

Investigation of organic media and surfactant sensitization in non-aqueous phase hydride generation-atomic fluorescence spectrometric determination of antimony



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ABSTRACT

This work was the expansion and in-depth study based on the previously established non-aqueous phase HG system. Aiming at the determination of antimony by the developed non-aqueous phase HG, the effect of different organic media and the sensitization effect of surfactant on the non-aqueous phase HG reaction were investigated. As an important condition, organic media has an important influence on this method. This study found that the surfactant Triton X-114 had very good sensitization effect for non-aqueous HG reaction. The sensitization effect of surfactant in different organic media was different. Among the investigated media, the comprehensive performance of octanol was the best under the sensitization effect of surfactant. Compared with the analytical performance of not adding surfactant, the sensitization factor of the surfactant was 1.55. The sensitization effect of surfactant Triton X-114 on the HG reaction in the octanol media was very significant, which greatly improved the analytical performance of the non-aqueous HG method. Under the optimal conditions, the limit of detection (LOD) was 0.04 µg/L. Compared to conventional HG-AFS, the efficiency of non-aqueous phase HG and the analytical performance of the developed method was considerably improved.

1. Introduction

As an efficient sampling technology, chemical vapor generation (CVG) has been widely applied and studied in the field of atomic spectrometry analysis [1,2]. Common CVG methods include hydride generation (HG) [3,4], photochemical vapor generation (PVG) [5–7], alkylation generation [8,9], carbonyl species generation [10,11], halide generation [12,13], oxide generation [14,15] and chelate chemical vapor generation [16,17], etc. Borohydride [18,19] and stannous chloride [20] is the most commonly used derivatization reagent (reductant) in CVG or HG. After acidification treatment, analytes in samples react with reductant in aqueous solution to generate volatile species, which is transferred into the atomizer by carrier gas to detect atomic absorption, emission or fluorescence signal. Some research groups have reported immobilizing the reductant (KBH₄) on the surface of the carrier to generate chemical vapor [21–23]. However, the aqueous solution injection determined that such chemical vapor generation was reaction in aqueous phase essentially. To the best of our knowledge, apart from the Grignard alkylation for the derivatization of organo-tin [24,25], alkyl-lead [26] and antimony [27] species, direct CVG of volatile species of analyte from organic phase with sufficient yield

has rarely been realized. Almost all the CVG or HG was carried out in the aqueous phase. Therefore, non-aqueous phase CVG or HG has been put forward and established recently by our group [28–30] and combined with elemental analysis instrument, including atomic fluorescence spectrometry (AFS) and potentially will be extended to atomic absorption spectrometry (AAS) and mass spectrometry (MS).

Non-aqueous phase CVG is dedicated to produce chemical vapor directly in non-aqueous media. The reaction pattern is to directly add solid reductant such as KBH₄, SnCl₂ and LiAlH₄ into organic phase after extraction (transferring) of target metals. Such reaction mode has the advantages of simple, direct and efficient, which is the innovative extension and complement of traditional CVG or HG in aqueous solution. Compared with the traditional method, the non-aqueous phase system has unique advantages due to its reaction in a specially designed CVG or HG reactor, such as reducing waste generation, elimination of the gas-liquid separator, alleviation of some interference in the process of gas-liquid separation and so on. It is necessary for basic research to carry out a series of studies to explore which elements, organic phase media or analytical instruments have the feasibility in this developed method.

