

Evaluation of tungsten coil electrothermal vaporization-Ar/H₂ flame atomic fluorescence spectrometry for determination of eight traditional hydride-forming elements and cadmium without chemical vapor generation

Peng Wu^a, Xiaodong Wen^b, Liang He^b, Yihua He^b,
Minzhu Chen^b, Xiandeng Hou^{a,b,*}

^a Analytical & Testing Center, Sichuan University, Chengdu, Sichuan 610064, China

^b College of Chemistry, Sichuan University, Chengdu, Sichuan 610064, China

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Abstract

A tungsten coil electrothermal vaporizer (W-coil ETV) was coupled to an Ar/H₂ flame atomic fluorescence spectrometer for the determination of eight traditional hydride-forming elements (i.e., As, Bi, Ge, Pb, Sb, Se, Sn, and Te) as well as cadmium without chemical vapor generation. A small sample volume, typically 20 μ L, was manually pipetted onto the W-coil and followed by a fixed electric heating program. During the vaporization step, analyte was vaporized off the coil surface and swept into the quartz tube atomizer of AFS for further atomization and excitation of atomic fluorescence by a flow of Ar/H₂ gas, which was ignited to produce the Ar/H₂ flame. The tungsten coil electrothermal vaporizer and Ar/H₂ flame formed a tandem atomizer to produce reliable atomic fluorescence signals. Under the optimal instrumental conditions, limits of detection (LODs) were found to be better than those by flame atomic absorption spectrometry (FAAS) or inductively coupled plasma optical emission spectrometry (ICP-OES) for all the nine elements investigated. The absolute LODs are better or equivalent to those by hydride generation atomic fluorescence spectrometry (HG-AFS). Possible scattering interferences were studied and preliminary application of the proposed method was also reported. © 2007 Elsevier B.V. All rights reserved.

Keywords: Tungsten coil; Electrothermal vaporization; Atomic fluorescence spectrometry; Hydride; Flame atomization; Cadmium

1. Introduction

Atomic fluorescence spectrometry (AFS) is a widely used atomic spectrometric technique in trace element analysis, particularly when coupled with hydride generation (HG) for sample introduction. Dědina and Tsalev [1] underlined that atomic fluorescence detection was able to improve the sensitivity by about one order of magnitude over atomic absorption spectrometry (AAS) and optical emission spectrometry (OES) detection, whenever these detection systems are coupled with comparable HG set-ups. In fact, commercialization of HG-AFS instruments has gained a success worldwide [2], especially in China.

Using HG, the current AFS instrument shows good sensitivity for hydride-forming elements. The equality or superiority of HG-AFS over other atomic spectrometric techniques for the determination of several hydride-forming elements, for example, arsenic, selenium and mercury, has already been well documented in the literature, especially as an element-selective detector for chromatographic separations [3]. However, the inherent drawbacks of HG, i.e. severe interference from transition metals and limited number of elements capable of forming volatile hydrides, largely hold back the wide applicability of this instrument. Although other elements may be determined by use of various chemical vapor generation techniques coupled to HG-AFS instrument, the number of detectable elements is still limited.

Electrothermal vaporization (ETV) has long been considered as an excellent sample introduction approach for analytical plasma spectrometry; however, this technique has rarely been

* Corresponding author at: Analytical & Testing Center, Sichuan University, Chengdu, Sichuan 610064, China.

E-mail address: [houxd@scu.edu.cn](mailto:houx@scu.edu.cn) (X. Hou).

expanded for other atomizers. With electrothermal vaporization, the sample introduction efficiency and absolute detectability, as compared with ICP-OES/MS using pneumatic nebulisation, are considerably increased. Graphite furnaces are the predominant ETV devices in analytical atomic spectrometry [4], but metals such as tungsten, tantalum and molybdenum have also been used [5–7]. Some advantages of tungsten coil vaporizers over graphite furnace vaporizers are obvious, i.e. freedom from carbide forming problems, a lower surface porosity, higher heating rate, much lower power supply, and compactness. A car battery can be employed as the power supply for tungsten coil atomizers [8], and this makes tungsten coil atomizer based portable atomic absorption spectrometer feasible for field use.

Tungsten coil ETV (W-coil ETV) has already been used as electrothermal vaporizers for inductively coupled plasma [9–11] and microwave plasma [12]. Because the sample vaporization occurs in the vaporizer, the plasma energy itself is not required to desolvate the sample so that more energy is available for atomization, ionization and excitation. Jones et al. [13] successfully developed a low-cost, modular W-coil ETV system coupled to ICP-OES for simultaneous determination of 10 elements. Picogram-level LODs were achieved and linear dynamic ranges (LDR) were 3–5 orders of magnitude. A W-coil ETV device has been developed for thermal separation of Pb from matrix elements, which may cause spectral interference, such as aluminium, manganese and iron, by use of a temperature ramp [14]. The W-coil ETV can also be applied to slurry sampling for vaporizing digestion-problematic samples, such as silicon carbide [15], silicon dioxide and silicon nitride powders [16,17], aluminium-based ceramic powders [18], and high purity quartz [19].

