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A new method for determination of lead ion (II) using the organic detector 2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole)

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KEYWORDS

Azo reagent,
Lead ion,
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ABSTRACT

The study includes the determination of pb(II) using azo reagent 2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole) spectrophotometric at (λ_{max} 548 nm) wave length. The effect of different parameters on determination is evaluated. The study shows that best PH= (7±0.1). The study includes obey concentration of Beer-Lambert law to determination of the calibration graph of Pb (II) by azo reagent. From this graph we note obey the concentration of law was between 5–60 µg of Pb(II) after this values the absorbance increased. The stoichiometry of complex lead ion is determined by molar ratio method. Result indicated reagent to metal ratio was (1:2) the stability constant of the complex determination through mole ratio method and its value indicated good stability. The UV-Vis spectra and the IR spectra of the complex and reagent were measured. From all mentioned studies proposed structural formula of Pb(II) complex.

Introduction

Lead is considered one of the oldest metals used in the human history. It was a metals as well as Arsenic and Antimony, which has been piloted in the Bronze Age, the first for the preparation of Bronze, whiles the use of tin. Lead was mentioned by Al Khwarizmi in his book (Aljemahir Fi Marifat Aljawahir) and he devoted a section to it. Raw Lead is a metallic element which color between blue and gray, and when cutting its surface, it would be a glimmering silver metallic, but it quickly turns to a pale color between gray and blue, and this color represents the main feature of the lead. Lead has low tensile strength and poor conductor of electricity and resistant to erosion. The most important ore of lead presence in nature is “Galena ore” which contains other elements, a sulfur, copper and zinc (Bulut and Baysal, 2006). Most oxidative cases of lead are the +2 and +4 (Kempf and Jonnborn, 1976). Lead compounds are used in large quantities in the storage batteries shingles that work in cars and in the packaging of electric cables, also large amounts of lead used in the lining of pipes, reservoirs and X-ray machine, and because of its high density and nuclear properties it used widely as a shield from radioactive materials and is also used in the metal mixing such as welding alloy and bearing metallic materials. Also large amounts of lead consume in the form of compounds especially paints and dyes (Bordas and Bourg, 2000). As a result of those extensive uses of lead compounds, serious environmental pollution produced such as radioactive contamination in the cities of Iraq, which showed the high proportion of air pollution with lead ions that released from chemicals and rocket-propelled grenades that have been used in wars fought previously (Landrige and Todel, 1994). Recently scientific studies have shown that the adverse health effects occur at low levels of exposure to lead, and may affect on the nervous system and lead exposure may have subtle effects on the intellectual development of infants and children. Also lead contamination results in problems in human reproduction which leads to underweight children birth and problems in the nervous system (Nriago, 1978).

Aldaithazon way is one of the methods used to set the duo ion lead it’s the most sensitive and selective for appointment of the metalloproteinase ion (Obrien, 2002). Other technologies are set to spectrally lead differ in their selectivity and sensitivity and their estimation detection limits (Prosi, 1989; WHO, 1977; Fernandes –Leborans and Olalla –Herrero, 2003; IPCS, 1995).

The present study was conducted to estimate the ion lead(II) spectrally using a reagent 2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole) at λ_{max} 548 nm wavelength which contains inhomogeneous loop in its structure different atoms is nitrogen (Karadjova *et al.,* 1998), which owns electronic pairs nonbonding qualifies them to link with the different elements especially metallic ones to form complexes (Fig. 1).

Materials

Solutions

1 - Ion lead (II) solution (1×10^{-3}) molar: attended by dissolving 0.332 grams of lead nitrate in 100 ml of distilled water and from which attended diluted concentrations using volumetric flask 10 ml.

2 - Organic reagent solution (1×10^{-3}) molar attended by dissolving 0.375 grams of reagent in 100 ml of ethanol and from which attended diluted concentrations using volumetric flask 10 ml.

3 - Buffer solutions with different PH functions between (4-10) as tablets form, which equipped directly from the MZN / India Company and have been calibrated with a pH meter.

