



## Determination of some metal ions in various meat and baby food samples by atomic spectrometry



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### ABSTRACT

In this paper, we report a simple and rapid solid phase extraction system for the separation/preconcentration and determination of Cd(II), Co(II), Cu(II), Fe(III), Cr(III), Pb(II), and Zn(II) ions by flame atomic absorption spectrometry (FAAS). This method is based upon the retention of metal ions on a column packed with poly[N-(3-methyl-1*H*-indole-1-yl)]-2-methacrylamide-co-2-acrylamido-2-methyl-1-propane sulphonic acid-co divinylbenzene] (MMAD) resin as a solid-phase extraction (SPE) sorbent at pH 8. At the optimized conditions, the limits of detection (3s/b) between 0.12 and 1.6 µg L<sup>-1</sup>, preconcentration factor of 100, and the relative standard deviation of ≤1.8% were achieved (*n* = 10). The accuracy of the method was verified by analyzing certified reference materials (CRMs) and performing recovery experiments. The developed method was successfully applied to the various natural water, meat products and baby food samples. The recoveries of analyte ions were found in added real samples and CRMs from 95% to 102%.

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### 1. Introduction

Some metals such as iron, zinc, copper, chromium, cobalt and manganese are essential for human metabolism. Some other metals such as lead, cadmium, nickel and mercury are toxic at certain levels (Onianwa, Adetola, Iwegbue, Ojo, & Tella, 1999). The essential metals play important roles in biological systems. For example iron is an integral fragment for many proteins and enzymes and is a crucial component of proteins involved in oxygen transport, regulation of cell growth and differentiation iron insufficient in human lead to fatigue, decreased immunity and death (Lemos, Santos, David, Maciel, & de Almeida Bezerra, 2008; Saçmacı & Kartal, 2008; Shakerian, Dadfarnia, Shabani, & Rohani, 2008). Chromium is considered to be an essential trace element for the maintenance of effective glucose, lipid and protein metabolism in mammals (Saçmacı & Kartal, 2008; Saçmacı, Kartal, Yılmaz, Saçmacı, & Soykan, 2012). Some of various clinical disorders such as anemia, depression of growth, impaired reproductive performance, heart failure and gastrointestinal disturbances have been associated with a dietary insufficient of copper (Gjerde, Wiederin, Smith, & Mattson, 1993; Kolachi et al., 2011; Kudrin, 2000; Onianwa, Adeyemo, Idowu, & Ogabiela, 2001). Zinc is an essential

part of more than 200 enzymes involved in digestion, metabolism, and reproduction and wound healing. Zinc deficiency also leads to cognitive defects and impairs the immune system (Saçmacı, Kartal, & Saçmacı, 2012a; Saçmacı, Kartal, & Saçmacı, 2012b). Cobalt present in vitamin B12 (cyanocobalamin) because it has been used in the treatment of anemia (<http://www.patient.co.uk/pdf/4372.pdf>).

Toxic elements may be very detrimental even at low concentrations when ingested over a long time (Coco, Monotti, Cozzi, & Adami, 2006). Human exposure to such elements can cause adverse health effects indicating toxic property leading to neurological defects, renal degradation, bone lesions and hypertension in humans even in trace levels, lead and cadmium have no known physiological functions (Viñas, Pardo-Martinez, & Hernández-Córdoba, 2000; Zhang, Wang, Cheng, Xia, & Liang, 2011).

