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Determination of Trace Amount of Lead in Potable Water by Flow Injection Flame Atomic Absorption Spectrometry Aided by a Pre-Concentration Micro Column

I.S. Bushina* H.M. Kut* and Seham S. Elmahmudi*

تحديد المقادير الضئيلة لعنصر الرصاص في مياه الشرب بواسطة جهاز لهب طيف الامتصاص الذرّي باستخدام طريقة التركيز المبدئي على العمود المصغر

إسماعيل بشينة، حسن كت و سهام المحمودي

لقد تم تحديد عنصر الرصاص لعينات مختارة من مياه الشرب بمدينة طرابلس وذلك باستخدام طريقة التركيز المباشر بواسطة عمود مبادل أيوني يحتوي على الألومينا المنشّطة.

حقنت المحاليل القياسية وكذلك العينات باستخدام منظومة الحقن المستمر بجهاز الامتصاص الذري. جمعت عينات المياه من مناطق مختلفة، حيث تم تعديل رقمها الهيدروجيني عند 4.5، وقيس تركيزها عند امرار محاليلها بمعدل 3.4 سم^3/دقيقة واستعمل حمض النيتريك (2M) لاخراج الرصاص.

وتشير النتائج إلى أن عينات المياه المدروسة تحتوي على 0.2–0.8 ميكروجرام/لتر، وكان الانحراف العياري النسبي 0.5%.

Abstract *Lead is determined by using on line pre-concentration micro column of activated alumina and a flow-injection atomic absorption spectrophotometer. Samples of groundwater were collected from different locations around Tripoli. Results were obtained for a flow rate of 3.4 ml/min. Nitric acid concentration for eluting lead was 2 M. Ammonia solution was used as a carrier at a concentration of 0.15 M. Samples and standards were injected at pH 4.5 using an injection loop of 1000 ul. Lead was determined at a range of 0.2–8.1 μg/l with RSD 0.5%.*

INTRODUCTION

The presence in water of toxic heavy metals such as lead, cadmium, and mercury has been of concern for many years. Major sources of lead pollution are industrial processes, such as lead smelting, and lead-containing industrial products, such as leaded motor gasoline, paints and water transporting pipes. Although the adverse health effects caused by lead have not been fully substantiated, most governments and environmental protection agencies around the world take precautionary measures against it, because it is believed that this element disrupts some of the normal reactions, occurring in the living body, when exceeding a certain level, causing damage of the nervous system and toxictiy [1]. Thus there is vital necessity for accurate lead determination.

Flame Atomic Absorption Spectrophotometry (FAAS) is the most commonly used technique for trace metal determination; it is easy to use and a lot of data base of information is now available. Although FAAS has been successfully used for lead determination, it has a limitation, that it is not sensitive enough for low level lead concentration.

Graphite furnace and hydride generation can offer this sensitivity but it needs skilled operators, it is of low precision and suffers from matrix inter-

*Petroleum Research Centre P.O. Box 6431 Tripoli, Libya, G.S.P.L.A.J.

ference [2]. Duel silica type atom trapping [3] can provide enough sensitivity at the level of μg/l for the determination of lead, but this technique requires a highly-skilled operators. Therefore the determination of trace elements below routine detection limit, using available equipment, becomes necessary.

Flow injection, flame atomic absorption analysis for trace elements has now been successfully developed for on line pre-concentration [4] procedure for many samples due to its improvement of sensitivity and reduction of analysis time. This technique, using a micro column of Chelex 100 ion exchanger, has been demonstrated by Olsen *et al.*, [5] in the determination of heavy metals in sea water. The potential of flow injection analysis (FIA) for rapid procedures using traditional ion exchangers have been studied for the determination of many elements [2, 6].

In this study on line pre-concentration by basic activated alumina in conjunction with flow anaylsis system was used for the evaluation of lead content in groundwater used for drinking purposes. The simplicity of the method and versatility of activated alumina in its basic form as on line pre-concentration column gives a well accepted system because it does not require major expenses for the determination of lead at trace level (μg/l), when a graphite furnace is not available.