The used devices

1- PH measurement device (PH meter Hanna/ Italy).
2- (UV-Vis) Spectroscopy measuring device (PG/ USA 990).
3- Spectroscopy FTIR measuring device (Shimadzu 66x).
4- Melting point measuring device (Thermo/ Germany).

Methods

The optimal conditions was studied for estimating Lead ion (II) spectrometry using the organic detector (2,2-(1,4-phenylene) bis(5-phenyl-1,3 oxazole)) the best acidic function for complexity has been appointed, by preparing several solutions of different acidic functions ranged between (4-10) using buffer solutions of ammonium acetate, and use equal sizes for both of the metal ion and organic reagent, in an equal concentrations. also the extent of concentrations compliance of Beer-Lambert law was studied by mixing 2 ml of the metal ion solution which containing (10-100) micrograms of the mentioned ion with the equivalent volume of the of the organic reagent with concentration (0.001%). Also the complex equivalence was study by the molar ratios method which included the preparation of different concentrations of the organic reagent ranged (0.5×10^{-4}-1.5×10^{-4}) Molar certifying the metal ion concentration and by which the stability constant for the lead complex(II) has been calculated, and to find out the effect of the presence of concentrations of some negative and positive ions on the complexity process these ions added to the metal ion solution and the organic detector. The melting point was measured for each of the metal ion and the organic reagent that used.

Result and Discussion

The effect of different factors was studied to determine the optimum conditions for the process of complexity of Lead ion (II) and reagent (1-Hydroxy-7-Azabenzotrizole), these factors are:

Acidic function

Buffer solutions of metal ion were prepared in a concentration of (1×10^{-5}) Molar with acidic function ranged between 4 and 10, then the same volume of the organic reagent added to them, of an equivalent concentration to the metal ion, it observed variation the complexity of the process with variation of the acidic function as shown in figure 2, the complex showed higher absorption when the acidic function (7) so it is the best function of the consistency process between all of the mentioned ion and the reagent, this results are consistent with previous studies to set the lead ion (II) (Galas-Gorcher, 1991; Sillanp M Jansson, 1992) because it is the best acidic function in which the reagent can give non covalent electronic couples to participate in the consistency process on the one hand and on the other hand the aggregates viable consistency become more freedom to contribute in the complexity process at this point.

While the acidic function (9) showed high absorption value but the color of the reagent has change, the reason may be attributed to aggregates arrangement change and ties in the structure of the organic reagent along the lines of the principle of chemical indicators.

Metallic ion concentration

Different concentrations of Lead ion(II) were prepared to determine the obey concentrations of Beer's- Lambert law ranged between (5-60)μg/ ml, then the same volume of organic reagent of the concentration (0.001%) was added for each solution, then measured at the optimum acidic function and the results were as set out in figure 3, we note that obey of the mentioned law is located between the concentrations of 10 and 40 μg/ml equivalent the concentrations (1.5×10^{-4} - 15×10^{-4}) molar after these concentrations absorbance values deviate a positive deviation, the reason may be due to increasing of the mentioned ion concentration comparing to the organic reagent concentration which upset the balance process and distort the reaction to back according to Le Shateli principle and in figure 3 we note that the value of the molar absorption constant is (1.5×10^{4}), where this value indicates the good stability of the formative complex.

Table 1 shows that this method using organic reagent (1-Hydroxy-7-Azabenzotrizole) fall within the most accurate, sensitive and selective for the appointment of Lead ion (II).

The formative complex valence

Molar ratio adopted for the study of valence of Lead complex(II), where different concentrations of organic reagent were prepared in the range between (0.25 × 10^{-4} - 2.5×10^{-4}) molar and constant concentration of the metal ion, the results are shown in figure 4, and the proportion of the organic reagent to the metal ion was found equal to 1:2 and by which the stability constant of the complex was calculated(14), which amounted (0.904 × 10^{4}), this value refers to the good stability of this complex.