The main sources of these elements in environment are natural or present as a result of industrial processes such as anthropogenic origin, mining, agricultural facilities, industrialization and traffic. Atmosphere, surface and ground waters, soil, foods and plants are affected by the trace elements owing to these facilities. As a conclusion, the determination of metals in the environment and food is important due to their positive and negative effects (Afzali, Mostafavi, Taher, & Moradian, 2007; Behbahani, Bagheri, Amini, et al., 2013; Behbahani, Bagheri, Taghizadeh, et al., 2013; Behbahani, Salarian, Amini, et al., 2013; Behbahani, Bide, et al., 2014; Behbahani, Salarian, et al., 2014; Lemos et al., 2006;

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Malekpour, Hajjaligol, & Taher, 2009; Pourreza & Ghanemi, 2009; Sharma & Pant, 2009; Viñas, Pardo-Martinez, & Hernández-Córdoba, 1999; Viñas et al., 2000). The determination of metals in the environmental and food samples is difficult due to its presence in the low concentration and matrices, and needs separation/preconcentration procedures (Daşbaşı, Saçmacı, Ülgen, & Kartal, 2015; Liang & Sang, 2008; Pourreza & Ghanemi, 2006).

Some separation/preconcentration procedures have been improved for trace metal determination involving different analytical techniques such as precipitation (Saito & Schneider, 2006; Saçmacı & Kartal, 2010; Yıldız, Saçmacı, Kartal, & Saçmacı, 2016), liquid–liquid extraction (Saçmacı & Kartal, 2011; Saçmacı, Kartal, & Saçmacı, 2011; Saçmacı et al., 2012a; Saçmacı et al., 2012b), cloud point extraction (Gil, Gasquez, Olsina, Martínez, & Cerutti, 2008; Lemos et al., 2008; Liang & Sang, 2008), electrochemical deposition (Burguera et al., 2005), membrane separation (Irigoyen, Moreno, Mendiguchia, & Vargas, 2006), or solid-phase extraction (Venkatesh & Singh, 2005).

Solid-phase extraction (SPE) have been prevalently used for separation and preconcentration of trace elements since this approach suggests a number of important utilities such as reducing of disposal costs, achievement of high recoveries and easy recovery of the solid phase. It is usually insufficient due to matrix interferences and very low concentrations of metal ions (Saçmacı, Kartal, Saçmacı, & Soykan, 2011). Some natural and synthetic resins are used as solid phase. Chelating resin is a resin that is employed as solid phase too because it has selective adsorption of trace metals (Kagaya et al., 2009; Rao, Daniel, & Gladis, 2004). For the determination of metals, it needs the instruments such as inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Boevski, Daskalova, & Havezov, 2000; Velitchkova, Pentcheva, & Daskalova 2004), inductively coupled plasma-mass spectrometry (ICP-MS) (Nicolai, Rosin, & Nicolai, 1999), laser-induced breakdown spectroscopy with laser ablation-ICP-MS (Meissner, Lippert, Okaun, & Guenther, 2004), atomic absorption spectrometry (AAS) (Saçmacı et al., 2011), and graphite furnace atomic absorption spectrometry (GFAAS) (Yang, Fen, Lei, Xiao, & Sun, 2009), which are the most extensively used techniques for analyzing these metals.

In this study, poly[N-(3-methyl-1H-indole-1-yl)]-2-methacrylamide-co-2-acrylamido-2-methyl-1-propane sulphonic acid-co divinylbenzene] (MMAD) as a chelating resin (Daşbaşı, Saçmacı, Şahan, Kartal, & Ülgen, 2013) for the separation/preconcentration of the trace metal ions has been used as solid phase extractor. Experimental parameters affecting the preconcentration of the analyte ions such as pH of sample, type, volume and concentration of eluent, flow rate of sample, sample volume, and matrix effects were studied and optimized. The proposed separation/preconcentration procedure was also applied certified reference materials such as SPS-WW2 Batch 108 waste water, and INCT-TL-1 Tea leaves, several natural waters, meat products and baby food samples.

## 2. Experimental

### 2.1. Instrument

A PerkinElmer model AAnalyst 800 flame atomic absorption spectrometer (Norwalk, CT, USA) equipped with a deuterium background correction system and an air-acetylene burner was used for the determination of metal ions. Spectral bandwidth of 0.7 nm, acetylene flow rate of 1.4 L min<sup>-1</sup>, and nebulizer flow rate of 10.0 mL min<sup>-1</sup> were conventional working parameters. For measuring pH values in the aqueous phase, a Consort model C533 pH meter combined with a glass-electrode and also a magnetic stirrer (Chiltern) were used.