EXPERIMENTAL

Sample Pre-treatment

Water samples, collected at the source, were immediately acidified with 2 M nitric acid and were then kept in polyethylene containers before conducting the analysis.

Materials and Reagents

All chemicals used were of high purity analytical grade. Nitric acid and ammonia solutions were obtained from (BDH) chemicals, Poole England. Standard solutions were prepared daily from stock standard solution supplied by VHG Lab. Tartaric acid (0.5 M), used as a complexing agent, was obtained from Chadwell Health, Essex, England. Aluminum oxide, activated, basic, 95+% (Aldrich Chemicals Co. Ltd.) and further activation [7] was made before it was used for packing the column. The PTFE (6 cm long, 3 mm i.d) mini column was packed with alumina (140 mesh). Water of high purity was used throughout the experimental work. Buffer solution (Ingold, Bestell. Nr. 9863) was used for calibration of pH metre.

Instrumentation and Apparatus

Atomic Absorption Spectrophotometer (Varian AA 1475) equipped with Epson printer, and lead hollow cathode lamp, was used in the determination of lead concentration. Knick Digital pH Meter 646 was used in our work for the adjustment of the pH at 4.5 Peristaltic pump, injection loop was calibrated at 1000 ul. PTFE syringes were used to inject the solutions.

Analytical Procedure

The acidified samples were kept for 24 h prior to analysis. The complexing agent (1 ml/100 ml of tartaric acid 0.5 M) was added.

Standards were adjusted to working pH using aqueous ammonia (2 M) and left 1 h. The alumina micro columns were prepared and flushed with the carrier solution (0.15 M NH_3). Water samples were pre-concentrated on the column prior to analysis. Three packed micro columns were used for every watere sample. When FAAS system is working using flame of air – acetylene the nebulizer tube was connected to the flow injection outlet system using a carrier flow rate of 3.4 ml/min. The micro column was connected to the system with injection loop of 1000 ul. Standards containing 10–1500 μg/l were first injected to the basic alumina column using ammonia as a carrier, then after 2 min. lead eluted using 1000 ul of 2 M HNO_3, the signal then recorded.

Micro column of activated alumina contained lead from water samples treated with identical procedure as that for standard solution. Lead was also eluted from the column using 1000 ul HNO_3 of 2 M to the FAAS system where signals were recorded.

RESULTS AND DISCUSSION

Lead II, 217 nm resonance line was used for the determination of lead by FAAS. The effects of various parameters, were determined. Acid concentration, flow rate, and pH were examined by using 1 μg/l ml^{-1} lead solution. The optimum conditions of the experimental work for lead determination in drinking water of Tripoli were thus established. Different concentrations of nitric acid solutions were prepared and were used for eluting lead content from activated alumina micro column. Fig. 1. represents the variation of the peak height response with nitric acid concentration.

Maximum absorbance of lead was obtained at a concentration of acid of 2 M, whereas below this concentration the absorbance is decreased by de-

creasing the concentration.

The maximum release of lead from the activated alumina surface being at acid concentration of 2 M may indicated either a shift to acid catalyzed alumina, which retains lead or a chemsorption between lead and alumina is taking place at acid concentration higher than 2 M. In both cases lead release will be reduced.

The effect of the flow rate of the carrier stream has been optimized. Fig. 2 depicts the flow rate of the carrier stream versus lead absorbance. Clearly the peak height response was found to increase very little when flow rate was extrapolated to 5 ml/min. The value of 3.4 ml/min. was thus selected as the operating flow rate for lead measurements.

0.15 M ammonia solution served as the carrier stream maintained the column basicity. Fig. 3. illustrates the dependence of absorbance on pH. It is evident that pH 4.5 is effective in accumulating lead on the alumina column. The calibration graph at the optimum conditions including flow rate, acid and ammonia concentrations showed a good linearity with the value of regression coefficient of 0.99963. The relative standard deviation ($n = 3$) at 100 ppb was found to be 0.5%.

