



Analytical Methods

Novel method for determination of zinc traces in beverages and water samples by solid surface fluorescence using a conventional quartz cuvette



María Carolina Talio^b, María Gimena Acosta^b, Mariano Acosta^b, Roberto Olsina^{a,b},
Liliana P. Fernández^{a,b,*}

^aÁrea de Química Analítica, Facultad de Química, Bioquímica y Farmacia, Universidad Nacional de San Luis, San Luis, Argentina

^bInstituto de Química de San Luis (INQUISAL-CONICET), Chacabuco y Pedernera, 5700 San Luis, Argentina

ARTICLE INFO

Article history:

Received 6 May 2013

Received in revised form 7 November 2014

Accepted 15 November 2014

Available online 29 November 2014

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

Keywords:

Zinc traces

o-Phenanthroline

Eosin

Solid surface fluorescence

Beverages

Tap water

ABSTRACT

A new method for zinc pre-concentration/separation and determination by molecular fluorescence is proposed. The metal was complexed with *o*-phenanthroline and eosin at pH 7.5 in Tris; a piece of filter paper was used as a solid support and solid fluorescent emission measured using a conventional quartz cuvette. Under optimal conditions, the limits of detection and quantification were 0.36×10^{-3} and $1.29 \times 10^{-3} \mu\text{g L}^{-1}$, respectively, and the linear range from 1.29×10^{-3} to $4.50 \mu\text{g L}^{-1}$. This method showed good sensitivity and selectivity, and it was applied to the determination of zinc in foods and tap water. The absence of filtration reduced the consumption of water and electricity. Additionally, the use of common filter papers makes it a simpler and more rapid alternative to conventional methods, with sensitivity and accuracy similar to atomic spectroscopies using a typical laboratory instrument.

© 2014 Elsevier Ltd. All rights reserved.

1. Introduction

Zinc is a metal with great nutritional importance, necessary in cellular replication and immune response development, with catalytic and regulatory actions on more than 300 enzymes (Seiler, Sigel, & Sigel, 1994, chap. 58; Terres-Martos et al., 2002). Growth retardation, altered immune response, premature birth, weight loss, and anorexia are some of the consequences of zinc deficiency (Gruber & Pista, 2013; Katz et al., 1987). Deficiency can be the result of low dietary intake of bioavailable zinc, but it can also be caused by illness, which impairs food intake (Haase, Overbeck, & Rink, 2008; Salgueiro et al., 2002). On the other hand, a diet with excess zinc can cause disturbances in energy metabolism or an

increase in oxidative stress (Tupe, Tupe, Tarwadi, & Agte, 2010; Yanagisawa et al., 2009).

The recommended zinc intake values are between 5 and 15 mg L^{-1} per day depending on sex and age (NRC, National Research Council, & National Academy of Sciences, 1999). Foods represent the main intake source of zinc as well as tap water. Although tap water should not have toxic levels of zinc, this metal can be present due to corrosion processes in pipes. (Alam & Sadiq, 1989). High levels of zinc associated to other toxic metals have been recently found to be present both in ground and tap water (Almeida, Chan, Pettigrove, Catrall, & Kolev, 2014; Patterson, Pehrsson, & Perry, 2013).

The determination of zinc in foods and tap water is one of the most difficult and complex analytical tasks, especially at trace levels. Different analytical techniques have been applied to the determination of low concentrations of zinc, such as atomic absorption spectrometry with flame (FAAS) (Abdolmohammad-Zadeh & Sadeghi, 2009; Carletto, Roux, Maltez, Martendal, & Carasek, 2008; Zhu, Liang, Zhao, Yan, & Han, 2008) and electrothermal

* Corresponding author at: Área de Química Analítica, Facultad de Química, Bioquímica y Farmacia, Universidad Nacional de San Luis, San Luis, Argentina. Tel.: +54 02664 425385.

E-mail address: lfernand@unsl.edu.ar (L.P. Fernández).

atomization (ETAAS) (Batista et al., 2008; Kiptoo, Ngila, & Silavwe, 2008), inductively coupled plasma optical emission spectrometry (ICP-OES) (Chang et al., 2008; Khajeh, 2009; Salonia, Wuilloud, Gásquez, Olsina, & Martinez, 2000; Suvardhan et al., 2007; Salonia, Cerutti, Martinez, Fernández Turiel, & Gásquez, 2008), and spectrofluorimetry (Haase et al., 2008).

Solid phase extraction (SPE) is an alternative rapid, simple, more environmentally-friendly, and economic pre-concentration step, which has advantages over the traditional liquid-liquid extraction. SPE followed by ETAAS, ICP-OES or ICP-mass spectrometry have been shown to be suitable for analysis of trace metal (Karbasia, Jahanparast, Shamsipur, & Hassan, 2009; Suleiman, Hu, Huang, & Zhang, 2008). Investigators are interested in improving the selectivity of the sorbents used in SPE. Several solid materials, such as silica gel, exchange resins, aluminum oxide, poly(vinylalcohol), C₁₈ membranes, cyclodextrines, filter papers, and nylon membranes, among others, have been employed successfully as solid supports for SPE (Correa & Escandar, 2006; Peralta, Fernández, & Masi, 2010).