The effect of positive and negative ions

A concentration of 50 mg/liter of a group of positive and negative ions separately added to the mentioned ion solution and reagent to find out the effect of the presence of a number of positive and negative ions on the spectral estimation process of Lead ion (II). The results displayed in table 2. Those results showed influence of negative ions on the complexity process and the reason has been attributed to the fact that negative Lead ion (II) produces stable complexes with both Tartrates Sulfates ion and less stable complexes with Chloride ion (IPCS, 1995) on the one hand and on the other hand the presence of positive ions affects the consistency process, especially Cadmium ion (II) and Nickel (II) with the organic reagent (International Resource Panel), which affects negatively on the basic consistency process.

Table.1 The values of the molar absorption coefficient to set the Lead ion (II) using different reagents

The ion	Molar absorption	Reagents
Pb(II)	6.86 x 10^{4}	Dithzone
	4x 10^{4}	4-(-2- Pyridylazo)resorcinol
	1x 10^{4}	6-MBTABP
	4.9 x 10^{4}	5-(5- BrmoPyridylazo)-5-diethylamino phenol
	1.04 x 10^{4}	Xylenol orang
	1.5 x 10^{4}	1-Hydroxy-7-azabenzotriazole
	1.24 x 10^{4}	**2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole**

Fig.1 The chemical composition of organic reagent 2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole) m.w 364.12

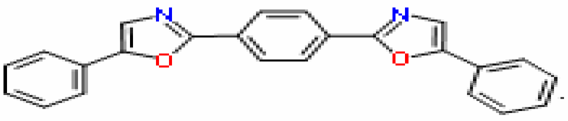


Table.2 The effect of the presence of some ions on the process of complexity through the values of the relative deviation (E) *

Positive ion	relative deviation (E)	Negative ion	relative deviation (E)
Na^+	0.043	$SO4^{-2}$	0.213
Mn^{+2}	0.207	Tartrate	0.152
Mg^{+2}	0.291	IO^{-3}	0.043
Ni^{+2}	0.324	Br^-	0.248
Zn^{+2}	0.231	MnO^{-4}	0.027
Ba^{+2}	0.126	CrO_4^{-2}	0.329
Cd^{+2}	0.364	-	-

*The concentration of the metal ion 1 × 10-4 molar

Table.3 Some qualities of each of the complex and the organic detector

The substance	Color	Melting point ^oC	Wave length λ_{max} 548 nm	Molecular weight g mol^{-1}
The reagent	Green	180	330	136.11
The complex	Red brown	206	548	490

Table.4 The values of the infrared spectra frequencies FTIR (cm-1) for both the organic and the complex detectors

Compound	**υ O-H str.**	**C-N strυ**	**υ C-H alp.str**	**υ C-H arm.str**	**υ C=O**
The reagent	**-**	**1531.37(s)**	**2921.96(m) 2904.6 (m)**	**3056.95(w)**	**1739.67(s)**
The complex	**3259.47(s)**	**1591.16(s)**	**2927.74(m) 2856.38(m)**	**3095(w)**	**1743.53(s)**

Fig.2 The relationship of the acidic function with the absorption for lead ion complex(II)

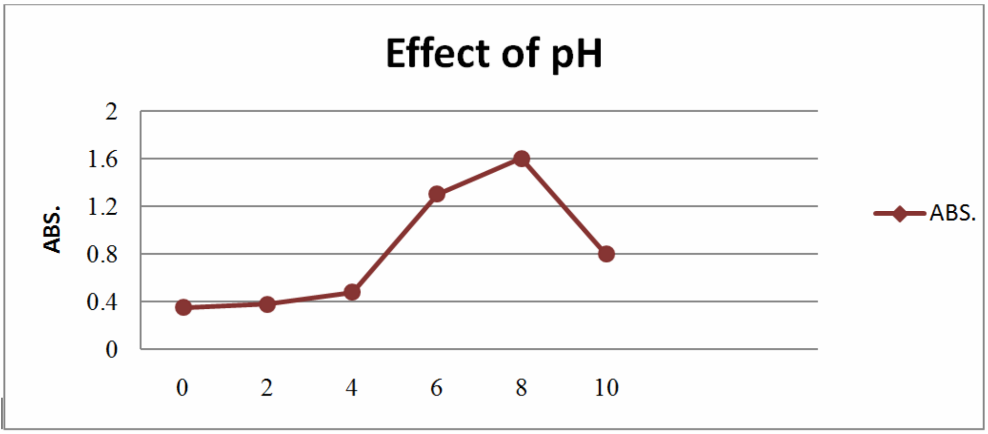


Fig.3 The standard calibration curve for duo ion lead using the new organic reagent (2,2-(1,4-phenylene)bis(5-phenyl-1,3 oxazole))

