



ISSN (E): 2277- 7695
 ISSN (P): 2349-8242
 NAAS Rating: 5.03
 TPI 2018; 7(3): 656-658
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 www.thepharmajournal.com
 Received: 22-01-2018
 Accepted: 23-02-2018

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Spectrophotometric determination of lead in water samples using salicylaldehyde isonicotinoylhydrazone

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Abstract

A very simple, highly selective and non - extractive Spectrophotometric method for the trace amounts of lead (II) has been developed. Salicylaldehyde Isonicotinoylhydrazone (SAINH) has been proposed as a new analytical reagent for the direct non- extractive Spectrophotometric determination of lead (II). The reagent reacts with lead in an basic medium (pH 8.25, ammonium hydroxide and ammonium chloride buffer) to form a yellow colored 1: 1(M : L) complex. The reaction is instantaneous and the maximum absorption was obtained at 390 nm and remains stable for 3h. The molar absorptivity and sandell's sensitivity were found to be $1.197 \times 10^4 \text{ L mol}^{-1}$. Linear calibration graphs were obtained for 1.0 -9.0 $\mu\text{g/ml}$ of lead (II). The method is highly selective for lead and successfully used for the determination of lead in various water samples.

Keywords: Spectrophotometric determination, salicylaldehyde isonicotinoylhydrazone, alloy and steel samples, molar absorptivity and sandell's sensitivity

Introduction

The lead is not necessary for living and consider as very important pollutant for vital systems [1, 2]. Lead supply to the environment in different ways [3-5]. The single most commercial use of the lead is in the manufacture of lead - acid storage batteries, antifriction metals, solders and type metal. Lead is used for covering cables and as a lining for laboratory sinks, tanks and the "chambers" in the lead chamber process for the manufacture of sulphuric acid. It is extensively used in plumbing. Lead is also employed as protective shielding against X - rays and radiation from nuclear reactors. Unlike other chemicals lead does not vaporize (or) break down over time. As a result lead poisoning occurs very easily through inhalation and ingestion.

Lead interferes with a variety of body processes and is toxic to many organs and tissues including the heart, bones, intestines, kidneys, reproductive and nervous systems. Although lead and most of its compounds are only soluble in water, the use of lead pipe to carry drinking water is dangerous since lead is a cumulative poison that is not excreted from body.

This paper describes the non - extractive Spectrophotometric determination of lead (II) using salicylaldehyde isonicotinoylhydrazone (SAINH) in aqueous medium. In continuation of our ongoing work, we report here the Spectrophotometric determination of lead in various water samples. A close literature survey reveals that SAINH is so far not been employed for the Spectrophotometric determination of lead (II). This method is far more selective, simple and rapid than the existing Spectrophotometric methods. Various Spectrophotometric used for the determination of lead are summarized in Table 1. [6-12].

Table 1: Spectrophotometric methods for the determination of lead (II) with different reagents

Name of the reagent	λ_{RmaxR} (nm)	pH	Beers law(ppm)	Molar absorptivity (ϵ) $\text{L mol}^{-1}\text{cm}^{-1}$	M:L	Ref
Molybdophosphoric acid	430	4.5	0.0-4.8	$0.13 \times 10^4 P$	1:1	6
Phthalaldehyde Dithiosemicarbazone (PADT)	390	7.5-9.0	Up to 4.5	$0.81 \times 10^4 P$	1:1	8
Salicylaldehyde Thiosemicarbazone (SAT)	365	6.5-7.0	0.3-4.6	$0.95 \times 10^4 P$	1:1	9
Salicylaldehyde Isonicotinoylhydrazone (SAINH)	375	9.00	7.5	$1.197 \times 10^4 P$	1:1	presently

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Materials and Methods

Preparation of SAINH: Salicylaldehyde (1.05 ml, 0.01 mol) and ionized (0.69g, 0.01 mol dissolved in 3 ml of ethanol) were mixed in a clean round bottom flask. Suitable quantity of (~10ml) of ethanol was added to the reaction mixture and refluxed with stirring for 2 hrs. A pale yellow colored product was separated out on cooling. It was collected by filtration, washed several times with hot water. This compound was recrystallized from methanol and dried in vacuum. Yield 2.8 gm, melting point is 250 – 252 °C.

Preparation of SAINH solution: It was prepared by taking 0.060 gm of SAINH reagent substance in a 25-ml standard flask. The reagent substance was dissolved in 10 ml of DMF and diluted up to the mark with the same solvent.

Preparation of lead (II) ion solution: Stock solution (0.01 M) of lead was prepared by dissolving 0.33 g of lead nitrate in doubly distilled water and made up to the mark in a 100- ml volumetric flask. A few drops of concentrated sulphuric acid was added before dilution of stock solution. The resulting solution was standardized gravimetrically [13]. Dilute solutions were prepared from this stock solution.

