



Bismuth as a general internal standard for lead in atomic absorption spectrometry



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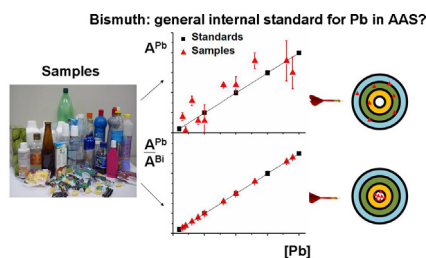
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HIGHLIGHTS

- Single internal standard is commonly proposed for definite application in AAS.
- Internal standard for general use in AAS techniques is original.
- Bi showed efficiency as internal standard for Pb determinations by FAAS and GFAAS.
- Assorted samples were analyzed and accurate results were found.

GRAPHICAL ABSTRACT



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ABSTRACT

Bismuth was evaluated as internal standard for Pb determination by line source flame atomic absorption spectrometry (LS FAAS), high-resolution continuum source flame atomic absorption spectrometry (HR-CS FAAS) and line source graphite furnace atomic absorption spectrometry (LS GFAAS). Analysis of samples containing different matrices indicated close relationship between Pb and Bi absorbances. Correlation coefficients of calibration curves built up by plotting $A^{\text{Pb}}/A^{\text{Bi}}$ versus Pb concentration were higher than 0.9953 (FAAS) and higher than 0.9993 (GFAAS). Recoveries of Pb improved from 52–118% (without IS) to 97–109% (IS, LS FAAS); 74–231% (without IS) to 96–109% (IS, HR-CS FAAS); and 36–125% (without IS) to 96–110% (IS, LS GFAAS). The relative standard deviations ($n=12$) were reduced from 0.6–9.2% (without IS) to 0.3–4.3% (IS, LS FAAS); 0.7–7.7% (without IS) to 0.1–4.0% (IS, HR-CS FAAS); and 2.1–13% (without IS) to 0.4–5.9% (IS, LS GFAAS).

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1. Introduction

Flame (F) and graphite furnace (GF) atomic absorption spectrometry (AAS) are considered well-established methods for elemental analysis of several samples, although interferences caused by matrices may frequently occur and affect the quality of

results without adopting proper strategies. Matrix matching [1], standard additions [2] and internal standardization [3] are calibration strategies to correct for such interferences. Internal standardization (IS) combines the simplicity of external calibration with the usefulness of standard additions without requiring matrix-matching.

Although several absorption lines can be monitored based on simultaneous [4] or sequential modes [5], the choice of a suitable internal standard is difficult because of the limited number of published works on this topic. Indeed, most studies deal with the

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selection of a single internal standard for a specific application [6–9]. The availability of a single element that could be employed as internal standard to determine an analyte (or more analytes) in several types of samples could expand the application of IS in AAS.

The literature has reported the use of Bi as internal standard for Pb determination in different matrices by GFAAS, including urine and blood [10], vinegar [11], wine [12], and sugar-cane spirit [13]. These findings suggest that Bi should be evaluated as internal standard in multiple analyses of assorted samples by FAAS and GFAAS, so that it could be designated as a 'universal' internal standard in Pb determination by AAS.

This study describes the evaluation of Bi as internal standard to determine Pb in several samples. Line source flame atomic absorption spectrometry (LS FAAS), high-resolution continuum source flame atomic absorption spectrometry (HR-CS FAAS) and line source graphite furnace atomic absorption spectrometry (LS GFAAS) were used to evaluate the suitability of using Bi as universal IS in Pb determinations by AAS.

2. Experimental

2.1. Instrumentation

Measurements by HR-CS FAAS were carried out in an Analytik Jena ContrAA 300 spectrometer (Jena, Germany) equipped with a xenon short-arc lamp operated in "hot-spot" mode as a continuum radiation source. Air-acetylene flame was used for Bi and Pb atomization. High-purity acetylene (99.7%, Air Liquide, Sertãozinho, SP, Brazil) was used as fuel gas. The optimum flow-rates of air and acetylene were 7.2 and 1.3 L min⁻¹, respectively, for both elements. All measurements were carried out in triplicate at 223.061 nm (Bi) and 283.306 nm (Pb) using the following instrumental parameters: 5.0 mL min⁻¹ aspiration flow-rate, 7 mm observation height, and wavelength-integrated absorbance (WIA) equivalent to 3 pixels (central pixel ±1; central pixel corresponds to measurement at core line).

A Varian AA240FS spectrometer (Mulgrave, Australia) equipped with a deuterium-lamp background correction system was used for measurements by LS FAAS. Hollow cathode lamps for Bi and Pb were operated at 10 and 7 mA currents, respectively. Absorbance was measured at 223.1 nm (Bi) and 283.3 nm (Pb) using a 3 s integration time and 0.5 nm spectral bandwidth. Air and acetylene flow-rates for both elements were 7.0 and 1.1 L min⁻¹, respectively. Sample aspiration flow-rate and observation height were 5.0 mL min⁻¹ and 7 mm, respectively.