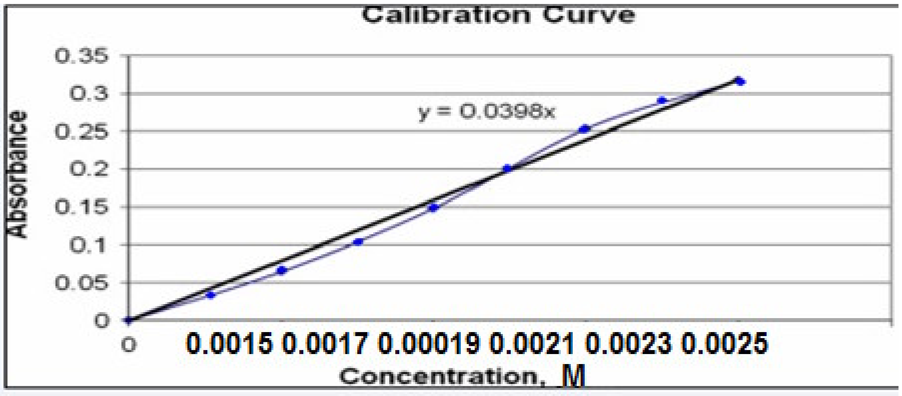


Fig.4 The relationship between the molar ratio M:L and the absorption of Lead complex (II) at the greatest wavelength

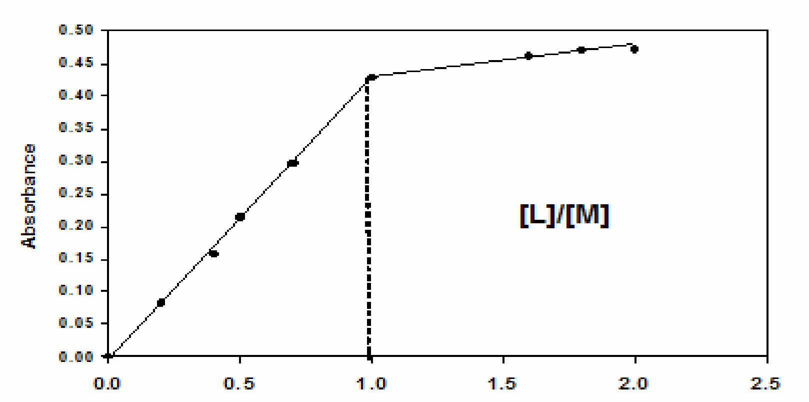
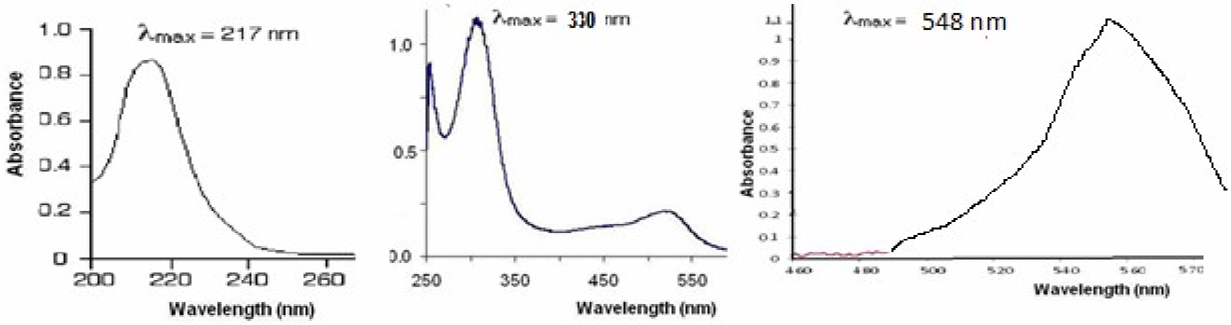


