



## Design of a compact, aluminum, tungsten-coil electrothermal vaporization device for inductively coupled plasma-optical emission spectrometry

Summer N. Hanna<sup>a</sup>, Clifton P. Calloway Jr.<sup>b</sup>, Jason D. Sanders<sup>b</sup>, Ronald A. Neslon<sup>b</sup>, Jamaal Cox<sup>b</sup>, Bradley T. Jones<sup>a,\*</sup>

<sup>a</sup> Department of Chemistry, Wake Forest University, Winston-Salem, NC, 27109 USA

<sup>b</sup> Department of Chemistry, Physics, and Geology, Winthrop University, Rock Hill, SC, 29733 USA

### ARTICLE INFO

#### Article history:

Received 6 April 2011

Received in revised form 11 April 2011

Accepted 11 April 2011

Available online 16 April 2011

#### Keywords:

Inductively coupled plasma-optical emission spectrometry

Tungsten coil

Electrothermal vaporization

### ABSTRACT

A new compact, aluminum electrothermal vaporization cell was constructed for inductively coupled plasma-optical emission spectrometry analysis. This cell is compact enough to fit within the space occupied by the spray chamber and fits directly to the quartz torch without extraneous tubing through the use of a simple Compression fitting. Sample volumes as low as 10  $\mu\text{L}$  were analyzed with an automated control program for efficient vaporization. Twelve elements were analyzed utilizing a time resolved acquisition method so that real-time data could be generated over a period of 10 s with an average improvement factor of 14 for elements over a wavelength range of 193–445 nm.

© 2011 Elsevier B.V. All rights reserved.

### 1. Introduction

Sample introduction for inductively coupled plasma-optical emission spectrometry (ICP-OES) typically relies on large volumes of liquid analytes. Methods historically fall within two categories: pneumatic and ultrasonic nebulization, both of which depend on the use of a spray chamber to prevent large liquid droplets from entering the plasma. When using nebulization, approximately only 3–12% of the sample is introduced into the plasma, with the remainder being lost to waste [1]. This method often encounters problems due to the autosampler and copious amounts of tubing necessary for sample transport, which can introduce air bubbles into samples. Poor connections between components can also create problems in otherwise routine methods [2].

Because of these limitations, all sample introduction techniques will focus on electrothermal vaporization (ETV). This method has been used extensively for the introduction of slurries, powders, solid materials, and even complex biological matrices [3–6]. ETVs are often made from graphite, which can be expensive because of the material's required purity, inability to tolerate mass production, and resulting low demand for production [7]. A large thermal gradient often occurs and interferences may be seen when determining carbide-forming elements. Samples with a large concentration of surfactants may also interfere with measurements by depositing carbon on the torch [8].

Because of these disadvantages, metal atomizers are becoming increasingly popular since they are nonporous, can provide quick and efficient vaporization of a wide variety of materials, and do not require the elaborate power supplies that are necessary when using graphite ETVs.

Tungsten is particularly well suited for these types of applications since it has the highest melting point of any element and is found as a boat, ribbon, or coil [9–11]. Extensive work has been done with tungsten ETVs which are regarded as a reliable vaporization source. Rare earth elements, which are notoriously difficult to analyze because of their chemical similarities and tendency to form oxides, have been well characterized using tungsten as an ETV in inductively coupled plasma mass spectrometry (ICP-MS) analysis [12]. Complex matrices, such as human urine, have also been successfully quantified using tungsten coil ETVs when paired with permanent modifiers or extraction techniques [9,13].

Tungsten coils such as the ones used in the determination of cadmium in urine samples are a great alternative ETV source. These coils are made for use in projector light bulbs and can be purchased for as little as two dollars each since they are commercially mass produced, which ensures adherence to strict structural specifications. Their small size of less than 20 mm<sup>3</sup> allows for the use of sample volumes on a microliter scale. This reduction in volume provides users the opportunity to analyze extremely small-volume or precious samples while utilizing the multielement capabilities of ICP-OES. Tungsten coil ETVs in the past have used cells that did not connect directly to the torch of the ICP, but instead relied on centimeters of tubing. Tubing makes analyte loss possible through condensation of

\* Corresponding author at: PO Box 4937 Winston-Salem, NC 27109, USA. Tel.: +1 336 758 5512; fax: +1 336 758 4656.

E-mail address: [jonesbt@wfu.edu](mailto:jonesbt@wfu.edu) (B.T. Jones).

the “hot” analyte vapor on the walls of “cool” transport tubing [14]. To overcome potential loss, a new ETV cell design that connects directly to the quartz torch of the ICP through a simple Compression fitting has been used. The reduction in cell size and elimination of tubing provides an ETV sample introduction source that can fit within the space formerly occupied by a spray chamber and nebulizer. The ETV components can be replaced with a traditional sample introduction system in less than a minute, which allows the user to quickly alternate between sample introduction methods.

