Concentration of Pb (II) by adsorption and its atomic absorption spectrometric determination

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ABSTRACT: A sorption concentration method using impregnated silica has been developed to determine small concentration of lead in water by Atomic Absorption Spectrometry. KEYWORD: Adsorption; Atomic Absorption Spectrometry; heavy metals.

Introduction

Recently, water contamination by heavy metals is gaining special importance, due to the great use of these substances in various industrial fields and their potential possibilities to severely affect the chemical status of the environment.1

It is known that lead affects human being and it is considered highly toxic to children. 5 Therefore the presence of lead in the environment requires systematic control. As a result, the control methods should be effective and attainable to know exactly the chemical status of the medium with a great tendency to widen the lower concentration limits at which an element is detectable.

Reports have been made2,3 in which the determination limits do not exceed 20 micrograms per litre using flame atomic absorption spectrometry, and diverse forms of preconcentration.

However, the best results with respect to lower limits have been obtained by preconcentration with organic solvents together with the atomic absorption spectrometric determination with graphite furnace3,4 taking as limiting factors the time for preparation, the methodology of work, and the cost of the installation.

In our work we propose a combined method of preconcentration by adsorption and subsequent
determination by atomic absorption spectrometry that permits the quantification of lead in water in quantities less than 20 micrograms per litre without great difficulties.

**Materials and methods**

These experiences were undertaken at 300 ± 2 K using Merck standard silica reagent with an iron content less than 0.03 %, with particle diameter between 0.063-0.2 mm, and sodium sulphide, zinc chloride, lead nitrate, nitric acid and hydrochloric acid chemically pure. Lead free bidistilled water was used to prepare the corresponding solutions.

The concentration of lead was undertaken in a column 2 cm length and 3 mm diameter in which 0.07 g of silica impregnated with zinc sulphide was introduced. The volume passed through the column was between 0.5-1.0 L depending on the concentration of lead in de solutions. The adsorbate-adsorbent complex obtained was treated with hot diluted nitric acid solution and water, was added to complete a volume of 10 ml, the concentration of lead was determined in a AAS-3 Carl Zeiss spectrometer at a wavelength of 217,0 nm and a determination limit of 0.1 mg/L.

**Result and discussion**

Before using the adsorbent for capturing lead in water, its dynamic adsorption capacity was 21 mg/g.

The weight of lead that is adsorbed is calculated by the difference between the initial concentration and the sum of all the weights collected and determined in the filtrated samples. This result was compared with the lead amount contained in the adsorbent which was determined as explained above the difference found was not greater than 2%.

Parallel test were made to determine the percentage and the order of the concentration. Results are given in Table 1.

| C (Pb²⁺) in the solution (mg/L) | Filtrated volume (ml) | Concentration order | % of concentration |
|--------------------------------|-----------------------|---------------------|-------------------|
| 2.1                            | 500                   | 5.10^3              | 96.4              |
| 2.1                            | 1000                  | 1.4.10^4            | 96.4              |

The effectiveness of the adsorbent from the analytical point of view was later determined, model solutions with known concentrations of lead were passed through adsorbent and the quantity of lead remove was determined in the concentrate by AAS and it was compared with the concentration of the initial solutions.

Result are shown in Table 2.
Based on the results obtained, the concentration of lead in samples of running water was determined. The samples were taken at intervals of 30 min during 4 hrs. and homogenized, the necessary portion for analysis was taken.

The results are shown in Table 3.

| Filtered volume (ml) | Concentration mg/L   |
|----------------------|----------------------|
| 1000                 | 0.050 ± 0.005        |
| 900                  | 0.010 ± 0.001        |

It was observed that one of the samples surpassed the permissible levels for this type of water.

**Conclusions**

It is concluded that the concentration method can be applied to determine small concentrations of lead by employing silica gel impregnated with ZnS, which makes the determination of lead possible from 10 microgram per liter and even less. The method could also be used to analyze small volume of sample, previously taken and filtered through the adsorbent in field conditions.

SEIJÓ ECHEVARRÍA, M., DEL TÓRO DÉNIZ, R., MARTINÉZ SÁEZ, S., ROSELL JUAN, F. Concentração de Pb (II) por adsorção e sua determinação por espectrometria de absorção atômica. *Ecl. Quim. (São Paulo)*, v.22, p.199-203, 1997.

**RESUMO:** Foi desenvolvido um método de concentração de sorção usando sílica impregnada, para determinar pequenas concentrações de chumbo em água através de espectroscopia atômica. **PALAVRAS-CHAVE:** Adsorção; espectroscopia de absorção atômica; metais pesados.
References

1 GABOURY, B. Clean technique measurement of Pb, Ag, and Cd in freshwater: A redefinition of metal pollution. *Environ. Sci. Technol.*, v.28, n.11, p.1987, 1994. [Links]

2 GARBARINO, J. R., STEINHEINER, T. R., TAYLOR, H. E. Water analysis. *Anal. Chem.*, v.57, p.46, 1985. [Links]

3 KANE, J. R. The determination of selected elements in coal ash by A.A.S. *US Geol. Surv. Bull.*, n.1823, p.47, 1989. [Links]

4 OBIOLS J., DEVESAA R., PEIRO L., SALAYET, J. Speciation of heavy metals. I: Concepts and techniques. *Rev. Afinidad*, n.408, p.93, 1987. [Links]

5 TAVARES, T. Ecological studies of the reconcave, Bahía, Brazil (1976 until 1990). *Int. J. Environ. Pollut.*, n.1, p.33, 1991. [Links]

 RECEBIDO EM 17.5.1996.
 ACEITO EM 12.9.1996.

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