Fig.5-1 UV-visible spectrum of metallic ion; **Fig.5-2** UV-visible spectrum of reagent; **Fig.5-3** UV-visible spectrum of complex



Figure(5 -1):The spectrum of Pb(II) solution Figure(5 -2):The spectrum of reagent Figure(5 -3):The spectrum of complex

Fig.6 Infrared image of the detector (1-Hydroxy-7-Azabenzotrizole)

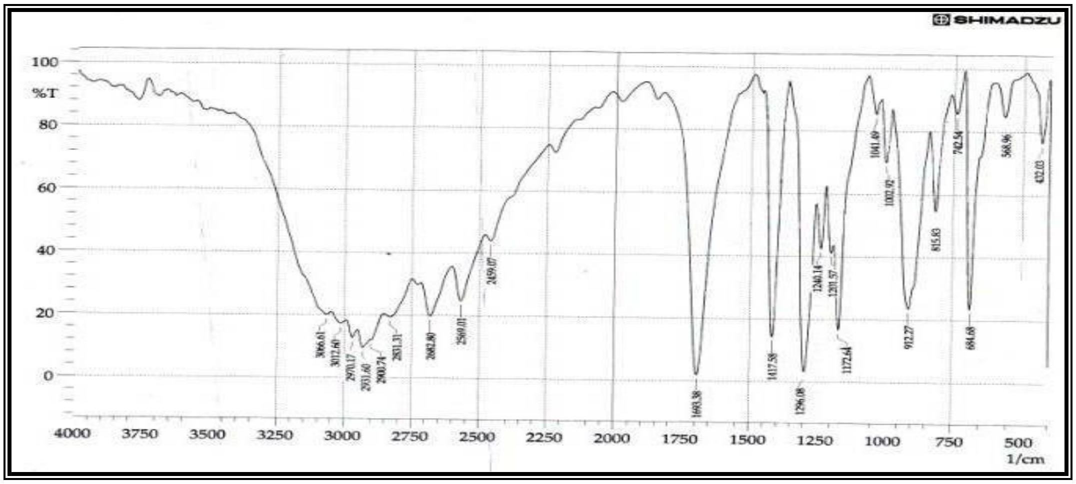


Fig.7 Infrared image of the complex

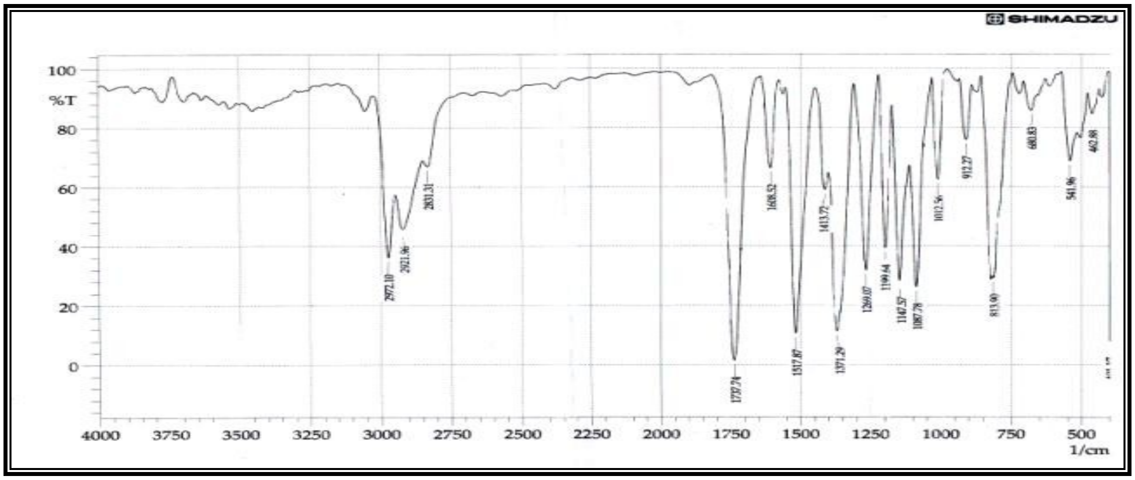
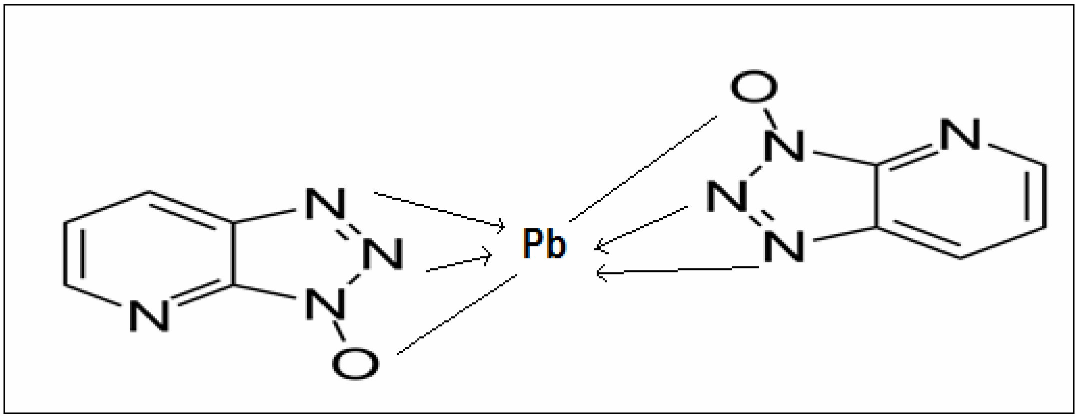


