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Sequential injection analysis implementing multiple standard additions for As speciation by liquid chromatography and atomic fluorescence spectrometry (SIA-HPLC-AFS)

José P. Jesus, Carlos A. Suárez, José R. Ferreira, Maria F. Giné*

Centro de Energia Nuclear na Agricultura, Universidade de São Paulo, Av. Centenário 303, 13416-000, Piracicaba, Brazil

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ABSTRACT

An analytical procedure for multiple standard additions of arsenic species using sequential injection analysis (SIA) is proposed for their quantification in seafood extracts. SIA presented flexibility for generating multiple specie standards at the ng mL^{-1} concentration level by adding different volumes of As(III), As(V), monomethylarsonic (MMA) and dimethylarsinic (DMA) to the sample. The mixed sample plus standard solutions were delivered from SIA to fill the HPLC injection loop. Subsequently, As species were separated by HPLC and analyzed by atomic fluorescence spectrometry (AFS). The proposed system comprised two independently controlled modules, with the HPLC loop acting as the intermediary device. The analytical frequency was enhanced by combining the actions of both modules. While the added sample was flowing through the chromatographic column towards the detection system, the SIA program started performing the standard additions to another sample. The proposed method was applied to spoiled seafood extracts. Detection limits based on 3σ for As(III), As(V), MMA and DMA were 0.023, 0.39, 0.45 and 1.0 ng mL^{-1}, respectively.

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1. Introduction

Speciation of arsenic has been accomplished by different hyphenated schemes, usually involving high-performance liquid chromatography (HPLC) or capillary electrophoresis (CE) columns coupled to atomic spectrometers. These separation techniques have been frequently coupled to inductively coupled plasma mass spectrometer (ICP-MS), due to its wide applicability, and seldom to atomic fluorescence spectrometry (AFS), which is limited to species forming volatile compounds. However, analytical characteristics of As speciation by hydride generation atomic fluorescence spectrometry (HG-AFS), namely detection limit, sensitivity, repeatability, and reproducibility were found comparable to those of HPLC-ICP-MS [1].

HPLC and HG-AFS have been combined to determine As species, in urine [2] and seafood [3] samples. The biotransformation of inorganic arsenic included the production of species named arsenite (As(III)) $As(OH)_3$, arsenate (As(V)) $AsO(OH)_3$, monomethylarsonic acid (MMA) $(CH_3)AsO(OH)_2$ and dimethylarsinic acid (DMA) $(CH_3)_2AsO(OH)$. Also, the less stable monomethylarsonous acid $(CH_3)As(OH)_2$ and dimethylarsinous $(CH_3)_2As(OH)$ were identified [2] by HPLC-HG-AFS using a reverse phase C18 column. The anion exchange chromatographic column Hamilton PRP-X100 is frequently used for separation of those arsenic species. In a recent review on speciation analysis by HPLC-HG-AFS, 89 citations corresponded to arsenic speciation by HPLC-AFS, 54 of which used the PRP-X100 column for As(III), As(V), MMA, and DMA [4].

Flow systems have usually been described for in-line extraction of As species from soil samples placed in a column and determination by HG-AFS [5]. The arsenic extracted fractions were in-line oxidized to As(V) previously to quantification involving the standard addition method (SAM). This procedure was needed for overcoming sample matrix effects.

The management of solutions by a sequential injection analyzer (SIA) has been exploited for automation of SAM [6]. Sequential injection standard addition was applied for quantification of Hg in river water samples. In that approach, in-line digestion carried out by a BrCl solution and UV-irradiation promoted the oxidation of Hg species to Hg^{2+}, allowing the sequential reduction of Hg in the sample with further detection by cold vapor AAS [6].

SAM was previously implemented in a SIA system [7] using a mono-segmented scheme to promote different dilutions of a single standard solution for the determination of Fe(II) in an anti-anemic medicine [8]. The mono-segmented approach is based on defining a constant total volume partitioned among the sample, reagent, and variable volumes of the standard solution. The mono-segmented sequential injection approach constituted the basis of a calibration method using SIA complementary dilutions [9]. The sample, stan-

* Corresponding author. Tel.: +55 19 3429 4636.
E-mail address: mfgine@cena.usp.br (M.F. Giné).

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dards and diluents solutions were combined and interacted in the SIA holding coil at various volumetric proportions under computer control.

The potentialities of SIA for chemical speciation have been reviewed elsewhere [10]. Its versatility for speciation is related to the combination of flow diagrams combining a 10-port selection valve and 1 or 2 detection systems. In a previous paper of our group, it was demonstrated that the management of arsenic solutions by SIA resulted in a pronounced reduction of the formed hazardous waste residues [11], so that SIA ranks among the environmentally friendly approaches towards chemical analysis.

In this paper, an instrumental configuration using SIA coupled to HPLC-HG-AFS is proposed for implementing SAM aiming at simultaneous quantification of four As species in each sample. The reason for coupling SIA to the HG-AFS was the high sensitivity of the combination to detect As(III), As(V), MMA and DMA which is comparable with that displayed by ICP-MS, in addition to the low cost of the equipment and operation. The proposed procedure was applied to the analysis of spoiled seafood samples to allow the joint quantification of the most toxic arsenic species avoiding interconversions.

2. Experimental

2.1. Instrumentation

The components of the SIA-HPLC-AFS system are presented in Fig. 1.

