): Effect of organic solvents.

### Effect of surfactants

By studying the effect of many different surfactants such as SLS, CTAB and Triton X 100, the results showed that the presence of 1.00 ml of SLS as a surfactant shifted the absorption peak to hyper and bathochromic shifts and this may be due to the formation of micelles or formation of double or triple complex with surfactant itself as showed in Table. (2).

Table (2): The effect of different surfactants on lead (II) complex

Effect of surfactant	$\lambda_{\max}$	Absorbance
Without	560	0.811
SLS	620	0.994
CTAB	612	0.678
Triton X100	578	0.756

### Other optimum conditions

Some other effects such as the effect of sequence of the addition, standing time and temperature were also studied on the formation of lead (II) with BDFM complex.

### Effect of Time

The effect of time on the formation and color stability of Pb (II) – BDFM complex was studied to determine the optimum standing time for the analytical procedure by measuring the absorbance of the identical samples containing the metal ions, after intervals of time ranging between 5 and 120 minutes versus the blank. The results indicated that, Pb (II) – BDFM complex was instantaneously formed and remain stable for a 20 h as shown in Table (3) with Fig (8).

Table (3): Time affecting on the formation of Pb (II) complex.

Time, min.	At once	5	10	15	30	45	60	75	90	120
Absorbance	0.81	0.86	0.87	0.88	0.89	0.90	0.90	0.90	0.91	0.92

### Effect of Temperature

The effect of temperature was studied for the complex under investigation at different temperatures ranging from (15-65°C) by heating or cooling the sample and the blank in water or ice bath, then the absorbance was measured against the reagent blank. It was found that, the maximum stability of the complex at 25°C and then decrease gradually by raising the temperature as shown in Table (4) with Fig (9).

Table (4): Temperature affecting on the formation of Pb (II) complex

Temperature, °C	20	25	35	45	55	65	75
Absorbance	0.800	0.811	0.705	0.633	0.627	0.630	0.550

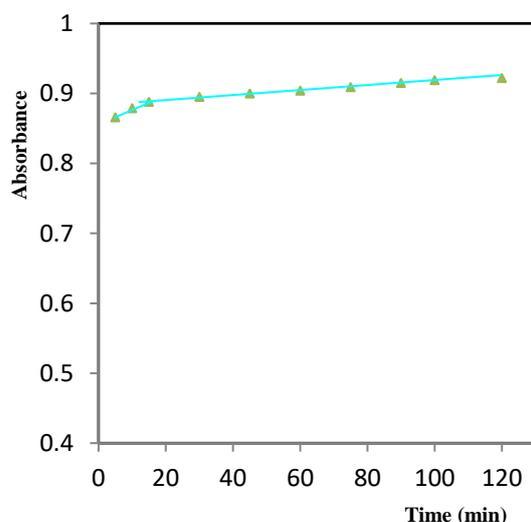


Fig (8): Effect of time on the formation of Pb (II)-BDFM complex.

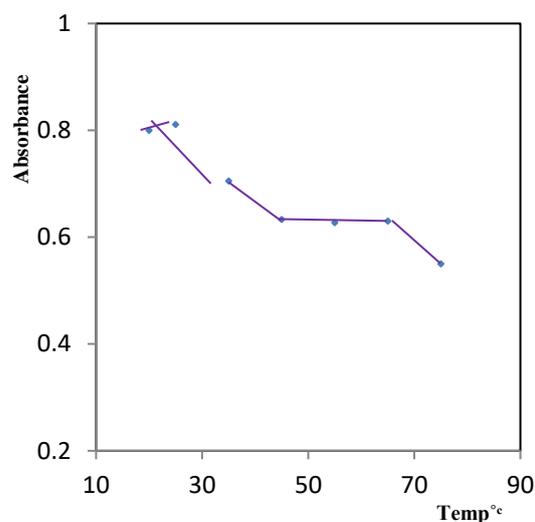


Fig (9): Effect of temperature on the formation of Pb (II)-BDFM complex.

### Determination of the stoichiometric ratio

#### The molar ratio method

For investigating the molecular structure of the complex formed between, Pb (II) and BDFM, a series of solutions was prepared in which the metal ion concentration was kept constant at 1.00 mL ( $1 \times 10^{-3}$  M), while that of the ligand was regularly varied from 0.1 to 0.8 mL of ( $1 \times 10^{-3}$  M). The absorbance of these solutions were measured at 620 nm with all optimum conditions, and

then plotted against the molar ratio [ligand] / [metal]. Fig. (10) gave the absorbance - molar ratio curve which showed that Pb (II) formed a 1:2 complex through the combination with the metal ion as shown in Fig (10).