Fig.8 The proposed vacuum Figure of Lead ion complex(II)



Also the melting point of the Lead ion complex(II) and the reagent (1-Hydroxy-7-Azabenzotrizole) were measured, the melting point of a metalloproteinase ion complex was (490 °C) while for the detector one was (180 ° C), referring to an increase in the molecular weight of the complex, resulting from the complexity process face increasing in the melting point. Table (3) shows some chemical characteristics for each of the organic reagent and complex.

A spectral study was conducted such as of UV-visible spectroscopy (Uv-Vis) and infrared spectroscopy FT-IR for both of the organic reagent and complex, the spectroscopy study of the ultraviolet-visible(UV-Uv-Vis) showed absorption spectrum in the ultraviolet - visual region for the metallic ion solution (fig. 5–1) at the wave length of 217 nm and for the organic reagent (1-Hydroxy-7-Azabenzotrizole) dissolving in ethanol (fig. 5–2), and shown absorption peak at the greatest wavelength of 330nm, and showed the absorption spectrum of the complex dissolved in the mentioned solvent the greatest absorption at the wavelength of 548 nm (fig. 5–3), and when comparing the two values red shift is took place of the complex spectrum comparing to the organic reagent spectrum of 145nm and this could be due to obtain a correlation between the metal ion and donor atoms in the reagent which is both of nitrogen and oxygen atom.

The complex have been diagnosed using spectroscopy Infrared IR (fig. 6), and when comparing its spectrum with the organic detector spectrum (fig. 7) we note clear changes, that appeared new packages were not already present before in the spectrum of the detector sometimes or disappearance of some packages or change its shape at other times as indicated in table 4 where we note the frequencies of the most important peaks and the changes that have obtained shows that the frequencies of the hydroxyl group (OH) move away from its position toward the higher frequency at consistency, and the reason of this attributed to break the implicit hydrogen bonds in the detector particle and involve the group in the overall episode of the complex after losing its proton by the effect of metalloproteinase ion and the acidic function of the solution (www.aspera-eu.org/index.php?option=com; http://www. nature.com/news/2010/100415/full/news).

Also the (C = N) pack suffer from change in its shape, that refer to force it in the consistency process (www.pencils.com). Changes got to vibrations of other groups such as (N = N), which are affected by the consistency process as well as the emergence of new packages such as (M-O). In the complex spectrum, we note the presence of a broad package at 3550cm^{-1} is attributed to the presence of water molecule within the complex structure and which confirms this conclusion is the weight difference of amount of the complex before and after the heating process to the degree of 105 °c. (Fig. 6 and 7) confirm the results we have obtained.

Depending on the mentioned above studies, vacuum figure of the formative complex have been proposed as shown in Figure 8.

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