

Determination of lead in river water by Flame Atomic Absorption Spectrometry

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Abstract

Simple method for determination of Pb in natural water by flame atomic absorption spectrometry (FAAS) has suggested. The calibration graph for lead was linear in the range (0.02- 0.1) $\mu\text{g ml}^{-1}$ and the detection limit was $0.4 \times 10^{-3} \mu\text{gL}^{-1}$.

For the determination of microamounts of lead in natural water, an extraction technique was developed. Lead was retained as Pb-dithizone complex and extracted into carbon tetrachloride. The preconcentration, liquid – liquid extraction, optimal conditions for the extraction of lead complex were determined. The FAAS method was applied to the determination of Pb in natural water from different areas of Missan province. The obtained results demonstrated that the procedure could be successfully applied for the analysis of water samples with satisfactory accuracy.

Keywords: FAAS, dithizone, liquid- liquid extraction.

Introduction

Water pollutants mainly consist of heavy metals, microorganism, fertilizer and thousand of toxic organic compounds (WHO, 1999). Lead is one of the most toxic elements, has an accumulative effect and is an environmental priority pollutant [1].

Lead pollution in water systems has seriously influenced the quality of life, especially in developing country. Lead is accumulated in tissues and may cause serious health problems for humans and animals. This metal inhibits biosynthesis and affects the kidneys, brain cells and the permeability the liver membrane, thereby reducing some of their functions. It can be accumulated in the body and can promote disturbance such as nausea, vomiting, diarrhea, sweating and, in some cases, convulsions, coma and death [2, 3].

The U.S. Environmental Protection Agency (EPA) has classified lead as a Group B2 (probable) human carcinogen [4]. The environmental and health problems arise fundamentally from the use of gasoline antiknock products and paint pigments [5]. Nevertheless, it is used as a raw material in the manufacturing industry such as automotive batteries, ceramic and ink [6]. The World Health Organization (WHO) has established the maximum allowable limit of $10 \mu\text{g L}^{-1}$ for lead in drinking water. It is, therefore, important to monitor the lead level in the environmental samples [7].

The most common analytical methods for the lead trace determination are the flame atomic absorption spectrometry (FAAS) [8, 9] the electrothermal atomic absorption spectrometry (ET AAS) [10-12] and the inductivity coupled plasma emission spectrometry (ICP) [13]. Of the three aforementioned methods, ET AAS is the most sensitive technique with a detection limit in the sub-picogram range for most

metals [14]. More recently, the inductively coupled plasma-mass spectrometry (ICP-MS)[15] has produced a detection limit in the same range with (ETAAS)[16]. Nevertheless, the detection of metal trace elements in aqueous samples is difficult due to various factors, particularly their low concentration and the matrix effects [1]. Precocentration and separation techniques, such as liquid-liquid extraction [17], ion exchange [18], co-precipitation[19] and solid phase extraction (SPE)[20, 21].

Flame atomic absorption spectrometry (FAAS) is a more frequently used technique due to its simplicity and lower cost, although this method has a limited sensitivity for lead so a preconcentration step is often required to improve the detection limit[22-24].

The aim of the present study was to propose an extraction procedure for the preconcentration of lead in natural water samples, determined by FAAS.

The experimental conditions for the extraction of the dithizone complex of lead into carbon tetrachloride (extraction time, amount of extractant, pH of the aqueous solution, etc.) were optimized. This paper also reports the application of the proposed method to the determination of Pb in river, well, marshes and tap water samples from different areas of Missan province.

Experimental

Reagents and solutions

The lead stock solution ($1000 \mu\text{g ml}^{-1}$) was prepared by dissolving 0.1598 g of Pb (NO_3) (Thomas Baker) in 1% (v/v) nitric acid solution in a 100 ml volumetric flask. The working solutions were prepared daily by serial dilutions of the stock solution prior to analysis. Dithizone reagent was prepared by dissolving 0.005 g in 100 ml CCl_4 . To determine the influence of pH, solution of 2 M HCl or NH_3 (1:1) were added to the aqueous solution samples. Deionized water was used throughout the experiments.

Instrumentation.

