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# Simultaneous determination of cadmium, iron and tin in canned foods using high-resolution continuum source graphite furnace atomic absorption spectrometry

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## ABSTRACT

A method was established to simultaneously determine cadmium, iron and tin in canned-food samples using high-resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS GF AAS). The quantification step has been performed using the primary line (228.802 nm) for cadmium and the adjacent secondary lines (228.725 nm and 228.668 nm) for iron and tin, respectively. The selected chemical modifier was an acid solution that contained a mixture of 0.1% (w/v) Pd and 0.05% (w/v) Mg. The absorbance signals were measured based on the peak area using 3 pixels for cadmium and 5 pixels for iron and tin. Under these conditions, cadmium, iron and tin have been determined in canned-food samples using the external calibration technique based on aqueous standards, where the limits of quantification were 2.10 ng  $g^{-1}$  for cadmium, 1.95 mg kg<sup>-1</sup> for iron and 3.00 mg kg<sup>-1</sup> for tin, and the characteristic masses were 1.0 pg for cadmium, 0.9 ng for iron and 1.1 ng for tin. The precision was evaluated using two solutions of each metal ion, and the results, which were expressed as the relative standard deviation (RSD%), were 3.4-6.8%. The method accuracy for cadmium and iron was confirmed by analyzing a certified reference material of apple leaves (NIST 1515), which was supplied by NIST. However, for tin, the accuracy was confirmed by comparing the results of the proposed method and another analytical technique (inductively coupled plasma optical emission spectrometry). The proposed procedure was applied to determine cadmium, iron and tin in canned samples of peeled tomato and sardine. Eleven samples were analyzed, and the analyte concentrations were  $3.57-62.9 \text{ ng g}^{-1}$ , 2.68-31.48 mg kg<sup>-1</sup> and 4.06–122.0 mg kg<sup>-1</sup> for cadmium, iron and tin, respectively. In all analyzed samples, the cadmium and tin contents were lower than the permissible maximum levels for these metals in canned foods in the Brazilian legislation.

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## 1. Introduction

Currently, the intake of canned foods is a growing practice worldwide. However, the daily ingestion of these foods is not recommended by nutritionists and health professionals. In general, these foods have tinplate packaging, which better preserves the foods in terms of taste and natural aspect [1-3]. However, the interaction between the foods and the internal surface of these packagings causes some metals and metalloids to migrate into the foods [1,4]. Tin determination in canned foods has often been performed because this element is a major component in the composition of packaging to preserve these foods [4]. In high

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http://dx.doi.org/10.1016/j.talanta.2016.02.023 0039-9140/© 2016 Published by Elsevier B.V. concentrations, tin can change the food properties such as color and taste and decrease the expiration deadline [5,6]. Although this metal has been considered non-toxic, some studies have demonstrated that tin causes gastrointestinal harm such as nausea, vomit and diarrhea [6,7].

Meanwhile, cadmium is known for its high toxic and bioaccumulative effect [8,9] and is considered one of the great contaminations in plant foods because of the frequent use of phosphate fertilizers in agriculture [10].

Iron is one of the most important elements for human life and is found in all cells of the human body. Iron is required to make the protein hemoglobin in erythrocytes, whose principal function is to transport oxygen from the lungs to the body cells. The decrease in the amount of hemoglobin in the blood causes iron deficiency anemia, which is a public health issue in some countries. Thus, the development of analytical methods to determine iron in food samples is notably necessary because food is the main form of iron







intake by humans [11–13].

Atomic absorption spectrometry (AAS) is an analytical methodology that is conventionally known as uni-element. However, several strategies have been performed to expand the capacity of determination of the AAS as a multi-element technique. Among these strategies, a system using a computer program provided a fast change of hollow cathode lamps and enabled the development of sequential methods using flame atomic absorption spectrometry. Then, this system enabled the use of the reference-element technique to correct the matrix interferences by directly determining iron and manganese in wine [14]. In addition, a speciation analysis method was established for arsenic and selenium, where the separation was performed using high-performance liquid chromatography, and arsenic(III), arsenic(V), selenium(IV) and selenium(VI) were quantified by hydride generation and the AAS of the sequential mode [15]. Furthermore, another procedure that involved slurry sampling and detection by FAAS was proposed for the sequential determination of copper, manganese and iron in seafood samples [16].

The high-resolution continuum source atomic absorption spectrometry (HR-CS AAS), which uses a xenon lamp as the continuum source and a CCD array as the detector, enabled the development of multi-element analytical strategies for sequential [17–19] or simultaneous determination of two or more elements [20–24]. Simultaneous determination is possible when the atomic lines of the elements are in the same spectral window of the CCD array detector [21]. Particularly, in graphite furnace atomic absorption spectrometry (GF AAS), simultaneous determination significantly increases the analytical frequency of the method and the number of analytical determinations using a single graphite tube because a single temperature program can determine two or more elements. The selected atomic lines should be compatible with the element concentrations in the samples, and the selected chemical modifier should be efficient to quantify all elements to be determined [25.26].

