



In-situ trapping arsenic hydride on tungsten coil and comparing interference effect of some hydride forming elements using different types of atomizers



Orkun Alp, Nusret Ertaş *

Gazi University, Faculty of Pharmacy, Analytical Chemistry Division, 06330 Ankara, Turkey

ARTICLE INFO

Article history:

Received 2 March 2016

Received in revised form 21 March 2016

Accepted 21 March 2016

Available online 20 April 2016

Keywords:

Arsenic
Hydride generation
Tungsten coil
Interference
Trap

ABSTRACT

Hydride generation atomic absorption spectrometry (HGAAS) with different types of atomizers (flame heated quartz T-tube and W-coil) was evaluated for comparison of interference effects of some hydride forming elements for arsenic determination. W-coil was used as an on-line atomizer or as a trapping surface. Mutual interferences of Sb(III), Se (IV), Sn (II) and Te(IV) on arsenic signal were compared with flame heated quartz T-tube atomizer and W-coil; on-line atomization and *in-situ* trapping modes. When W-coil was operated at on-line atomization mode the interference magnitudes could not be improved as much as expected with respect to quartz T-tube atomizer. However, decent improvements were achieved for interference magnitudes of each interferent species with *in-situ* trapping on iridium coated W-coil. Under optimized conditions and using 60 s trapping period (sample volume 6 ml), the limit of detection was found as 0.03 ng ml⁻¹ and the calibration was linear in between 0.25–4.0 ng ml⁻¹ As. The precision of the analytical method was less than 2.5% in terms of RSD (n = 11) for 1.0 ng ml⁻¹ arsenic concentration. The accuracy of the method was tested with certified reference materials; fortified water TMDA 61 (NWRI) and waste water EU-L-1 (SCP). The results were in a good agreement with the certified values at 95% confidence level.

© 2016 Elsevier B.V. All rights reserved.

1. Introduction

Arsenic (As) is a well-known toxic heavy metal for living organisms. Because of its toxicity arsenic determination at trace and ultra-trace levels is of interest in biological and environmental samples. Atomic techniques such as inductively coupled plasma mass spectrometry (ICP-MS), electrothermal atomic absorption spectrometry (ETAAS) are the first choice to determine arsenic at trace and ultra-trace levels in various matrices. Although these techniques are highly sensitive, generally they suffer from the interferences. Hydride generation atomic absorption spectrometry (HGAAS) is a well-known method which can offer comparable sensitivities and limit of detection with above mentioned methods. In addition HGAAS can eliminate the interferences by separating the analyte from the matrix components during hydride generation step. Various approaches showed that the generated hydrides could be either directly transferred into an atomizer or to a trapping surface prior to atomization. *In-situ* trapping methods which the atomizer used as both collection surface and as an atomizer are drawn attention because of providing higher sensitivities resulting in lower limits of detection values and especially eliminating the interferences in the vapor phase [1].

In-situ trapping of arsenic hydride using a pre-heated graphite tube prior to the atomization was first shown by Drash et al. [2]. Sturgeon et al. used a quartz capillary for the introduction of arsenic hydride into the graphite tube for the determination of arsenic [3]. In the last decade, the need of developing low cost and portable instruments, using tungsten coils (W-coil) or tungsten tubes seem to be an alternative to graphite furnaces in atomic absorption spectrometry [4,5]. The advantages of W-coil over the graphite tubes are; works with a simple power, the heating rate of W-coil is approximately ten times faster than graphite tube and there is no need for an external cooling system. Also it can be easily adapted to portable instrumentation [6]. Besides that, W-coil can also be used as either an on-line atomizer or a trapping surface [7–9].

There are several studies using graphite tube [1,10] for *in-situ* trapping of hydride forming elements, however studies on using W-coil as a trap surface is rather limited. Docekal and Marek [11] determined Se and As by *in-situ* trapping in W-tube atomizer and the trapping efficiency was improved by coating the trapping surface with Pt, Ir and Re. Selenium hydride trapped on a Rh coated W-coil and the selenium signal did not change significantly up to 300 measurements [12]. Instead of *in-situ* trapping, Cankur et al. trapped bismuth hydride at a temperature of 270 °C on W-coil which was placed in the inlet arm of a quartz tube; then trapped species were volatilized and transported to flame heated quartz tube atomizer [13]. Also the same instrumental setup was used

* Corresponding author.

E-mail address: nertas@gazi.edu.tr (N. Ertaş).

for trapping Se, Sb and Cd hydrides [14–16]. Se and As in biological and water samples were determined by *in-situ* hydride trapping on a resistively heated W-coil [17]. Liu et al. [18] used W-coil for speciation of inorganic arsenic species in water samples with hydride generation atomic fluorescence spectrometry.

Hydride generation comprises of analyte separation from the matrix components. This results in improved sensitivities and especially suppression of interferences during atomization. However, volatile species can affect the analyte signal in the gaseous phase. The gaseous phase interferences can take place either during the transport of hydrides or in the atomizer and the magnitude of the interference which occurs in the atomizer depends on the type of atomizer, so that the atomization mechanism [19]. Lajunen et al. [20] used flame in tube atomizer to evaluate the interference effect of some hydride forming elements on antimony signal and concluded that the interferences are related to the depletion of free hydrogen radicals. Dittrich and Mandry used externally heated quartz tube as atomizer and explained the interferences by formation of diatomic molecules (AsSb) [21]. Controversely, in another study the interferences occur in the atomizer were due to the analyte decay instead of radical mechanism [22,23]. Besides these observations the gaseous phase interferences are less pronounced in graphite tubes and diffusion flames [24–26].