All measurements were carried out with an AI 1200 Flame Atomic Absorption Spectrometer (Aurora Instruments Ltd., Canada). The instrumental parameters were adjusted according to the manufacturer's recommendations. A lead hollow cathode lamp was used as a radiation source, operated at 5 mA with a slit width 0.2 nm, the wavelength was set at 217 nm resonance line. The air-acetylene flame was used, fuel flow rate was 1.5 Lmin.^{-1} pH meter (HANNA instruments pH-211).

Procedure

A calibration curve for determination of lead in natural water by FAAS method was established by using the following procedure. First, an appropriate amount of working standard solutions of lead was transferred to an extraction tube, then, 1 ml of 0.005% dithizone was added, and the obtained solutions were extracted for 120 sec. with carbon tetrachloride. Finally, after removing the organic solvent by slow evaporation, the residue was dissolved in 10 ml of NH_3 (1:1), transferred to 100 ml volumetric flask and filled to the mark with deionized water.

For the determination of lead in natural water samples, the analyzed sample (100 ml) was transferred into an extraction tube and the same preconcentration procedure was followed.

Sampling

The water samples were collected from different regions of Missan province during Mar. 2013, in clean and dry plastic bottles 1.5 L, filtered by filter paper (Munktell Filter AB, Grade 292),

5 drops of HNO₃ Conc. 69% (Applichem) were added for each bottle. The samples were kept in Refregrator at 4°C before the analysis.

Results and discussion

In the present study, an extraction procedure for the concentration of the analyte has been suggested. To demonstrate the suitability of the proposed method, the conditions for the extraction of the Pb-dithizone complex with carbon tetrachloride have been investigated.

The extraction procedure involves transfer of complex through the aqueous/organic phase interface, the efficiency of this procedure depends on the contact time, the effect of extraction time on the analytical signal of the Pb was considers estimated (Fig.1).As can be seen, the absorbance is stable after 120 Sec. Consequently, this time could be considered as the time necessary for complete extraction.

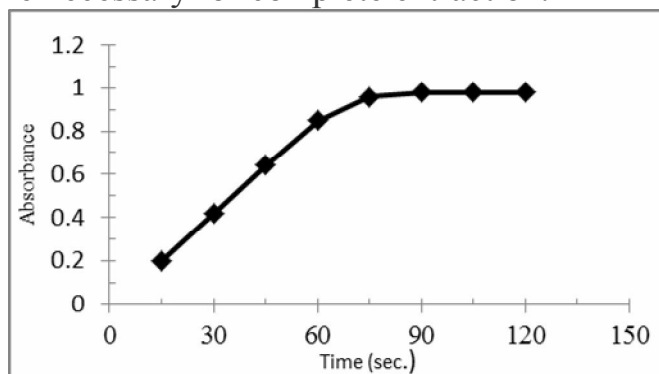


Fig.1 Influence of the extraction time on the analytical signal of Pb. Preconcentration step: 0.02 $\mu\text{g ml}^{-1}$ Pb, pH = 9.5, 10 ml CCl₄

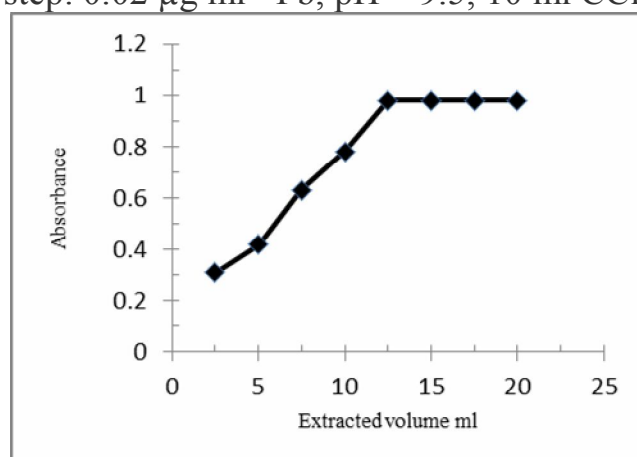


Fig.2 Influence of the extractant volume on the analytical signal of Pb. Preconcentration step: 0.02 $\mu\text{g ml}^{-1}$ Pb, pH= 9.5, 120 sec. extraction time.

