



Non-aqueous phase hydride generation and determination of trace bismuth by atomic fluorescence spectrometry

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ARTICLE INFO

Article history:

Received 18 September 2017
Received in revised form 17 November 2017
Accepted 17 November 2017
Available online 21 November 2017

Keywords:

Non-aqueous phase hydride generation
Solid reductant
Atomic fluorescence spectrometry
Rapid cloud point extraction
Bismuth

ABSTRACT

Hydride generation (HG) of bismuth was accomplished in non-aqueous media by using solid reductant of potassium borohydride (KBH_4) as a derivation reagent. The riched micelle phase of surfactant Triton X-114 was used as the non-aqueous media for the chemical vapor generation (CVG), and atomic fluorescence spectrometry (AFS) was used for the elemental determination. The analyte ions were firstly extracted into the non-aqueous media from the bulk aqueous phase of analyte/sample solution via a rapid cloud point extraction (RCPE) process and then directly mixed with the solid reductant KBH_4 to generate volatile bismuth hydride in a specially designed reactor, which was then rapidly transported to a commercial atomic fluorescence spectrometer for detection. Under the optimal conditions, the limit of detection (LOD) for bismuth was $0.08 \mu\text{g/L}$. Compared to conventional HG-AFS, the efficiency of non-aqueous phase HG accomplished using solid reductant and the analytical performance of the developed method was considerably improved.

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1. Introduction

As one of the most efficiency and important methods for trace and ultra trace elemental analysis, atomic fluorescence spectrometry (AFS) plays an important role with many advances in instrumentation and novel applications [1]. Traditional hydride generation atomic fluorescence spectrometry (HG-AFS) has been widely applied and developed in the field of atomic spectrometry analysis [2,3,4,5,6,7]. It is also an important component of the chemical vapor generation (CVG) system, which is an elegant means of sample introduction provides unique advantages including efficient matrix separation, enhanced selectivity, reduced interferences, increased precision, improved efficiency of sample introduction and detection limits [8,9,10,11]. As we presented in our earlier research work [12], to the best of our knowledge, apart from the Grignard alkylation for the derivatization of organo-tin, alkyl-lead and antimony species, direct CVG of volatile species of analyte from organic extraction phase with sufficient yield has rarely been realized [13, 14,15]. Almost all the hydride generation was carried out in the aqueous phase. The purpose of our work is to explore the direct CVG including HG in non-aqueous media and combine the proposed non-aqueous phase CVG/HG system with elemental analysis instruments, including AFS, atomic absorption spectrometry (AAS), and even mass spectrometry (MS).

Such research is an innovative extension and meaningful supplement for CVG or HG carried out in traditional aqueous media. Compared with the traditional methods, the new method realizes chemical vapor or hydride generation in a specially designed reactor and provides several unique advantages over conventional systems such as direct generation of volatile species from organic media, rapid separation of volatile species, low sample consumption, alleviation of matrix and spectral interferences, elimination of the gas-liquid separator, sample dilution and tedious sample preparation after microextraction. It is a basic research work to explore what elements, which organic media, or which instrument analysis methods have such feasibility to be applied in the proposed system.

In this work, nonionic surfactant Triton X-114 was firstly used as the non-aqueous media for the HG of bismuth. This study on the conditions of non-aqueous media extended the research of non-aqueous phase CVG from the ionic liquids [12] and several traditional organic extractants [16] to a new non-aqueous media environment. It is one of the important research contents to investigate which elements could be determined by the new system. In this project the non-aqueous CVG-AFS was first extended to the determination of bismuth after the detection of mercury, arsenic and antimony by non-aqueous CVG-AFS system in our previous research [12,16]. It was very interesting that, the pre-experiment found that some traditional organic reagents including tetrachloromethane, chloroform and petroleum ether were almost impossible to work as non-aqueous media for bismuth. The effect was very bad when using octanol. The phenomenon was different from the determination of mercury by non-aqueous phase CVG-AFS,

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which was reported in our previous work [16]. It was found the surfactant Triton X-114 had good performance in the non-aqueous phase HG system, possibly due to the sensitization effect of surfactant in the traditional HG [17,18].

In addition, it is worth mentioning that as an extraction method for transferring and enriching the target elements from aqueous phase to organic phase media, it is also an important part of this work. In this research, there is some innovation in the extraction method besides the non-aqueous media and the target element. Since the surfactant was selected as the non-aqueous media, cloud point extraction (CPE) naturally became the chosen extraction pattern. Compared with traditional CPE accomplished in water bath after long heating, incubation and cooling time [19,20], the established rapid cloud point extraction (RCPE) was fast, convenient and efficient. In this work, ascorbic acid worked as cloud point revulsant decreasing the cloud point temperature (CPT) of Triton X-114 significantly so as to achieve the rapid CPE at room temperature (about 25 °C). The ultrasound treatment process for 10 min at 25 °C was adopted to assist and accomplish the extraction. Compared to our previous RS-CPE [21,22,23,24,25], the revulsant octanol was replaced by ascorbic acid, which was relatively environmentally friendly.