The SIA system (FIA-Lab, Fiatron, Seattle, USA) with an 8-port selecting valve, a 1 mL syringe pump, and a 0.8 mL holding coil was used to perform standard additions to the sample. Solutions from the holding loop were delivered through port number 8 to fill a loop 150 cm long made of peek tubing 0.5 mm id, installed in the chromatographic injector (mod 7125, Rheodine, Berkeley, USA). The isocratic HPLC pump (Shimadzu, LC-10AD, Kyoto, Japan) was connected to a selector valve of the mobile phase solutions C or E. The chromatographic guard pre-column and column PRP-X100 (250 mm × 4.1 mm, 10 μm packed particles) (Hamilton, Reno, USA) were used. The parts of the equipment used for atomic fluorescence (Excalibur, PS Analytical, Orpington, UK) were a detector, a hydride generator separator, a gas liquid separator (GLS), and an atomization source. An 8-channel peristaltic pump (Ismatec, Glattbrug, Swiss) was used for pumping the sodium tetraborohydride and HCl reagents. An auxiliary argon flow was introduced just before the GLS in order to accelerate the transport of the evolved gases towards the atomizer.

Sample extractions were performed using an ultra-sonic bath (40 kHz, Thornton, Piracicaba, Braz.). Total As was determined by ICP OES (Optima 3000, Perkin Elmer, Norwalk, USA).

2.1.1. The SIA program

The SIA system was programmed according to the steps presented in Table 1. The analytical sequence started by introducing defined volumes of water, sample, As species, and water into the holding coil (HC). Solutions inside the HC were mixed by reversing the flow direction. After mixing, the syringe was filled with a defined volume of water by the lateral inlet used to pump the HC solutions through port 8 to fill the sample loop of the chromatographic injector.

2.1.2. SIA-HPLC-HG-AFS system

The systems scheme in Fig. 1, left side, shows the SIA components used to perform standard additions to the sample. The volume pumped by the syringe pump was calculated so as exactly fill the loop in the HPLC injector port. This was followed by operation of the injector, leaving the sample solution inside the loop to be carried by the isocratic pump through the PRP-X100 column. In this case, the less concentrated phosphate buffer E was pumped. Thereafter, the HPLC pump was operated and the buffer solution in C was sent through the HPLC column. Species leaving the HPLC column were carried to a hydride generation unit, receiving the reagents pumped by a peristaltic pump (PP). This was followed by introductions of Ar plus H_2 into a gas/liquid separation chamber (GLS), so as to efficiently carry the gases to the atomic fluoresce spectrometer (AFS).

2.2. Chemicals

All solutions were prepared with deionized water (>18.2 MΩ cm) from a Milli-Q system (Millipore, Bedford, USA). Standard solutions for arsenite and arsenate were prepared by dissolving $AsNaO_2$ or $Na_2HAsO_4{\cdot}7H_2O$ (Merck, Darmstadt, Germany) in phosphoric acid. The monomethylarsonic (MMA) and dimethylarsinic (DMA) acids were from Supelco (Sigma–Aldrich, Munich, Germany). Working standard solutions of 100.0 ng mL^{-1} of each As specie were used for performing the standard additions.

Mono and dibasic potassium phosphate solutions at pH 6.2 in concentrations of 12 and 24 mmol L^{-1} were used as mobile phase solutions for the chromatographic separations [12]. Chromatographic grade methanol and nitric acid from Merck (Darmstadt, Germany) were used for column regeneration. Sodium tetrahydroborate 1.3% $NaBH_4$ (Nuclear, São Paulo, Brazil) was prepared with 0.1 mol L^{-1} NaOH. Hydrochloric acid 1.5 mol L^{-1} was used for post-column hydride generation of As species.

2.3. Sample treatment

About 0.25 g of lyophilized shark (*Pleonacea glauca*) muscular tissue was extracted with 20.0 mL of water in an ultrasonic bath for 1 h. This solution was stored in a refrigerator for 4 months. Also, the liquid from a can of natural bivalves (*Mesodesma donacium*), whose expiration date had occurred 3 years prior to the analysis, was filtered through a 0.2 μm pore cellulose acetate filter just before analysis. Accordingly to the manufacturer, the sample contained water, salt, and the preservatives INS 451i (a stabilizer) and INS 385 (an antioxidant). A mass of 0.25 g of dogfish muscle (DORM-2) CRM from the National Research Council of Canada was extracted with 10 mL of water. Extracts were centrifuged at 3500 rpm and the filtrated supernatant made up to 10 mL final volume.

2.4. The in-line standard additions of multiple As species

The SIA system in Fig. 1 was programmed to perform standard additions of three concentrations of the four As species to the sample. Samples were analyzed in a first run to observe the main As species occurring and the dilution degree required to support additions. This was attained by programming steps 1, 2, 7 and 8 in Table 2, which control the volumes of water and sample introduced into HC. Depending on the number of species to be determined and their concentrations, the volumes of sample, standards and water were then reprogrammed according to the steps 3–6 sequence in Table 2.

The system was dimensioned to perform the addition of four As species standard solutions to the sample in less than 1 min by performing the actions represented in Table 2. Taking in consideration the different concentration ranges of sample species, the experimental sequence of additions presented in Table 2 was followed. To perform the additions, a variable plug of water was inserted through valve 6, in both extremes of the sample and standard solutions, in order to keep constant the total volume. This water amount added in each run was programmed following the equation in Table 2.

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