### Using the continuous variation method

A series of solutions were prepared by mixing an equimolar solution of Pb (II) and ligand in different proportions from 0.1 mL to 0.9 mL ( $1 \times 10^{-3}$  M) of the same concentration while keeping the total molar concentration constant. A plot of absorbance measured at the recommend wavelength versus mole fraction was used to determine the metal-ligand ratio. Fig. (11) showed that the maximum absorbance obtained at a ratio of 1:2 for the complex.

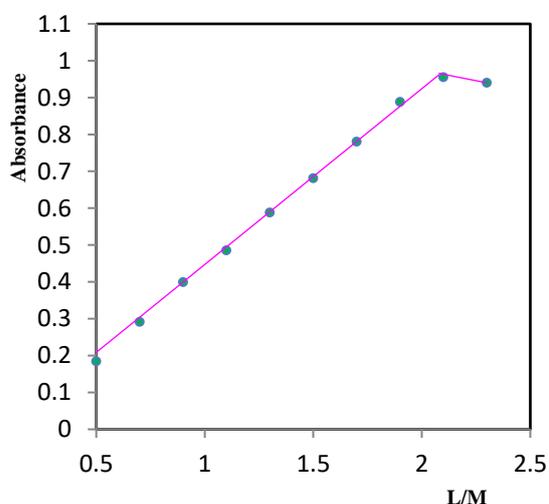


Fig. (10): Determination of the metal-reagent ratio of Pb (II) with BDFM complexes using molar ratio method.

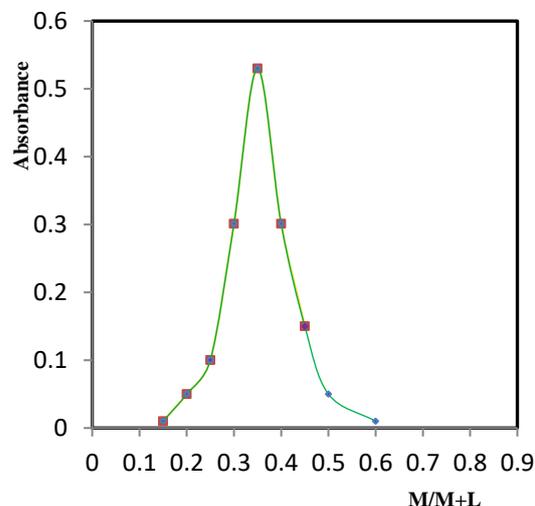


Fig. (11): Determination of the metal-reagent ratio of Pb (II) with BDFM complexes using continuous variation method.

### Calibration curve

Several complexes were prepared where the concentration of the ligand was kept constant at  $1 \times 10^{-2}$  M while the concentration of the metal ion was varied from  $1 \times 10^{-6}$  to  $1 \times 10^{-3}$  M and their absorbance values were measured versus the concentration of lead (II) at 620 nm in presence of SLS and other optimum conditions. Beer's law was obeyed from 10 to  $110 \mu\text{g mL}^{-1}$  for Pb (II), as shown in Fig. (12).

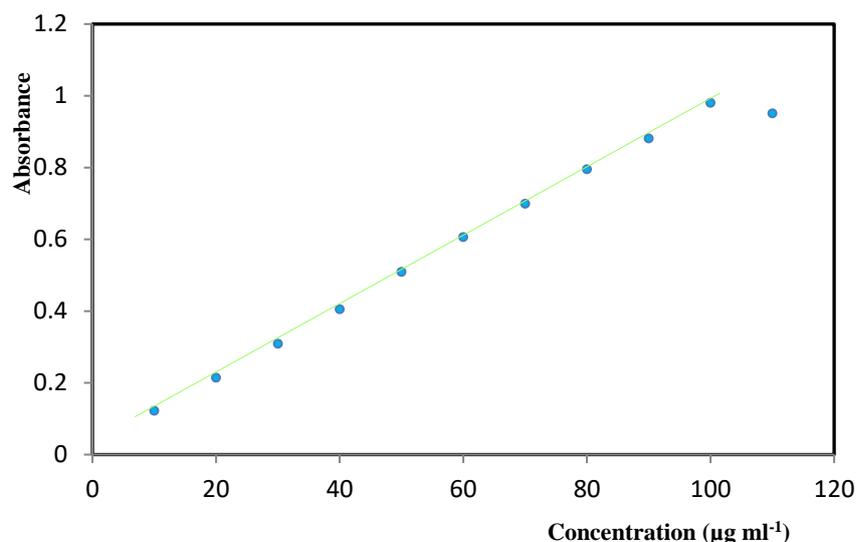


Fig. (12): Calibration curve of Pb (II) With BDFM complex.

