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Talanta

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# Sequential determination of lead and cobalt in tap water and foods samples by fluorescence <sup>☆</sup>



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## ARTICLE INFO

### Article history:

Received 14 March 2014

Received in revised form

7 April 2014

Accepted 16 April 2014

Available online 24 April 2014

### Keywords:

Lead

Cobalt

8-Hydroxyquinoline

Luminescence

Tap water

Foods

## ABSTRACT

In this work, a new procedure was developed for the separation and preconcentration of lead(II) and cobalt(II) in several water and foods samples. Complexes of metal ions with 8-hydroxyquinoline (8-HQ) were formed in aqueous solution. The proposed methodology is based on the preconcentration/separation of Pb(II) by solid-phase extraction using paper filter, followed by spectrofluorimetric determination of both metals, on the solid support and the filtered aqueous solution, respectively. The solid surface fluorescence determination was carried out at  $\lambda_{em}=455$  nm ( $\lambda_{ex}=385$  nm) for Pb(II)-8-HQ complex and the fluorescence of Co(II)-8-HQ was determined in aqueous solution using  $\lambda_{em}=355$  nm ( $\lambda_{ex}=225$  nm). The calibration graphs are linear in the range  $0.14\text{--}8.03 \times 10^4 \mu\text{g L}^{-1}$  and  $7.3 \times 10^{-2}\text{--}4.12 \times 10^3 \mu\text{g L}^{-1}$ , for Pb(II) and Co(II), respectively, with a detection limit of  $4.3 \times 10^{-2}$  and  $2.19 \times 10^{-2} \mu\text{g L}^{-1}$  ( $S/N=3$ ). The developed methodology showed good sensitivity and adequate selectivity and it was successfully applied to the determination of trace amounts of lead and cobalt in tap waters belonging to different regions of Argentina and foods samples (milk powder, express coffee, cocoa powder) with satisfactory results. The new methodology was validated by electrothermal atomic absorption spectroscopy with adequate agreement. The proposed methodology represents a novel application of fluorescence to Pb(II) and Co(II) quantification with sensitivity and accuracy similar to atomic spectroscopies.

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## 1. Introduction

Today it is well known that many diseases have an environmental origin, considering environmental everything about life: air, water, food, habits, solar radiation and pharmaceuticals, among others [1]. The knowledge of the harmful compounds to human health present in water and foods is of interest to scientists, environmentalist, governments and general public. The scientific community has the responsibility to provide reliable data with the aim of informing people.

Metals are widespread non-biodegradable chemical contaminant found in the environment because of the anthropogenic activities such as industrial production, fertilizer use and sludge

fertilization. Heavy metal contamination is a known causative of various disorders such as genomic instability, endocrine disruption, neurotoxicity, carcinogenicity, immunological problems and also impaired psycho-social behavior [2,3].

Lead has a high toxicity, product of exposure in the workplace, environmental and household pollution. The main routes of incorporation into the body are the respiratory and digestive, through consumption of contaminated food and water. Chronic poisoning with this metal leads to a condition known as *saturnism*, which is characterized by severe anemia, digestive, cardiovascular, renal and nervous disorders [4,5]. Cobalt, meanwhile, is an essential element for humans. Cobalt deficiency can cause megaloblastic anemia, affects the development of the nervous system and the immune response. On the other hand, high intakes can cause polycythemia, hypothyroidism, thyroid and heart failure [6,7].

The determination of traces of both metals is of interest in the fields of environmental analysis, process control and clinical toxicological analysis. Also, the development of analytical methodologies with high sensitivity and accuracy is necessary for their quantification.

The determination of metals in foods and tap water is usually associated with a step of preconcentration of the analyte before

<sup>☆</sup>“In memoriam” of Dr. Adriana Masi, prominent researcher, dear colleague and a friend, who passed away prematurely as a consequence of public insecurity, killed by a shot in the head at the door of her house.

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detection, due to low concentrations present in these matrices [8,9]. A variety of procedures for preconcentration of metals, such as solid phase extraction (SPE), liquid–liquid extraction (LLE), coprecipitation and cloud point extraction (CPE) have been developed [10–12].

SPE has become a preferred method for concentrating the analyte prior to its analysis by instrumental techniques. SPE has come to the forefront compared to other preconcentration and/or separation techniques, as it offers several advantages such as flexibility, higher enrichment factors, absence of emulsion, low cost, high speed and simplicity, safety with respect to hazardous samples and more importantly environment friendly [13–15]. Nowadays, investigators are interested in improving the selectivity of the sorbents used in SPE. This objective is particularly important when analyzing complex matrices. Several solid materials as silica gel, exchange resins, aluminum oxide, poly(vinylalcohol), C18 membranes, cyclodextrines, filter papers, Nylon membranes, among others have been successfully employed as supports for SPE [16,17].

SPE followed by ETAAS, ICP-OES or ICP-mass spectrometry are shown to be suitable for analysis of metal traces [18,19]. The application of molecular fluorescence in the determination of traces of lead and cobalt has shown several analytical advantages such as high sensitivity, proper selectivity and wide dynamic range when it is associated to separation/preconcentration step [20].

The aim of present work is to propose an alternative methodology to traditional atomic spectroscopies for both metals monitoring in real samples of tap waters belonging different regions of Argentina and foods using an instrument accessible in control laboratories. Considering economic aspects and operating simplicity, in this opportunity filters papers ribbon blue are proposed as support for Pb(II) and Co(II) complexes retention for their posterior determination by fluorescence.