Tungsten coil has already been used as an atomizer in AFS [5,20,21]. Winefordner et al. [22] firstly utilized a tungsten wire loop as an atomizer for AFS. Samples containing Ag, Be, Bi, Cu, Mg, Pb, Tl, and Zn were atomized from the tungsten wire loop into an argon stream. Muzgin et al. [23] built a pulsed W-coil electrothermal atomizer for application in AFS and investigated its fundamental analytical characteristics. In all W-coil AFS applications, picogram or sub-picogram instrumental limits of detection for Cd, Pb, Bi and Te were reported [5]. Recently, tungsten coil atomizers have also found promising applications in laser-excited AFS [14,24]. However, W-coil has not been employed as sample introduction device for AFS, therefore, the purpose of this work is to introduce W-coil ETV to AFS to expand the number of detectable elements without chemical vapor generation. Analyte was firstly sputtered off the W-coil ETV and then swept into an argon–hydrogen flame for further atomization and excitation of atomic fluorescence of traditional hydride-forming elements and cadmium.

2. Experimental

2.1. Instrumentation

Fig. 1 shows the schematic diagram of the W-coil ETV-Ar/H₂ flame AFS instrument. A model AFS-2202 non-dispersive atomic fluorescence spectrometer (Beijing Haiguang Instrument

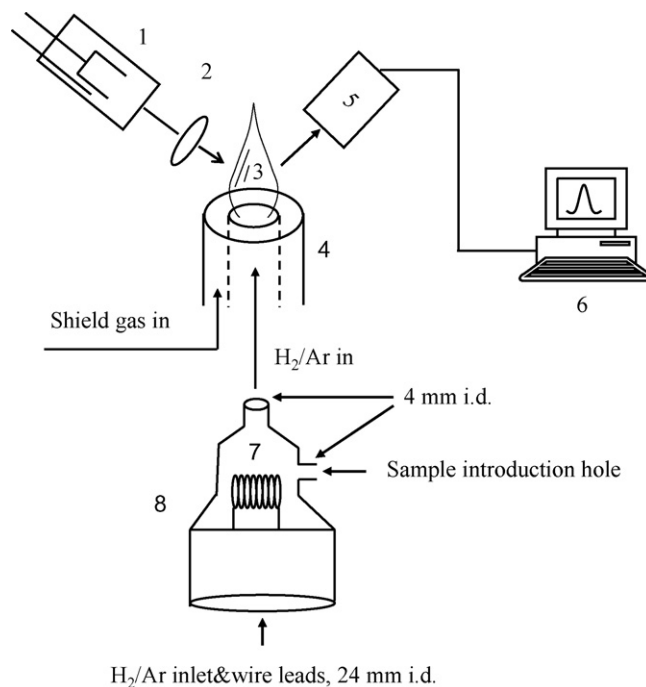


Fig. 1. Schematic diagram of the instrumental arrangement. 1: Hollow cathode lamp; 2: lens; 3: miniaturized Ar/H₂ flame; 4: QTA; 5: PMT and electronics; 6: personal computer; 7: tungsten coil; 8: vaporization cell.

Co., Beijing, China) was used in this work. The instrument is equipped with a programmable intermittent reactor and two gas–liquid separators by the manufacturer to facilitate hydride generation and separation, but all these accessories were replaced with a W-coil ETV in this work. Hollow cathode lamps (HCLs, Research Institute of Non-Ferrous Metals, Beijing, China) of conventional hydride-forming elements, namely As, Bi, Ge, Pb, Sb, Se, Sn and Te, plus Cd, were used as the radiation sources for these nine elements, respectively. These modulated high-intensity HCLs are specially designed for AFS. All HCLs were operated at 80 mA except Cd at 70 mA. The emission light beam of the HCL was focused into a quartz tube atomizer (QTA) for the excitation of atomic fluorescence. An electrically heated wire is coiled around the top edge of the QTA to ignite the carrier gas (Ar/H₂) for a stable flame on the top of the QTA for the further atomization and excitation. The resultant atomic fluorescence was detected by a “solar blind” photomultiplier tube (PMT, with its working voltage set at –300 V for all the nine elements), and followed by the amplification of the electric signal and data processing with a personal computer. With a total reading time of 6 s, the peak area of the temporal atomic fluorescence signal was used for quantification because of its good day-to-day repeatability. Other instrumental parameters were summarized in Table 1.

Tungsten coils are obtained from commercially available slide projector bulbs (HLX 64633, OSRAM, Munich, Germany) rated at 15 V and 150 W with a color temperature of 3000 °C at full power. The glass envelope was carefully removed away, and the bulb base was then fitted into a laboratory-constructed mount that connected the power supply and the purge gas tubing. A laboratory-made ETV glass cell was used to protect the

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