The main parameters influencing extraction and determination were investigated in detail. The characteristics and analytical performance of the established non-aqueous phase HG-AFS were compared to traditional HG-AFS, which were described below.

2. Experimental

2.1. Apparatus

A commercial hydride generation non-dispersive atomic fluorescence spectrometer (HG-AFS) (AFS-3100, Beijing Kechuang Haiguang instrument Co., Beijing, China) was used for quantification. It should be noted that the accessories of the peristaltic pump and gas-liquid separator (GLS) were replaced with a specially designed reactor for non-aqueous phase HG in this work (Fig. 1). The instrument was optimized daily and typical conditions for the determination of Bi are summarized in Table 1. Atomic fluorescence signal was recorded with peak area

Table 1

Optimal instrumental parameters for the AFS.

Parameter	Optimal condition
Lamp current, mA	80
Voltage for photomultiplier tube, V	–300
Carrier argon flow rate, mL/min	400
Hydrogen flow rate, mL/min	120
Shield argon flow rate, mL/min	1000
Atomization mode	Ar/H ₂ flame atomization
Quantification mode	Peak area

mode and Ar/H₂ flame atomization was adopted during the measurements. Argon containing about 20% H₂ was used as carrier gas to light the Ar/H₂ flame.

An ultrasonic cleaner with temperature control Model SB5200DT (Ningbo Xinzhi Biotechnology Co., Ltd., Ningbo, China) was used for RCPE.

The pH values were measured by a pH-meter Model pH-25 (Shanghai Hongyi Instrumentation Co., Ltd., Shanghai, China).

A centrifuge Model 80–2 (Jiangsu Jintan Yitong Electronic Co., Ltd., Jiangsu, China) was used for phase separation.

A laboratory pure water system Model DZG-303A (Chengdu Tangshi Kangning Science and Technology Development Co., Ltd., Chengdu, China) was used to prepare ultra pure water.

2.2. Reagents

Bismuth standard solution (1000 mg/L) was purchased from National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials (Beijing, China). Working standard solution was obtained daily by stepwise dilution from standard stock solution in ultra pure water. Non-ionic surfactant Triton X-114 (TX-114) (Sigma-Aldrich) was used as extractant. Dithizone (The Sixth Factory of Chemical Reagent, Tianjin, China) was used as the chelating reagent. Thiourea and ascorbic acid (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) were used as reductant of bismuth and cloud point revulsant of Triton X-114. KBH₄ (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China)

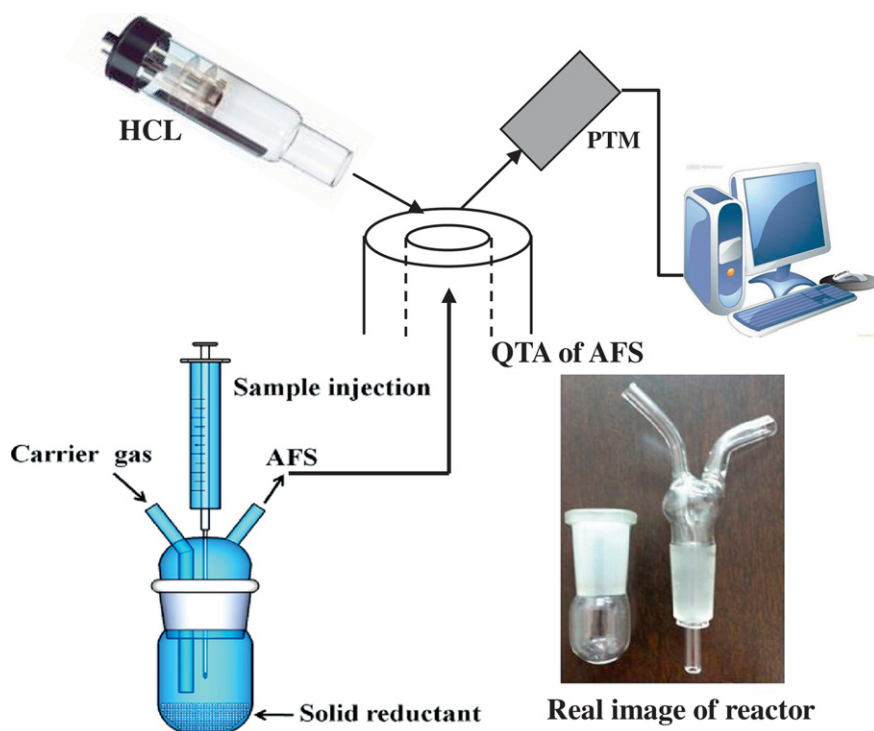


Fig. 1. Schematic of the non-aqueous phase HG-AFS.

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