The effect of the extractant volume on the analytical responses is shown in Fig.2. A variation of the analytical signal as a function of the extractant volume is evident. From these results, 10 ml of CCl₄ was chosen as the optimum volume.

In order to optimize the preconcentration conditions, the extraction was also performed at different pH values of the aqueous solution. Fig.3 shows that the optimal pH values is in the range (8 – 11.5), the results are understandable since a better complexation occurs in this pH range.

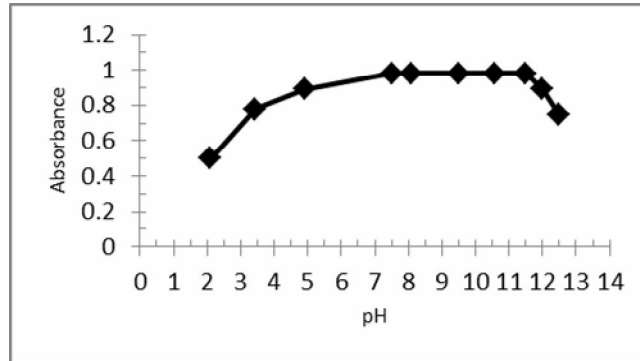


Fig.3 Influence of the pH of the aqueous solution on the analytical signal of Pb.
Preconcentration step: $0.02 \mu\text{g ml}^{-1}$ Pb, 120 sec. extraction time, 10 ml CCl_4 .

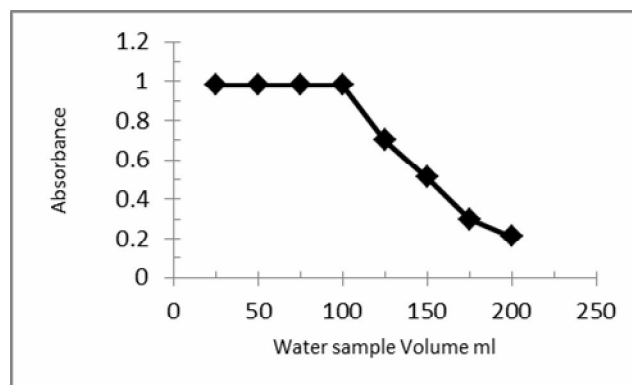


Fig.4 Influence of the water sample volume on the analytical signal of Pb.
Preconcentration step: $0.02 \mu\text{g ml}^{-1}$ Pb, pH = 9.5, 120 sec. extraction time, 10 ml CCl_4 .

The recovery of heavy metals when extracted as different complexes from water samples in some cases depends on the total volume of the analyzed sample [25].

Therefore the limiting amount of sample, which would still be suitable for quantitative extraction, is a very important parameter. It can be seen from Fig.4 that there is no significant effect on the analytical response for sample volume up to 100 ml.

