



# An on-line preconcentration/separation system for the determination of bismuth in environmental samples by FAAS

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## ABSTRACT

An on-line preconcentration procedure for the determination of bismuth by flame atomic absorption spectrometry (FAAS) has been described. Lewatit TP-207 chelating resin, including iminodiacetate group, packed in a minicolumn was used as adsorbent material. Bi(III) was sorbed on the chelating resin, from which it could be eluted with  $3 \text{ mol L}^{-1} \text{ HNO}_3$  and then introduced directly to the nebulizer-burner system of FAAS. Best preconcentration conditions were established by testing different resin quantities, acidity of sample, types of eluent, sample and eluent solution volumes, adsorption and elution flow rates, and effect of interfering ions. The detection limit of the method was  $2.75 \mu\text{g L}^{-1}$  while the relative standard deviation was 3.0% for  $0.4 \mu\text{g mL}^{-1}$  Bi(III) concentration. The developed method has been applied successfully to the determination of bismuth in pharmaceutical cream, standard reference materials and various natural water samples with satisfactory results.

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## 1. Introduction

Determination of bismuth is important in environmental chemistry, cosmetic industry, medicine, semiconductors, alloys, metallurgical additives, recycling of uranium nuclear fuels and fabrication of catalysts. Since the use of bismuth and its compounds in different areas of life increased and this element started to be considered toxic for humans, animals and plants [1–3]. Although intestinal absorption of bismuth is limited in humans, because of its low solubility and its propensity to form insoluble oxychloride salts, some absorption must occur to produce measurable concentrations in body fluids and tissues [4]. As the use of bismuth in medicine increases, it has spread in the environment and the chance of exposure of organisms to bismuth has increased. A number of toxic effects in humans have been attributed to bismuth compounds [5–7]. Therefore, new methods are required for the low cost and rapid determination of Bi(III) in the pharmaceutical manufacture and quality control process. Cui et al. have proposed a new method for the determination of bismuth in pharmaceutical products using methyltriphenylphosphonium bromide as a molecular probe by resonance light scattering technique [8].

Several methods have been reported for the determination of Bi(III) in rock, water, alloy and biological samples. Some spectrophotometric methods have been applied for the determination of bismuth [9–11]. The extraction, in the presence of masking

agents, with dithizone in chloroform or carbon tetrachloride is a selective spectrophotometric method for separating traces of bismuth [9]. 2,6-Dichloroarsenazo as chromogenic reagent has been used by Zhang et al. in the spectrophotometric determination of bismuth, in aqueous  $0.6 \text{ mol L}^{-1} \text{ HClO}_4$  solution, forming a deep blue complex with a maximum absorption at 631 nm [10]. Also, Hoshi et al. have used thiourea as a complexing agent in the spectrophotometric determination of bismuth after collection and elution of Bi(III)–thiourea complex on chitin [11]. Tzanavaras et al. reported a spectrophotometric method for the determination of bismuth in pharmaceutical products using sequential injection analysis [12]. Methylthymol blue (MTB) was used as a color forming reagent and the absorbance of the Bi(III)–MTB complex was monitored at 548 nm. On the other hand, hydride generation atomic absorption spectrometry (HG-AAS) [2,3,13], hydride generation inductively coupled plasma atomic emission spectrometry (HG-ICP-AES) [14], electrothermal vaporization ICP mass spectrometry (ETV-ICP-MS) [15], potentiometric stripping analysis (PSA) [16], anodic [17,18] and cathodic [19] stripping voltammetry have also been applied in determination of bismuth in various samples. In another work made by Wang et al., the preconcentration and voltammetric behavior of Bi on a sodium humate modified carbon paste electrode were studied using cyclic voltammetry and differential pulse stripping voltammetry for the determination of bismuth in medicine for stomach [18].

Conventional off-line preconcentration procedures, although effective, are usually time consuming and tedious, require large quantities of sample and reagents, in consequence are vulnerable to contamination and analyte losses. Flow injection (FI) methods

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provide an opportunity to avoid contamination and large reagent consumption by working with closed systems. In addition, on-line systems are excellent tools for solution management, allowing the easy implementation of different steps required for selectivity and/or sensitivity enhancement. On-line methods offer higher sample throughput and better precision and accuracy compared to off-line methods. In most on-line FAAS methods, the carrier and reagent solutions are continuously fed to the flow system, mixed on-line and passed through the microcolumns prior to detection [20,21].

In the present paper, we have evaluated a new on-line system with a home-made controller having five independent channels for preconcentration and determination of Bi(III) species. Because the studies made on determination of bismuth in various matrices are insufficient in the literature, especially using on-line systems with FAAS. For this purpose, Lewatit TP-207 resin was firstly used for the on-line determination of Bi(III) ions. According to our knowledge, an on-line preconcentration system (home-made) has not been used before in the determination of Bi(III) by FAAS.

## 2. Experimental

### 2.1. Instrument

A Perkin Elmer (Norwalk, CT, USA) model AAnalyst 800 flame atomic absorption spectrometer equipped with a deuterium background correction system and an air-acetylene burner was used for the determination of bismuth. The wavelength used for bismuth was 223.1 nm. Spectral bandwidth of 0.2 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. During the signal measurements, the integration time was 0.1 s.