Results of analyzed drinking groundwater samples from different localities in Tripoli area are summarised in Table 1.

It is quite clear from the table that the maximum amount of lead was found to be in Ghot ash Shaal area (8.13 μgl^{-1}). The value in that area is higher that the others probably because the water distribution pipe network is very old, extensively corroded and situated in a highly populated area of very heavy traffic. Contamination of water with polluted soil should not be ruled out.

Table 1. Present lead determination and associated standard deviation in drinking water from selected locations in Tripoli city

Sample name	*Lead concentration* (μgl^{-1})	*Sample name*	*Lead concentration* (μgl^{-1})
Hai 2 March	0.45 ± 0.03	Sidi El masry	0.75 ± 0.03
Zagouny	0.33 ± 0.02	Milk Factory*	0.60 ± 0.04
Petroleum Institute	0.80 ± 0.02	Zawit El mazy*	0.50 ± 0.02
Hai al Andalus*	0.60 ± 0.02	Mawashi Company*	2.10 ± 0.05
Ghot ash Shaal*	8.13 ± 0.04	Al Gasi mosque	2.50 ± 0.08

*No of replicate "n"= 4, otherwise No. $n = 3$

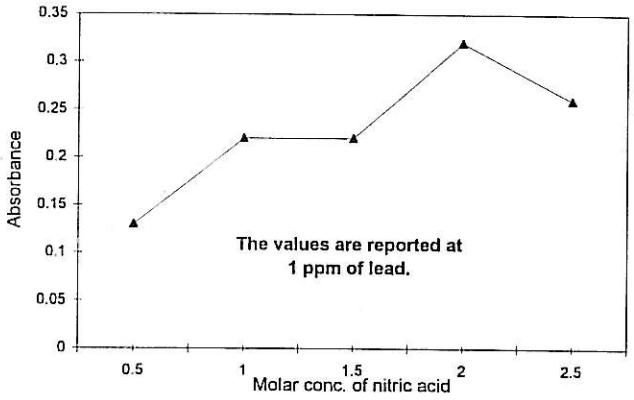


Fig. 1. Variation of absorbance with nitric acid concentration.

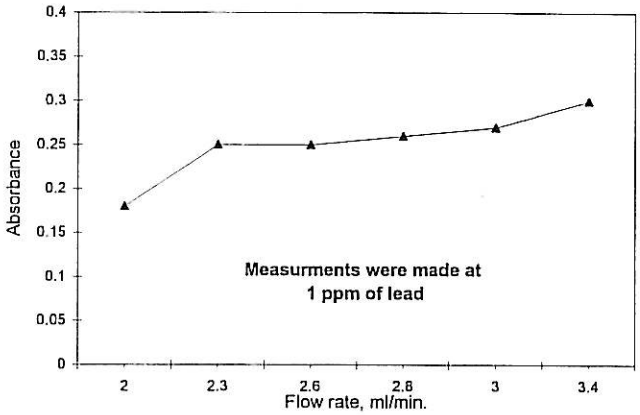


Fig. 2. Dependance of absorbance on flow rate.

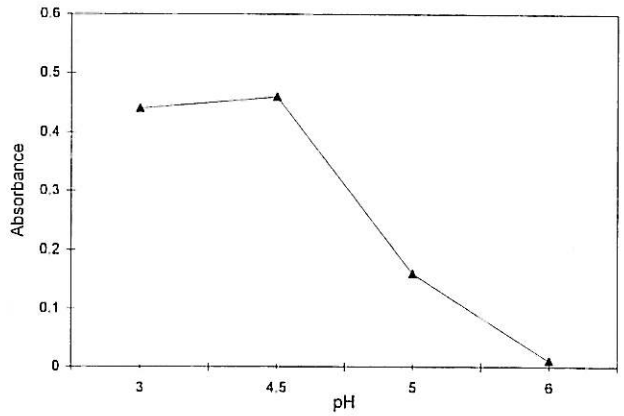


Fig. 3. Dependance of absorbance on pH.

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