The aim of this study is to determine arsenic by using resistively heated W-coil either as an online atomizer or as a trap surface and investigate the mutual interferences of some hydride forming elements. Also another purpose of this study is to compare the interference tolerance of different atomizer types such as W-coil and flame heated quartz T-tube atomizer. To the best of our knowledge this would be the first study using different types of atomizers to compare the interference effects of some hydride forming elements on arsenic signal.

2. Experimental

2.1. Instrumentation

A Unicam 939 atomic absorption spectrometer equipped with a deuterium background corrector was used throughout the experiments. An arsenic hollow cathode lamp (Unicam) was operated at 193.7 nm, with 9 mA and 0.5 nm spectral band pass. The hydride generation (HG) system consists of, a quartz T-tube atomizer heated by air-acetylene flame. It has an optical path length of 120 mm and an id of 8.0 mm; the inlet arm was 80 mm in length and 3.0 mm in id. The W-coil was obtained from 150 W-15 V projector bulb (Osram), was placed in the middle of the glass cell from an open hole, sealed and fixed by play dough. The glass cell has an optical path length of 120 mm and an id of 13 mm, the inlet arm was 40 mm in length and 4.0 mm in id. The W-coil temperature was manually controlled by a variable potential power supply and a transformer. The transformer was used to obtain a 24 V output from main electricity (220 V ac). The trapping temperatures of W-coil were measured with a Pt/Rh thermocouple (TETCIS, Ankara) which enables measuring temperatures up to 1800 °C. The atomization temperatures were measured with a portable T1315 series pyrometer (TIME, Beijing, China) which was capable of measuring temperatures between 500 and 3000 °C. During the trapping temperature measurements, the tip of the thermocouple was kept 1 mm in front of the central part of the W-coil, while a gas mixture of Ar and H₂ was flowing with an optimized composition and flow rate. Therefore, gas phase temperatures were measured rather than the

surface temperatures of W-coil. On the other hand, the atomization temperatures were the exact surface temperatures of the W-coil, since the pyrometer was focused on the surface of the W-coil while measuring the temperatures. A calibration plot was constructed for the measured temperature versus the applied voltage.

2.1.1. Continuous flow HGAAS system

Continuous flow hydride generation system was constructed using Tygon peristaltic pump tubings with 1.85 mm id. The connecting tubings, reaction and stripping coils were made of PTFE with 0.8 mm id. Two peristaltic pumps were used. A three channel ALITEA VS 3 (Sweden) was used to pump the acidified sample and reducing agent at flow rates of 6 ml min⁻¹ and a Gilson Minipuls 3 (France) for draining the gas-liquid separator (GLS). A home-made cylindrical GLS was used. A three way connector was placed between GLS and atomization cell to introduce hydrogen gas to the atomizer for preventing oxidation of W-coil. The same continuous flow system was used with quartz T-tube atomizer as well. More detailed information about HGAAS system can be found in our previous studies [8,9].

2.2. Reagents

All working solutions were prepared in deionized water (18.2 MΩ·cm). Carrier gas Ar and H₂ were supplied from Habaş, Turkey. All reagents were of analytical reagent grade. Sample and standard solutions were prepared in HCl (Riedel de Haen) by appropriate dilutions. NaBH₄ (min. 96% purity, Aldrich) was used as reducing reagent and dissolved in 0.3% (w v⁻¹) NaOH (Merck) was used for stabilizing. Working As (III) standards were prepared by appropriate dilution of 1000 mg l⁻¹ As (III) (Fisher Scientific, USA) stock solution. In order to improve trapping efficiency of W-coil, 1000 mg l⁻¹ Ir (Accustandard) standard was used for coating. For the interference studies Sn (II) (High Purity Standards), Sb (III), Te (IV), Se (IV) (Fisher Scientific) standards were prepared by appropriate dilutions of stock 1000 mg l⁻¹ solutions. Certified reference materials TMDA-61, trace element fortified water obtained from NIWR (Canada), waste water, EU-L-1 from SCP (Canada) were used to test the accuracy of the proposed method.

2.3. Ir coating procedure

The coating was performed by manual injection of 25 µl aliquots of 1000 mg l⁻¹ Ir stock solution on the W-coil and the temperature program shown in Table 1 was applied. The procedure was repeated four times in order to assure the signal stability for longer time. During coating procedure, H₂ and Ar flow rates were 175 and 150 ml min⁻¹, respectively.

2.4. Atomizer types and general procedure

Two types of atomizers were used in this study; W-coil and quartz tube atomizer. Besides these specified atomizer types, W-coil was used either as an online atomizer by pre-heating the coil to an optimized atomization temperature or as a trapping surface. The same continuous flow manifold was used for both atomizer types (W-coil and quartz

Table 1
Temperature program for Ir coating of W-coil.

	Time, s	Temperature, °C
1. Step	45	175
2. Step	30	600
3. Step	2	1300

Table 2
Optimum parameters for continuous HG-AAS.

Parameter	
NaBH ₄ , % (m v ⁻¹)	0.75
HCl, mol l ⁻¹	1.0
Ir coating mass, µg	100
Trapping temperature, °C	400
Atomization temperature, °C	1900
H ₂ flow rate, ml min ⁻¹	175
Ar flow rate, ml min ⁻¹	150

Download English Version:

<https://daneshyari.com/en/article/1227548>

Download Persian Version:

<https://daneshyari.com/article/1227548>

[Daneshyari.com](https://daneshyari.com)