



Chemical vapor generation of bismuth in non-aqueous phase based on cloud point extraction coupled with thermospray flame furnace atomic absorption spectrometric determination



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ABSTRACT

A novel method for the chemical vapor generation (CVG) of bismuth in non-aqueous phase based on cloud point extraction (CPE) coupled with thermospray flame furnace atomic absorption spectrometric (TS-FF-AAS) determination was developed. The method is based on the complex of bismuth with ammonium pyrrolidine dithiocarbamate (APDC). When the temperature of the system was higher than the cloud point of Triton X-100, Bi-APDC complex was extracted into Triton X-100 and separation of the analyte from the matrix was achieved. Volatile species of Bi were effectively generated through reduction of the extraction organic phase with solid KBH_4 in non-aqueous phase. Some parameters that influenced CVG, extraction and subsequent determination were evaluated in detail, such as the concentrations of Triton X-100 and APDC, pH and extraction time, flow rates of carrier gas, as well as interferences. Under optimized conditions, the detection limit (LOD) for Bi was 8 ng mL^{-1} . Relative standard deviation for five replicate determinations of the standard solution containing 200 ng mL^{-1} Bi was 3.8%. The proposed method was successfully applied to the determination of Bi in natural water samples, ore samples and certified reference materials with satisfactory results.

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1. Introduction

Chemical vapor generation (CVG) technique has been widely used in atomic spectroscopy because of its distinguishing characteristics, such as efficient matrix separation, high transport efficiency, nearly full analyte atomization, and potential speciation analysis as compared with conventional pneumatic nebulization [1–3]. CVG methods based on the reduction of analyte acidified solution with sodium tetrahydroborate(III). However, elements of CVG are still few, which stimulate the development of new CVG systems for more elements. CVG of some transition and noble metal elements has been found, such as Cu, Fe, Co, Zn, Mn, Au, Ag, Pt and Pd [4–16]. The CVG efficiency of transition and noble metals is very low. Many of enhancement reagents have been used to improve the CVG efficiency of transition and noble metals, such as sodium diethyl-dithiocarbamate (DDTC), 1,10-phenanthroline, 8-hydroxyquinoline, cobalt ion and room temperature ionic liquids [17–27]. Some CVG schemes, such as hydride generation, carbonyl generation, oxide generation, chelate generation, Alkyl generation, halide generation and Ultraviolet photochemical mediated vapor generation have been also developed.

Bismuth compounds are used orally in human and veterinary medicine for antacid action and mildly astringent action in gastrointestinal disorders, including diarrhea, flatulence and ulcerative gastritis and colitis [28]. There have been several reported cases of nephrotoxic, neurotoxic and kidney damage symptoms attributable to the use of bismuth containing pharmaceuticals [29,30]. The concentration of Bi in environmental and biological samples is very low. In order to obtain reliable results, an efficient separation and preconcentration step is usually necessary prior to analysis. Some CVG methods have been reported for Bi analysis [31,32].

Traditional CVG of Bi was effectively generated through reduction of acidified analyte solutions with KBH_4 in water-phase. The present work was aimed to develop a novel CVG of Bi in non-aqueous phase based on cloud point extraction (CPE) coupled with TS-FF-AAS determination. To the best of our knowledge, solid-liquid CVG in non-aqueous phase based on Triton X-100 as the extractant and the reaction medium has not been reported. The effect of concentrations of Triton X-100 and APDC on the CVG of Bi, variables of the CVG and TS-FF-AAS, and interference of coexisting ions were investigated in detail. The proposed method possesses the advantages of low sample and reagent consumption, green and environmental friendly, high sensitivity, good precision and fine selectivity. The proposed method was successfully applied to the determination of Bi in natural water samples, ore samples and certified reference materials with satisfactory results.

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2. Experimental

2.1. Instrumentation

A model TAS-990 flame atomic absorption spectrometer equipped with deuterium lamp background correction (Beijing Purkinje General Instrumental Co., China) was used for the determination of Bi. Hollow cathode lamp of Bi (Ningqiang Source, Hengshui, China) was used as the radiation source at 223.1 nm. A HH-S thermostated water bath (Jintan, Medical Instrument Corporation, Jiangsu, China) was used to heat up the sample solution. The pH values of samples solution were determined by PHS-3E pH meter (Leici Instruments, Shanghai, China). A 25- μL microsyringe (Anting Corporation, Shanghai, China) was used for transferring sample solution. The schematic diagram of the CVG-TS-FF-AAS system was illustrated in Fig. 1. The TS-FF-AAS system was the same as our previous report [33].