## 2. Experimental

### 2.1. Reagents

Stock solutions of Pb(II) and Co(II)  $1 \times 10^{-4}$  mol L<sup>-1</sup> were prepared by dilution of 100 µg mL<sup>-1</sup> standard solution plasma-pure (Leeman Labs, Inc.).

A  $1 \times 10^{-2}$  mol L<sup>-1</sup> sodium tetraborate (Mallinckrodt Chemical Works, New York, Los Angeles, St. Louis, USA) solution was prepared, obtaining the desired pH by addition of dilute HClO<sub>4</sub> (Merck) or NaOH (Mallinckrodt Chemical Works) solutions.

Acetic/acetate (Mallinckrodt Chemical Works, New York, Los Angeles, St. Louis, USA) buffer solution  $1 \times 10^{-2}$  mol L<sup>-1</sup> was prepared by dissolution of the appropriate amount of each, in ultrapure water. The pH was adjusted to the desired value, by adding NaOH (Mallinckrodt Chemical Works) solutions using a pHmeter (Orion Expandable Ion Analyzer, Orion Research, Cambridge, MA, USA) Model EA 940.

Stock solution of 8-hydroxyquinoline (8-HQ, H.E – Daniel Ltd., England)  $1 \times 10^{-6}$  mol L<sup>-1</sup> was weekly prepared by dissolving the appropriate amount of reagent in ultrapure water. The stability of solutions was checked by spectrophotometric measurements.

All used glass materials were previously washed with a 10% v/v HNO<sub>3</sub> solution and then with Milli-Q ultrapure water. All used reagent were analytical grade.

Nylon membranes (Millipore, Sao Paulo, Brazil) 0.45 µm pore size and 47 mm diameter, cellulose acetate (Whatman, England) 0.45 µm pore size and 47 mm, mixed esters (Schleicher & Schuell, Germany) 0.45 µm pore size and 47 mm, Immobilon (+) (Millipore, Sao Paulo, Brazil), Filter papers Blue Ribbon (FP, Whatman,

England) 2–5 µm pore size and 12.5 cm diameter were used in sorption studies.

### 2.2. Apparatus

Spectrofluorimetric measurements were made using a Shimadzu RF-5301 PC spectrofluorometer equipped with a 150 W Xenon lamp and 1.00 cm quartz cells. For solid surface fluorescence measurements a solid sample holder was used. A combined glass electrode and a pHmeter (Orion Expandable Ion Analyzer, Orion Research, Cambridge, MA, USA) Model EA 940 were used for pH adjustments.

Measurements were performed with a Shimadzu Model AA-6800 Atomic Absorption Spectrometer (Tokyo, Japan) equipped with a deuterium background corrector, an EX7-GFA electrothermal atomizer and an ASC-6100 autosampler. L'vov graphite tubes (Shimadzu, Tokyo, Japan) were used in all experiments. Lead and cobalt hollow-cathode lamps (Hamamatsu, Photonics K., Japan) was employed as the radiation sources. Wavelengths used were 240.7 (Slit Width: 0.2 nm) for lead and 283.3 nm (Slit Width: 1 nm) for cobalt using a pyrolysis times of 13 and 10 s at 400 and 800 °C and atomization times of 3 s at 2500 °C and 2 s at 2400 °C, respectively.

### 2.3. Sampling procedure

Tap water samples were allowed to run for 10 min, and approximately 1000 mL of each were collected. Tap water samples were processed once they arrived in the laboratory, without previous treatment.

Food samples were acquired in local shops, choosing products manufactured in Argentina. In order to guarantee representative samples, a randomize strategy sampling was used; a total of three recipients of the same brand for each product were acquired. The whole of the contents of each product was homogenized and reserved for sample preparation.

About 0.5 g of each solid sample (milk powder, express coffee, cocoa powder) were digested using a microwave furnace. Then resulting solutions were transferred to 100 mL volumetric flasks and made to the top with bidistilled water. Portions of 100 µL of each sample were put into 100 mL volumetric flasks and diluted with bidistilled water. These solutions were reserved to Pb(II) and Co(II) determinations by general procedure.

### 2.4. General procedure

Adequate volumes of sample/standard solutions containing Pb(II) and Co(II) ( $0.14\text{--}8.03 \times 10^4$  µg L<sup>-1</sup> and  $7.3 \times 10^{-2}\text{--}4.12 \times 10^3$  µg L<sup>-1</sup>, respectively), 500 µL 8-HQ  $1 \times 10^{-6}$  mol L<sup>-1</sup>, 200 µL buffer sodium tetraborate solution  $1 \times 10^{-3}$  mol L<sup>-1</sup> (pH=10.0) were placed into a 10 mL volumetric flask. The whole mixture was made to 10 mL with ultrapure water. Systems were filtered across the membranes of filters paper (FPs), using a vacuum pump and dried at room temperature. The filtrated solution containing Co(II)-8-HQ complex was collected and pH was reconditioned by adding 1 mL acetic acid/acetate buffer  $1 \times 10^{-2}$  mol L<sup>-1</sup> (pH=4.0). Pb(II) concentration was determined on FP by solid surface fluorescence (SSF) at  $\lambda_{em}=455$  nm ( $\lambda_{exc}=385$  nm), using a solid sample holder; cobalt concentration was determined in the filtrated solution by fluorescent emission at  $\lambda_{em}=355$  nm ( $\lambda_{exc}=225$  nm) using the conventional quartz cell (see Fig. 1).

### 2.5. Interferences study

Different amounts of ions which may be present in water samples (1/1, 1/10, 1/100 and 1/1000 Pb(II) or Co(II)/interferent

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