Recommended procedure: An aliquot of the solution containing lead in optimum concentration range, 10 ml of

buffer solution (pH 8.25) and 1 ml of 0.01M reagent solution were combined in 25 – ml volumetric standard flask and resulting solution was diluted to the mark with distilled water. The absorbance of the solution was measured at 390 nm against reagent (SAINH) blank. The measured absorbance was used to compute the amount of lead from predetermined calibration plot.

Preparation of water sample: Each filtered (with whatman No.40) water sample (250 ml) was mixed with 10 ml of concentrated nitric acid in a 500 ml distillation flask. The sample was digested in the presence of an excess potassium permanganate solution according to the method recommended by Fifield *et al.* [14]. The solution was cooled and neutralized with a dilute NHR4ROH solution. The digest was transferred into a 25-ml calibrated flask and diluted up to the mark with deionised water.

Instrumentation: Perkin – Elmer (Lamda 25), UV – Visible spectrophotometer equipped with 1.0 cm (path length) quartz cell and Elico model LI- 610 pH meter were used in the present study.

Results and Discussions

The reagent (SAINH) are easily obtained by condensation reaction and is shown in Fig 1.

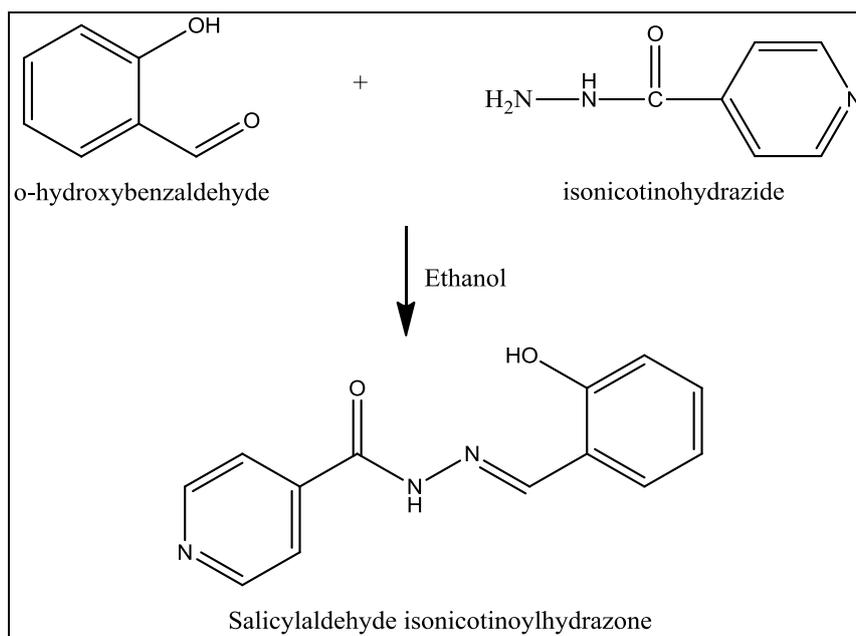


Fig 1: Condensation reaction of SAINH

It has been characterized using IR, NMR and Mass spectral data. The infrared spectrum of SAINH showed bands (cm^{-1}) at 3271(m), 3120(m), 3051(m), 1649(s), 1619(s), 1535(m), 1473(s), 1290 (s), 768(s), 711(s) and 682(m), are respectively assigned to ν (NH) secondary stretching, ν (C-H) stretching, (isoniazid), ν (C-H) aromatic stretching (salicylaldehyde), ν (C=O) isonicotinoyl stretching, ν (N-H) plane bending, ν (C-C) aromatic ring stretching, ν (C-H) asymmetric bending, ν (C-N) stretching vibrations, ν (C-H) aromatic oop bend (salicylaldehyde), and ν (N-N) stretch and ν (C-H) aromatic oop bending respectively.

Applications

The present method was successfully applied for the

determination of lead in various environmental water samples and the results were presented in Table 2.

Table 2: Determination of lead in various water samples.

S. No	Sample	Amount of Lead* found ($\mu\text{g/ml}$)
SAINH method		
1	Tap water	2.1983
2	Sewage water	2.1118
3	R O water	2.0945
4	well water	2.1464
5	River water	2.0945

Conclusion

The synthesized reagent salicylaldehyde isonicotinoyl

hydrazones (SAINH) is characterized by Analytical and spectral studies. The reagent forms a yellow colored complex with lead (II). The Pb (II)-SAINH complex structure is predicted and various physic-chemical and analytical characteristics are determined. This reagent SAINH is successfully used for the determination of lead (II) in various environmental samples.

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