2.2. Reagent and materials

The stock standard solution of Bi(III) (1 mg mL^{-1}) was purchased from the National Center for Reference Materials (Beijing, China). Working standard solutions were obtained by serial dilution of the stock standard solutions. All chemicals used were at least of analytical-reagent grade and all solutions were prepared with double distilled water (DDW). 0.02% (m/v) APDC solution was prepared by dissolving appropriate amounts of APDC in DDW. Non-ionic surfactant Triton X-100 (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) was used as extractant. APDC, KBH_4 , sodium acetate (NaAc) and acetic acid (HAc) etc. used in this experiment were all obtained from Lantian Chemical Reagent Corporation (Nanning, China).

2.3. Cloud point extraction procedure

For CPE preconcentration, 50 mL analytical solution containing Bi ion and proper amount of chelating agent APDC were mixed in a 50 mL centrifuge tube. After adjusting the pH 5.0, the proper amounts of Triton X-100 were added into the centrifuge tube. After shaking the centrifuge tube, the analytical solution was heated in a thermostated water bath at 90°C for 15 min. After heated, the resultant solution was subjected to centrifugation at 4000 rpm for 5 min for phase separation, and then cooled in an ice-water bath for 5 min to increase the viscosity of the surfactant-rich phase. The water phase was carefully removed with a syringe, and the surfactant-rich phase was collected into a 25 μL microsyringe for the CVG-TS-FF-AAS.

2.4. Procedure of CVG

The schematic diagram of the CVG-TS-FF-AAS system was shown in Fig. 1. Solid of KBH_4 was placed at the bottom in the reaction tube. The hydrophobic rich phase was injected into the bottom of reaction tube with KBH_4 reaction for the CVG of Bi by a 25 μL microsyringe. Solid-liquid CVG based on Triton X-100 as the extractant and the reaction medium in the non-water phase were achieved. The generated volatile species of Bi was swept into the TS-FF-AAS atomizer with the aid of the carrier gas for the determination. The operating conditions of TS-FF-AAS for Bi were lamp current: 3 mA; spectral bandpass: 0.2 nm; acetylene flow rate: 1.3 L min^{-1} .

2.5. Sample preparation

Certified reference materials of GBW (E) 080135 (water sample) and GBW07284 (ore sample) were purchased from the Institute of Reference Materials of State of Environment Protection Agency (Beijing, China), which were employed to validate the accuracy of the proposed method. Certified reference materials of 0.1005 g ore sample GBW07284 were accurately weighed and dissolved with 10 mL aqua regia in digestion tank. Microwave digestion was chosen for decomposing the certified reference sample and the digestion procedure was based on a reference method [34]. The heating program was 33% power for 3 min, 55% power for 5 min, 100% power (700 W) for 3 min, and 77% power for 3 min. The residues were transferred into a 50 mL centrifuge tube, appropriate amounts of APDC and Triton X-100 were added and the solution was made up to volume with DDW. The pH of samples was adjusted to 5.0. The samples were ready for CPE. Pond water and river water were obtained from Tiannan Pond in the campus of Yulin Normal College and Nanliu River (Yulin, China), respectively. Industrial waste water was taken from the sewage system of the industrial zone in Yulin (China). All the samples were immediately filtered with a $0.45 \mu\text{m}$ membrane filter, then the samples were stored at 4°C .

3. Results and discussion

3.1. Effect of APDC

The chelating concentration was very important to the CPE efficiency and subsequent CVG-TS-FF-AAS determination. The effects of the concentration of APDC on the absorbance of 200 ng mL^{-1} Bi was studied. The results were shown in Fig. 2. It can be seen that the absorbance of Bi increased with the increasing of the concentration of APDC from 0.002% to 0.01% (m/v). Then, the absorbance of Bi remarkably

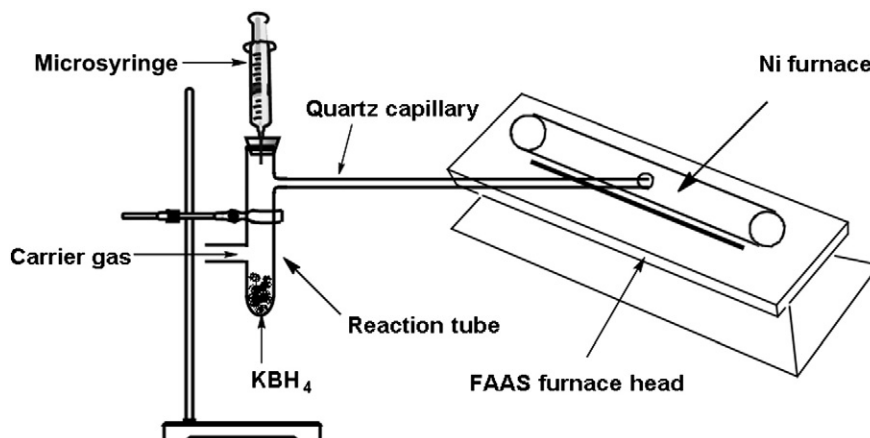


Fig. 1. Schematic diagram of the CVG-TS-FF-AAS system.

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