A PerkinElmer SIMAA-6000 simultaneous line source atomic absorption spectrometer (Shelton, CT, USA) equipped with a longitudinal Zeeman-effect background correction system, a transversely heated graphite tube atomizer (THGA) with integrated platforms, and an AS-72 autosampler was used for measurements by LS GFAAS. Electrodeless discharge lamps employed for Bi (223.1 nm) and Pb (283.3 nm) were operated at 380 and 450 mA, respectively. Atomic absorption signals were measured in triplicate. High-purity argon (99.999%, Air Liquide) was used as purge gas. Synthetic air (99%, Air Liquide) was used for air-assisted pyrolysis. The optimized heating program of the graphite tube (temperature, °C; ramp time, s; hold time, s, respectively) was: dry – step 1 (110; 1; 30); dry – step 2 (130; 10; 30); air-assisted pyrolysis – step 3 (600; 10; 40); purge gas – step 4 (40; 5; 35); argon-assisted pyrolysis – step 5 (1200; 10; 10); atomization – step 6 (1800; 0; 5); cleaning – step 7 (2500; 1; 5). A flow-rate of argon at 250 mL min⁻¹ was fixed for steps 1, 2, 4, 5 and 7. Step 3 employed an air flow-rate at 250 mL min⁻¹. The argon flow was interrupted during atomization. All experiments were carried out using stabilized temperature platform furnace (STPF) conditions [14].

Microwave-assisted acid digestion of samples and certified reference materials (CRMs) was carried out employing an Anton Paar Multiwave microwave oven (Graz, Austria) equipped with 25-mL quartz vessels.

2.2. Reagents and analytical solutions

High purity water obtained from a Millipore Rios 5[®] reverse osmosis and a Millipore Milli-Q[™] Academic[®] deionizer system (resistivity 18.2 MΩ cm, Millipore, Bedford, MA, USA), and nitric acid (Suprapur[®], Merck, Darmstadt, Germany) were used to prepare all solutions.

Single element standard stock solutions containing 1000 mg L⁻¹ of Pb or Bi (Merck) were properly diluted to daily prepare analytical solutions in the following concentration ranges: 0.1–2.0 mg L⁻¹ Pb + 2.0 mg L⁻¹ Bi (HR-CS FAAS), 0.3–10 mg L⁻¹ Pb + 4.0 mg L⁻¹ Bi (LS FAAS), and 5.0–50 μg L⁻¹ Pb + 100 μg L⁻¹ Bi (LS GFAAS). Analytical solutions and samples were acidified to 2.0% (v/v) HNO₃. A 1000 mg L⁻¹ Ru stock solution (RuCl₃·3H₂O; Fluka GmbH, Steinheim, Switzerland) was used as permanent chemical modifier.

Matrix effects were replicated simulated by using sucrose and ethanol solutions. A 500 g L⁻¹ sucrose stock solution was prepared by dissolving 50 g of the reagent (Merck) in 100 mL of deionized water. Ethanol–water solutions containing within 0–95% ethanol (v/v) were prepared by appropriated dilution from 99.7% (v/v) ethanol (JT Baker, Phillipsburg, NJ, USA).

All solutions were stored in high-density polypropylene bottles (Nalgene, Rochester, NY, USA). Plastic bottles and glassware materials were decontaminated by soaking in 10% (v/v) HNO₃ for at least 24 h, and then rinsing abundantly in deionized water before use.

2.3. Procedure

2.3.1. Sample preparation

The following samples were acquired at the local market of Araraquara (SP, Brazil): household cleaning solutions, colored sugars, hard candies, mouthwash, fruit juices (orange, lemon and grape), energy drink, tea drink, soft drinks, beer, vodka, sugar-cane spirit, mineral water, vinegar, ethanol fuels, peanut, polyethylene terephthalate (PET) bottle, medicinal plants (*Maytenus ilicifolia*, *Peumus boldus*), liquid fertilizer, solid fertilizer, shampoo and milk. All samples were prepared in triplicate as described below:

(i) Liquid samples were diluted (1 + 1 v/v) with 4.0% (v/v) HNO₃. In addition to dilution, milk samples were also spiked with 100 μL of Triton X-100.

(ii) Colored sugars and hard candies (0.25 g) were dissolved in 25 mL of 2.0% (v/v) HNO₃.

(iii) Medicinal plant and peanut samples were prepared by microwave-assisted acid digestion according of the following procedure: approximately 0.25 g of sample was accurately weighed and transferred to a microwave flask. Aliquots of 3.0 mL of concentrated nitric acid, 2.0 mL of 30% (m/v) H₂O₂ and 1.0 mL of deionized water were then added and the mixture was submitted to the following optimized 3-step microwave-assisted digestion program: (1) 15 min from 0 to 900 W; (2) 30 min at 900 W and (3) 20 min at 0 W (ventilation). After digestion and cooling, the resulting solutions were transferred to 25-mL volumetric flasks and the volumes were made up to the mark using distilled–deionized water.

(iv) The PET sample was prepared by microwave-assisted acid digestion. Approximately 0.15 g of sample was accurately weighed and transferred to a microwave flask. Aliquots of 2.0 mL of concentrated nitric acid, 1.0 mL of concentrated hydrochloric acid and 3.0 mL of deionized water were then added and the mixture

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