The flow system comprises a peristaltic pump with variable speed (Watson-Marlow Inc., Wilmington, MA, USA), a multichannel peristaltic pump (Ismatec SA, Glattbrugg, Switzerland) furnished with silicone tubes to deliver all solutions, and two three-way valves (Cole-Parmer Inc. Co., IL, USA) to select solution ways. Peristaltic pumps (PP) and valves (V) were controlled by the five-channel controller and each channel can be set for 36 different timing periods with thumb switches. This unit was constructed in our instrumentation laboratory (at low cost). The flow system was made using fittings, unions and tees made of plastic and high density polyethylene (HDPE) materials. Lewatit TP-207, a weakly acidic, macroporous-type ion exchange resin with chelating iminodiacetate groups packed into a home-made minicolumn (glass, 3.5 cm length and 0.3 cm i.d.) was used for the on-line preconcentration of the bismuth(III) ions.

### 2.2. Reagents and standard solutions

All the chemicals were of analytical reagent grade and provided by Merck (Darmstadt, Germany). Deionized ultra pure water was used for the preparation of the solutions. 1000 µg mL<sup>-1</sup> Bi(III) was prepared by dissolving 0.2321 g of Bi(NO<sub>3</sub>)<sub>3</sub>·5H<sub>2</sub>O in a 100 mL volumetric flask using 3 mol L<sup>-1</sup> HNO<sub>3</sub> to prevent hydrolysis of the Bi(III) ions. Dilute working solutions were prepared by appropriate dilutions of the stock solution of Bi(III) with 0.1 mol L<sup>-1</sup> HNO<sub>3</sub> just before use [12,22]. Nitric acid was also used to adjust the acidity of the solutions and used as eluent throughout the experiments.

Lewatit TP-207 resin with iminodiacetate group was used (Fluka, Buchs, Switzerland) as the solid phase extractant. In order to remove organic and inorganic contaminants, the resin was washed with distilled water, 1 mol L<sup>-1</sup> HNO<sub>3</sub> in acetone and distilled water, respectively. The glassware used was cleaned by soaking overnight in dilute HNO<sub>3</sub> (1:5, v/v), and then rinsed with distilled water several times.

### 2.3. Preparation of the minicolumn

The minicolumn was prepared by introducing 135 mg of the chelating resin, as a slurry of the polymer beads using a syringe. The ends of the column were fitted with glass wool to retain the packing material. Before use, ethanol, acetone, 5% (w/v) nitric acid solution and deionized water were passed through the column at a flow rate of 3.0 mL min<sup>-1</sup> in order to clean it. The resin bed was approximately 3.5 cm length. The packed column was washed with blank solutions to condition the Lewatit TP-207 resin. After each elution, it was automatically pre-treated with deionized water and buffer solution, respectively.

### 2.4. On-line preconcentration system

The performance of the on-line preconcentration method was tested with model solutions before its application to the samples. The schematic diagram of the programmable on-line system was illustrated in Fig. 1. In the preconcentration step, PP1 and V1 are active while PP2 and V2 are inactive, and the sample and/or standard solutions acidified properly with 0.1 mol L<sup>-1</sup> HNO<sub>3</sub> were continuously passed through the minicolumn (MC) for 3.4 min at a flow rate of 7 mL min<sup>-1</sup>. The bismuth was retained on the minicolumn while the effluent was sent to waste. Afterwards, the minicolumn was washed with water in order to remove matrix ions from the resin. At this period, PP1 and V2 are active while PP2 and V1 are inactive. Finally, in the elution step; the eluent (E), 3 mol L<sup>-1</sup> HNO<sub>3</sub>, was aspirated by the PP2 at a flow rate of 7.2 mL min<sup>-1</sup>. During the elution, PP1 is inactive. The released metal ions are directly

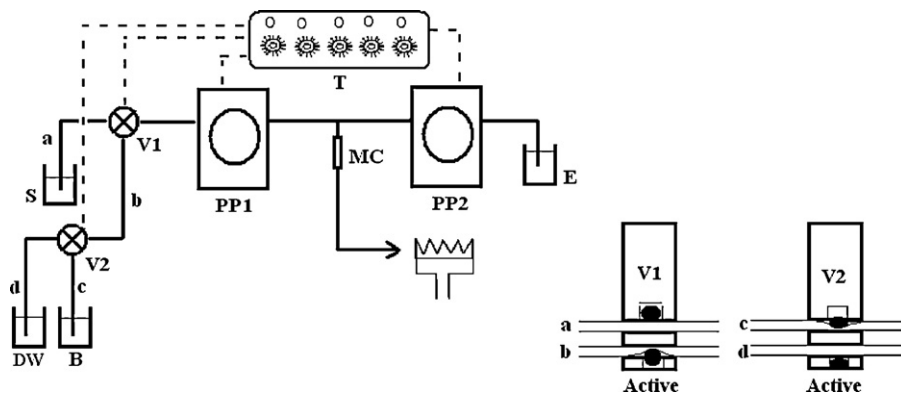


Fig. 1. Schematic diagram of on-line preconcentration system (V1: valve 1, V2: valve 2, PP: peristaltic pump, E: eluent, MC: minicolumn, T: timer, and a–d: solution ways; S: sample, B: buffer, and DW: deionized water).

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