### Analytical and statistical characteristics of Pb (II) - BDFM complex

The molar absorptivity, limit of detection, limit of quantification, relative standard deviation and student t-test were calculated. The data indicates high sensitivity and the good accuracy of the proposed method as showed in the Table-5.

Table-5: Analytical and statistical characteristics of Pb (II) complex.

Parameter	Value
Molar absorptivity ( $\epsilon$ ) L mol <sup>-1</sup> cm <sup>-1</sup>	$\times 10^3$ 10.16
Standard deviation (SD)	0.017320
Relative standard deviation (RSD %)	0.0288
Standard error of the mean (SEM)	0.0065463
Student t-test	3.3605
Limit of quantification (LOQ) µg ml <sup>-1</sup>	19.2541
Limit of detection (LOD) µg ml <sup>-1</sup>	6.7389

### Effect of foreign ions

The effect of 50 fold molar excess of many species of different cations and anions were tested in the determination of Pb (II) with Schiff's base ligand. The results showed that as (III), Co (II), W (VI), Cd (II) and Ni (II) interfere in the determination of lead (II) with BDFM.

### Application

The proposed method can be applied for the determination of lead (II) in gasoline, solders and gutters as the above procedures. The results give good and accurate results as shown in table (6).

Table (6): Determination Pb (II) in some industrial samples

Sample	Taken ( $\mu\text{g ml}^{-1}$ )	Found ( $\mu\text{g ml}^{-1}$ )	Recovery %	RSD %
<b>Determination of Pb (II) in</b>				
Gasoline	11	10.88	98.90	1.1
Solders	20	19.89	99.45	0.55
Gutters	24	24.40	101.66	1.66
Wastewater	11	10.88	98.90	1.1

## Conclusion

New, simple and accurate spectrophotometric method was described for the determination of lead (II) based on the formation of colored complex between the metal ion and the reagent (BDFM). The complex has absorbance maxima at 620 nm in presence of SLS as a surfactant; with molar absorptivity  $10.16 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$  the complex was stable at room temperature and for a long time. The method was applied for the determination of lead (II) in some industrial samples.

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## التقدير الطيفي الدقيق للرصاص الثنائي في عينات صناعية باستخدام 3,1 بنزين ثنائي الامين 2- ثنائي فيورنيل ميثيلين في وجود مواد نشطة سطحيا

منير زكى أ ، علاء أمين ب، خالد الجندى أ و أيه جمعه

أ قسم الكيمياء كلية العلوم، جامعه الزقازيق، الزقازيق، مصر  
ب قسم الكيمياء كلية العلوم، جامعه بنها، بنها، مصر

تتميز قواعد شيف بقدرتها على تكوين متراكبات ملونه مع ايونات العناصر بطريقه طيفيه. ولوحظ أنه 3,1 بنزين ثنائي الامين 2- ثنائي فيورنيل ميثيلين تكون متراكب بنى اللون مع عنصر الرصاص عند طول موجى 620 نانومتر. وهذا المتراكب المتكون مع قاعد الشيف يتكون عند الاس الهيدروجينى 3,5 باستخدام محلول منظم من حامض الخليك وخلات الصوديوم وفي وجود ماده نشطه سطحيا وهى صوديوم لوريل سالفيت. وقد تم دراسته تأثير عوامل كل من الوقت ودرجه الحراره ودراسة تأثير الانواع المختلفه من الايونات والكاتيونات وما قد تسببه من تداخل على تكوين المتراكب قيد الدراسة. ووجد أن نسبه تكوين المتراكب المتكون هى 2:1 باستخدام طريقتى التغير المستمر و النسبه المولاريه. وبتطبيق قانون بيير وجد أن هناك علاقه خطيه بين الامتصاص ومدى من التركيز تتراوح بين 10-110 ميكروجرام / مللى كما تم حساب قيمه الامتصاص  $10 \times 10^3$  لتر/ مول. سم. تم تطبيق الطريقه المقترحة لتقدير الرصاص في البنزين واللحم وأنابيب الرصاص الخام ومياه الصرف الصحي.