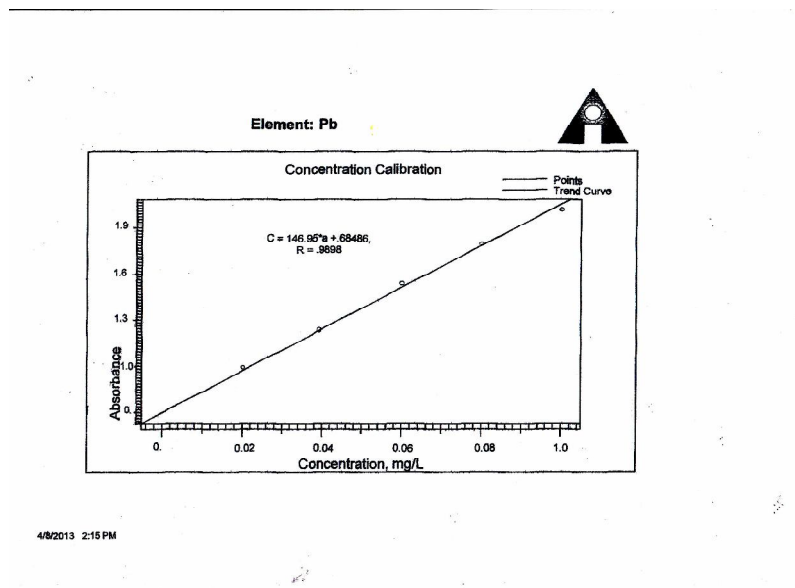


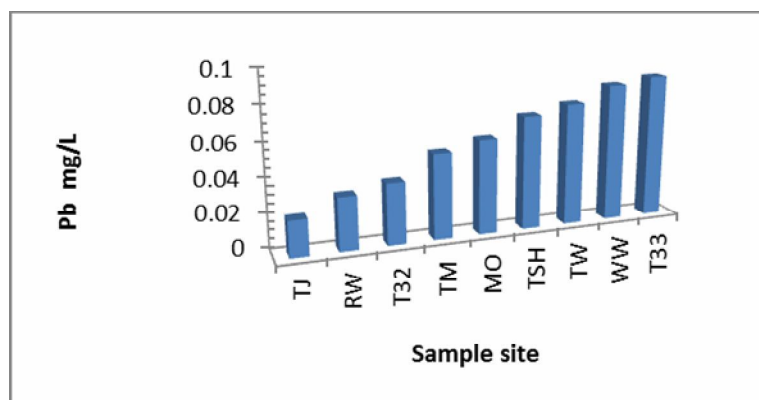
Fig.6 The calibration curve of Pb, obtained by FAAS

Table1. Results obtained for determination of lead in water samples

Ser.	Sample	Lead* concentration determined by FAAS / mgL ⁻¹
1.	TSH ¹ site	0.067 ± 0.011
2.	TM ² site	0.050 ± 0.020
3.	T33 ³ site	0.085 ± 0.003
4.	TJ ⁴ site	0.021 ± 0.002
5.	T32 ⁵ site	0.036 ± 0.010
6.	Tap water	0.072 ± 0.001
7.	Rain water	0.031 ± 0.014
8.	Well water	0.081 ± 0.015
9.	MO	0.056 ± 0.007

*Mean ± SD

TSH¹ Al-musherh sample collection station of Missan environmental directorate.
 TM² Al-meymonah sample collection station of Missan environmental directorate.
 TJ⁴ Al-meger sample collection station of Missan environmental directorate.
 T33³ Al-uzer sample collection station of Missan environmental directorate.
 T32⁵ Al-Kalah sample collection station of Missan environmental directorate.
 MO Al-Oudah marshes sample collection station of Missan environmental directorate.



CONCLUSIONS

A procedure for determination trace amount of Pb in natural water is described. The method combines atomic absorption spectrometry with preconcentration of analyte by liquid-liquid extraction. The complexation of lead with dithizone was efficient enabling its preconcentrate from water. The determination of lead in natural water samples were selected from different areas of Missan province.

In addition, the analytical costs of the proposed extraction method are low, it is also simple, quick and the operation techniques are fairly easy. Thus, the proposed preconcentration methodology offers a simple and sensitive alternative to other preconcentration technique. This proposed procedure for determination of lead can be suitable for routine application for water analysis.

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تقدير الرصاص في ماء النهر بمطيافية الأمتصاص الذري اللهبى

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الخلاصة:

طريقة بسيطة مقترحة لتقدير الرصاص في المياه الطبيعية بمطيافية الأمتصاص الذري اللهبى. منحنى المعايرة للرصاص كان خطياً" ضمن المدى (0.02 – 0.1) $\mu\text{g/ml}$ وحده الكشف كان $0.4 \times 10^{-3} \mu\text{g/ml}$. طورت تقنية معينة في الأستخلاص لتقدير الكميات المايكروية للرصاص في المياه الطبيعية حيث يرتبط الرصاص على هيئة معقد Pb- دايتايزون ثم استخلص في رابع كلوريد الكربون. قدرت الظروف المثلى للأستخلاص بالتركيز المسبق بطريقة الأستخلاص سائل- سائل ثم استخدمت مطيافية الأمتصاص الذري اللهبى لتقدير الرصاص في المياه الطبيعية ومن مناطق مختلفة لمحافظة ميسان. اوضحت النتائج إمكانية تطبيق الطريقة في تحليل نماذج مختلفة من الماء وبقناعة دقيقة.

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