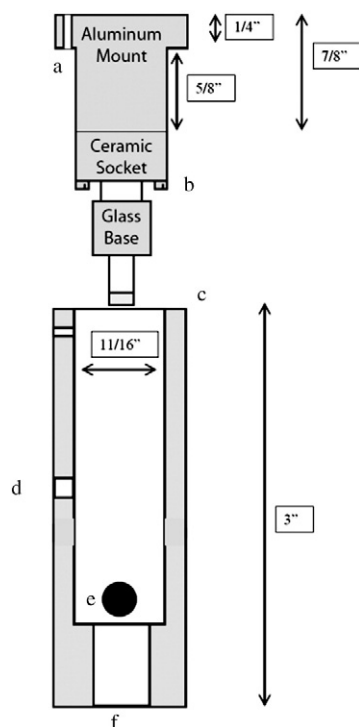
## 2. Materials and methods

### 2.1. Reagents

All standard solutions were prepared from their metal salts using 15.8 N trace metal grade nitric acid (Fischer Scientific, Pittsburg, PA, USA) and 18 M $\Omega$  distilled, deionized water (Milli-Q Systems, Millipore). Working solutions were prepared from NIST Standard Reference Material (SRM) 1643e, Trace Elements in Water, by simple dilution using distilled, deionized water and also utilized the standard additions method to mitigate matrix effects for analysis typically encountered with ICP-OES.

### 2.2. Instrumental

W-coil ETV-ICP-OES is composed of an aluminum electrothermal vaporization cell and the ICP-OES system. Fig. 1 shows a schematic diagram of the ETV-ICP-OES cell. In this system, a tungsten coil is extracted from a commercially available 150 W\*15 V projector light bulb (Osram HLX 64633 Xenophot, Augsburg, Germany). The filament is exposed by removing the silica envelope while leaving the base intact and is then mounted in the ETV cell's ceramic base. The ceramic base is secured using two screws to an aluminum mount. This mount has a 1" outer diameter and is 7/8" in length. The mount and socket fit



**Fig. 1.** Illustration of ETV cell. a) tapped hole for riser screw b) 2 screws to attach socket to mount c) 1" aluminum rod with 11/16" inner diameter to fit ceramic socket d) 1/8" hole centered and in line of sight of coil e) 1/16" NPT for gas fitting in back near bottom f) 1/4" NPT for glass outer joint fitting.

was flushed into an aluminum rod and used as a screw to create an airtight seal. This rod is 3" in length with an 11/16" inner diameter to accommodate the socket and mount. The arrangement of the socket on the aluminum mount allows for the W-coil to align with a 1/8" hole which is used for sample introduction. This opening is plugged with a screw and rubber o-ring to prevent oxidizing gases to enter the ETV cell. A 1/16" NPT for gas fittings is located on the back side of the cell and immediately following is a 1/4" NPT at the base of the rod which allows for the connection of a glass outer Compression joint fitting union (SS-4-UT-6-400, Swagelok, Solon, OH, USA). The compression fitting provides an immediate glass-to-glass connection to the ICP-OES quartz torch without use of extraneous tubing normally necessary in ETV-ICP-OES.

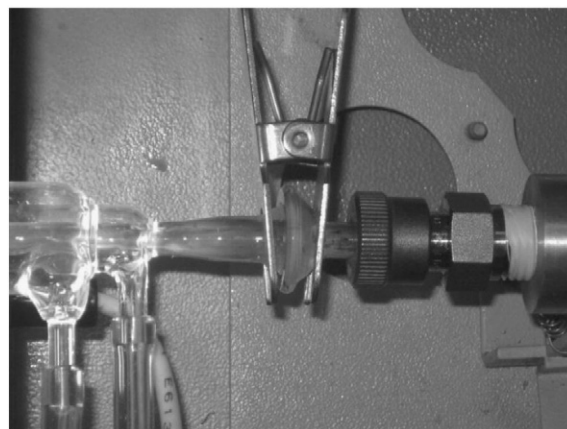
Fig. 2 depicts the joining of these two parts in a manner identical to the glass-to-glass connection that would be used for a spray chamber. This connection allows for the quick and easy replacement of a spray chamber with the ETV cell in less than a minute.

A Visual Basic program controls the coil drying and vaporization cycle while simultaneously initiating ICP spectra collection. The program automatically dries the coil by measuring the potential across the coil at a constant current (3.4 A). As the liquid sample evaporates the potential across the coil increases. The Visual Basic program runs a variable time loop, and every 0.5 s reads the voltage across the coil. Using this measured voltage, a slope is calculated using the formula:

$$m = \frac{(V_2 - V_1)}{0.5 \text{ seconds}}$$

The two most recent slope values are averaged and displayed on the screen. Once the voltage potential slope reaches 0.38 (value determined empirically), the coil is dry. The program then continues to a set of user defined values which vaporize the sample and trigger data collection. Step 2 in Table 1 at 8 A is vaporization and designated trigger step, which signals the ICP data software to begin collection of the analytical signal through the time resolved analysis feature [15].

The ETV connects directly to the ICP-OES torch. The axially arranged quartz torch, (ML155064, Meinhard Glass, Golden, CO, USA) is surrounded by three water-cooled radio frequency (RF) coils which uses a frequency of 40.68 MHz to generate an argon plasma. The analytical zone of the plasma is aligned with the 40  $\mu\text{m}$   $\times$  100  $\mu\text{m}$  entrance slit of the argon-purged optical system. The Prodigy High Dispersion ICP-OES (Teledyne Leeman Labs, Hudson, NH, USA) uses an 110 mm  $\times$  110 mm Echelle grating of 52.13 g/mm to focus (focal length (f), f= 800 mm) the analytical signal on a solid-state, Large Format, Programmable Array Detector (LPAD). This enables continuous coverage over a wavelength range of 165–1100 nm. The



**Fig. 2.** Photograph of the ETV cell and ICP torch connection.

Download English Version:

<https://daneshyari.com/en/article/10556826>

Download Persian Version:

<https://daneshyari.com/article/10556826>

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