In the previous work, mercury (Hg), arsenic (As), antimony (Sb) and bismuth (Bi) have been successfully detected by the new method, which

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were transferred from water solution into organic media by different extraction methods and determined by non-aqueous phase HG-AFS [28,30]. In addition to extend the new elements that can be applied to the system, analytical performance of different organic phase media has been studied in detail as one of the key factors influencing non-aqueous phase CVG [29]. The study shows that the influence of organic media is the condition worth studying intensively in the non-aqueous CVG system. The current work is also a kind of expansion and in-depth study after the establishment of the non-aqueous phase HG system. Based on the application of non-aqueous phase system to the determination of antimony, the effect of organic media and the surfactant sensitization on non-aqueous HG reaction was studied. The study found that the surfactant Triton X-114 had very good sensitization effect for non-aqueous HG reaction and such effect in different organic media was different. Through the investigation, the comprehensive performance of octanol was the best under the surfactant sensitization. In addition, as a target element in this work, antimony itself is also a very toxic heavy metal element worth studying. Antimony stimulates the human eye, nose, throat and skin. Continuous contact can disrupt the function of the heart and liver. Inhalation of high content of antimony can lead to antimony poisoning. Symptoms include vomiting, headache, and dyspnea, which may lead to death. Antimony and many of its compounds are toxic, and the mechanism of action is to inhibit the activity of the enzyme. The toxicity of Sb (III) is larger than that of Sb (V). This research improved the analytical performance of antimony determination, and can be potentially extended to other elements.

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 Sb are summarized

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	300
Hydrogen flow rate, mL/min	120
Shield argon flow rate, mL/min	850
Atomization mode	Ar/H ₂ flame atomization
Quantification mode	Peak area

in Table 1. Atomic fluorescence signal was recorded with 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 Ultrasound-assisted dispersive liquid-liquid microextraction (UA-DLLME).

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

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

Sb (III) standard solution (1000 mg/L) was purchased from National Center of Analysis and Testing for Nonferrous Metals and Electronic Materials (NCATN, Beijing, China). Working standard solution was prepared daily by stepwise dilution of the standard stock solution using ultra-pure water. Ammonium pyrrolidinedithiocarbamate (APDC, Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) was used as the chelating agent for antimony. KBH₄ (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) was used as solid reductant in non-aqueous phase HG. Other chemical reagents were all of analytical grade. Ultra-pure water was used to prepare solutions throughout the study.

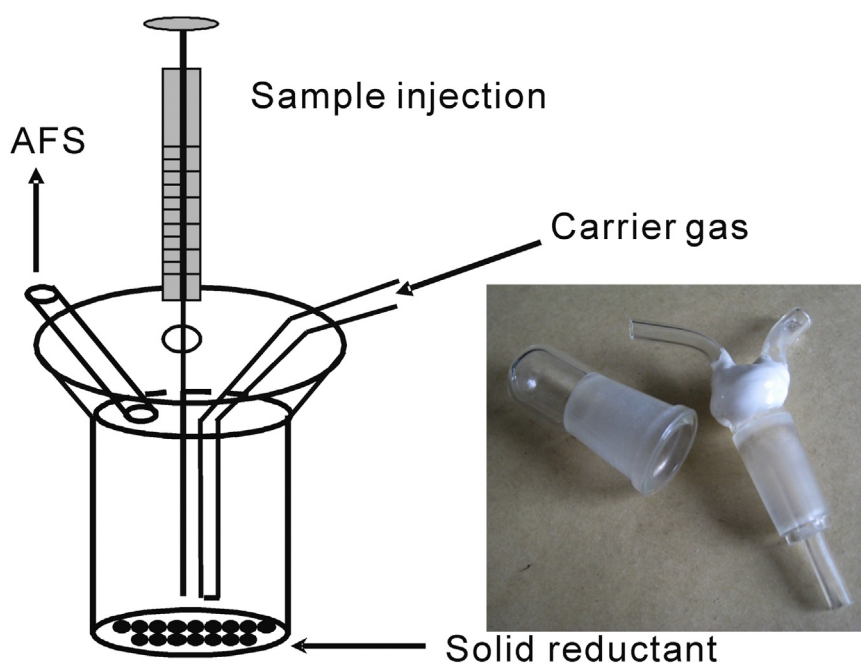


Fig. 1. Schematic of the non-aqueous phase HG-AFS.

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