The first proposed analytical method for simultaneous determination using HR-CS GF AAS was established to determine cadmium and iron in grain products, and the used lines were 228.802 nm for cadmium and 228.726 nm for iron [22]. Since then, several other works have been developed using this analytical methodology [20-29]. Welz et al. simultaneously determined cadmium and iron in sludge using slurry sampling [23]. An analytical strategy was proposed by Curtius et al. to simultaneously determine nickel and vanadium in oil samples [24]. Ozbek and Ozcan simultaneously determined cobalt, aluminum and iron in several certificated reference materials and waste water samples [26]. Resano et al. proposed a method to simultaneously and directly determine cobalt, iron, lead and nickel in solid samples of carbon nanotubes. The used atomic lines were: 283.245 nm for iron, 283.306 nm for lead, 283.393 and 283.443 nm for cobalt and 283.455 nm for nickel [27]. Procopio et al. also developed a direct method to simultaneously determine iron and nickel in solid samples of biological materials [28]. Boschetti et al. published a method to simultaneously determine Mo and Ni in wine and soil amendments [29].

This paper proposes a method to simultaneously determine cadmium, iron and tin in canned foods using HR-CS GFAAS. The elements were measured in the same spectral window using the following atomic lines: 228.802 nm for cadmium, 228.725 nm for iron and 228.668 nm for tin.

## 2. Experimental

### 2.1. Instrumentation

All determinations were performed using a model ContrAA 700 high-resolution continuum source electrothermal atomic absorption spectrometer from Analytik Jena AG (Jena, Germany). This equipment has a xenon short-arc lamp (GLE, Berlin, Germany), which operated in the hot-spot mode as a continuum radiation source, a high-resolution double monochromator, a charge-couple device (CCD) array detector and a transversally heated tube atomizer. A pyrolytically coated graphite tube with an integrated platform (Analytik Jena) was used to atomize the analytes. An MPE 60 autosampler (Analytik Jena) was used to introduce the liquid samples. Argon (White Martins, Brazil) with 99.998% purity was used as the purge gas and protective gas with a rate flow of 2.0 L min<sup>-1</sup> except during the atomization stage. The determinations were performed using the primary absorption line at 228.802 nm for cadmium and the adjacent secondary lines at 228.725 and 228.668 nm for iron and tin, respectively. The absorbance signals were obtained by measuring the peak area with integration times of 2.0 s for cadmium and 6.0 s for iron and tin. In addition, three pixels (the central pixel plus the adjacent ones,  $CP \pm 1$ ) were used to obtain the absorbance signal of cadmium, whereas five pixels (the central pixel plus two adjacent ones,  $(CP \pm 2)$  were used to obtain the absorbance signals of iron and tin, considering the wavelength of each element. The optimized graphite furnace temperature program is shown in Table 1.

A model ETHOS EZ closed-vessel microwave oven from Milestone (Sorile, Italy) was used for the acid decomposition of the canned-food samples and certified reference material.

## 2.2. Reagents, solutions and samples

All solutions were prepared using ultrapure water with resistivity of 18.2 M $\Omega$  cm, which was obtained from a Milli-Q water purification system from Millipore (Bedford, MA, USA). Analytical-grade nitric acid from Merck (Darmstadt, Germany) was used to prepare the calibration and modifier solutions.

The cadmium, iron and tin calibration solutions were prepared by sequentially diluting the stock solutions, which contained 1000 mg L<sup>-1</sup> of each element (Merck). The solutions of the chemical modifiers were prepared from the stock solutions that contained 1% (w/v) palladium (Merck) and 4000 mg L<sup>-1</sup> magnesium (Merck): one solution that contained the mixture of 0.1% (w/ v) Pd, 0.05% (w/v) Mg and 0.5% (v/v) HNO<sub>3</sub>, and another contained 0.05% (w/v) Mg and 0.5% (v/v) HNO<sub>3</sub>.

All canned-food samples (sardine and tomato) were acquired in the supermarkets in Salvador City, Bahia, Brazil. The certified reference material of apple leaves SRM NIST 1515 was obtained from the National Institute of Standard and Technology (Gaithersburg, MD, USA) and used to evaluate the method accuracy for cadmium and iron.

#### Table 1

Temperature program adopted for the simultaneous determination of cadmium, iron and tin in canned foods by HR-CS GF AAS.

Stage	Temperature (°C)	Ramp (°C s <sup><math>-1</math></sup> )	Hold time (s)	Ar flow rate (L min <sup>-1</sup> )
Drying 1 Drying 2 Drying 3 Pyrolysis Atomization	80 110 140 700 2550	6 3 5 300 3000	20 20 10 10 6	2 2 2 2 0
Cleaning	2551	500	4	2

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