### 2.2. Reagents and standard solutions

All reagents used were of the highest available purity and at least analytical reagent grade (Merck, Darmstadt, Germany). Deionized water was used for the preparation of the solutions. Standard stock solutions of Cd(II), Co(II), Cr(III), Cu(II), Fe(III), Pb(II), and Zn(II) (1000 mg L<sup>-1</sup>) were prepared by dissolving their nitrate salts in distilled deionized water with the addition of 1 mol L<sup>-1</sup> HNO<sub>3</sub> and further diluted daily prior to use. The calibration standards were not submitted to the separation-preconcentration procedure. All the plastic and glassware were cleaned by soaking in dilute HNO<sub>3</sub> (1:1) and rinsed with distilled deionized water prior to use.

The chelating resin, poly[N-(3-methyl-1H-indole-1-yl)-2-methacrylamide-co-2-acrylamido-2-methyl-1-propane sulphonic acid-co divinylbenzene] (MMAD), was synthesized as reported in the literature (Daşbaşı et al., 2013) and used. The synthesized polymer was washed with 1 mol L<sup>-1</sup> HNO<sub>3</sub>, NaOH, ethyl alcohol and distilled deionized water. Then the chelating resin was dried in an oven at about 60 °C. A glass mini column containing 0.5 g of the resin in water suspension has 10 cm length and 1.0 cm diameter. The resin bed height in the column was approximately 1.0 cm. A small amount of glass-wool was placed on the top of the resin to avoid disturbance during the sample passage. The mini column was washed thoroughly with distilled water and then preconditioned at the working pH 8 using NH<sub>3</sub>/NH<sub>4</sub>Cl buffer solution before passing the solutions containing the analyte ions.

### 2.3. General procedure

The preconcentration procedure was optimized by using model solutions containing known amounts of the metal ions before application of the method to real samples. For doing this, 25 mL of the model solution containing the analytes was adjusted to pH 8 using 2 mL of NH<sub>3</sub>/NH<sub>4</sub>Cl buffer solution and the sample solution was loaded to the column. The flow of the sample solution through the column was gravitationally performed at a flow rate of 2 mL min<sup>-1</sup>. The flow rate of the sample solution was controlled by using the stopcock of the mini column. After being completed the passing of the sample solution, the column was washed with a small amount of distilled water. Then, the retained metal ions were eluted from the column with 10 mL of 3 mol L<sup>-1</sup> HNO<sub>3</sub> at a flow rate of 2 mL min<sup>-1</sup>. The metal ion concentrations in the eluate solution were determined by flame atomic absorption spectrometry (FAAS).

### 2.4. Preparation of samples for analysis

1 g of each food sample was put into a 100-mL beaker and a mixture of 10 mL of concentrated HNO<sub>3</sub> and 2 mL of concentrated H<sub>2</sub>O<sub>2</sub> solutions was added, and then the digestion was performed on a hot plate adjusted to 200 °C. After the digestion, the suspension obtained was filtered through a filter paper with blue ribbon and completed to 25 mL with distilled deionized water. Analyses for blanks were carried out in the same way without use sample. Then the preconcentration/separation procedure given above was applied to the obtained solutions. After the preconcentration step, the final measurement volumes were completed to 5 mL, and the measurements were performed by FAAS.

The proposed procedure was also applied to the two certified reference materials, SPS-WW2 Batch 108 (10 mL) and 1.0 g of INCT-TL-1 tea leaves, the later was digested using a mixture of 10 mL of concentrated HNO<sub>3</sub> and 2 mL of concentrated H<sub>2</sub>O<sub>2</sub>. The other processes in analyzing of these certified reference materials were the same just mentioned above.

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