The application of molecular fluorescence in zinc determination has shown several analytical advantages, such as sensitivity, selectivity and a wide dynamic range in association with separation/pre-concentration step (Vega, Augusto, Talio, & Fernández, 2011).

The aim of present work was to propose an alternative method for zinc determination in real-world samples, using instruments accessible in quality control laboratories. Considering economic aspects and operating simplicity, filter papers were used as a solid support for Zn(II)-complex retention for determination by solid surface fluorescence (SSF). A conventional 10 mm cuvette adapted to SSF was used for the measurement step.

2. Experimental

2.1. Reagents

Blue Ribbon Filter papers (Whatman, UK) of 2–5 µm pore size and 12.5 cm diameter were used in retention studies.

Stock solutions of Zn(II) 6.54 mg L⁻¹ were prepared by dilution of 100 µg mL⁻¹ standard solution plasma-pure (Leeman Labs, Inc., Hudson, NH, USA).

Tris-(hydroxymethyl)-aminomethane (Mallinckrodt Chemical Works, St. Louis, USA – 1 mmol L⁻¹), sodium tetraborate (Merck & Co., Inc., – 1 mmol L⁻¹), potassium phthalate acid (Merck & Co., Inc., – 1 mmol L⁻¹) and acetic acid/acetate (Mallinckrodt Chemical Works) buffer solutions were prepared.

The desired pH was obtained by adding HClO₄ (Merck & Co., Inc., Darmstadt, Germany) or NaOH, with the assistance of a pH meter (Orion Expandable Ion Analyzer Model EA 940, Orion Research, Cambridge MA, USA).

Stock solutions of eosin (H.E Daniel Ltd., UK – 1 × 10⁻³ mmol L⁻¹) and o-phenanthroline (Merck & Co., Inc., – 1 × 10⁻² mmol L⁻¹) were prepared weekly by dissolving the appropriate amount of each reagent in ultrapure water. The stability of the solutions was checked using a spectrophotometer. All glass materials were previously rinsed with a 10% (v/v) HNO₃, and then with Millie-Q ultrapure water. All reagents were analytical grade.

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 adapted to SSF.

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. A zinc hollow-cathode lamp (Hamamatsu, Photonics K., Japan) was employed as the radiation source. The wavelength used was 213.9 nm using a pyrolysis time of 13 s at 450 °C, and an atomization time of 3 s at 1800 °C.

A Milestone STARTD microwave furnace (Italy) and Milestone polytetrafluoroethylene (PTFE) reactors hermetically sealed (internal volume 100 mL and 1 cm wall thickness) were used for microwave digestion.

2.3. Sampling procedure and sample preparation

Tap water samples (1000 mL), from the locations specified in Table 4, were collected in sampling bottles, after being allowed to run for 10 min, and transported to the lab in refrigerated boxes. Tap water samples were processed once they arrived in the laboratory, without previous treatment.

Food samples (beverages) were purchased from supermarkets popular with consumers and typically sold throughout Argentina. Samples were selected taking into account the main products consumed by segments of the population with different dietary requirements due to their age and lifestyle. In order to guarantee representative samples, a randomize strategy sampling was used; a total of three examples of the same brand for each product were acquired. Entire products were homogenized and reserved for sample preparation.

About 0.5 g of each solid sample (milk powder, express coffee, cocoa powder) was digested using a microwave furnace. Then resulting solutions were transferred to 100 mL volumetric flask and made to volume with Millie-Q ultrapure water. Portions of 100 µL of each sample were put into 100 mL volumetric flask and diluted with Millie-Q ultrapure water (*n* = 4). These solutions were kept for Zn(II) determination.

Beverages (cola drink, instant coffee, energy and hydrating drinks) were diluted and analyzed for Zn(II) directly. Solid and liquid samples were analyzed using the same method subsequently.

2.4. Analysis

Sample and standards Zn(II) (1.29 × 10⁻³ to 4.50 µg L⁻¹, pH7.5) and 1 mL methanol were put in a crystallizer flask, and the mixture diluted to 10 mL with ultrapure water. Pieces of Blue Ribbon filter paper (1 × 3 cm) were impregnated through contact with each solution for 1 min (*n* = 4). The filter papers were dried at room temperature and kept in a dried ambient (20 °C–25 °C) atmosphere until analysis. Sample filter paper were arranged in a conventional quartz cell, adapted for solid support, and SSF determined at λ_{em} = 440 nm (emission), using λ_{ext} = 370 nm (excitation).

2.5. Interferences study

Different amounts of zinc, which may be present in water samples (1/1, 1/10, 1/100 and 1/1000 Zn(II)/interference ratio) were added to a test solution containing 32.6 × 10⁻³ µg L⁻¹ Zn(II), and analyzed.

2.6. Accuracy study

Tap water samples were spiked with increasing amounts of Zn(II) (1.29 × 10⁻³ to 4.50 µg L⁻¹) and analyzed using the proposed SSF method.

Download English Version:

<https://daneshyari.com/en/article/7593906>

Download Persian Version:

<https://daneshyari.com/article/7593906>

[Daneshyari.com](https://daneshyari.com)