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Dielectric barrier discharge micro-plasma emission source for the determination of lead in water samples by tungsten coil electro-thermal vaporization

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ABSTRACT

In this study, a fast and simple approach to directly determinate lead in water samples by a low power dielectric barrier discharge (DBD) excitation source was developed using tungsten coil electro-thermal vaporization (WC ETV) for liquid microsample introduction. A 20 μL sample was dropped onto the WC, and then the sample went through the drying, pyrolysis, subsequently the analyte was vaporized and swept directly into the dielectric barrier discharge micro-plasma for emission, and the whole process took only 3 min. The effects of operating parameters such as plasma gas flow rate, plasma input voltage, pyrolysis current, vaporization current and interferences from concomitant elements were investigated. Under the optimal conditions, the limit of detection (LODs, 3σ) was calculated to be $7.7 \mu\text{g L}^{-1}$. Repeatability, expressed as the relative standard deviation of the spectral peak height, was 4.6% ($n=11$) for 0.1 mg L^{-1} lead standard solution. The proposed method was successfully applied to the determinations of Pb in water samples.

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1. Introduction

The contamination of lead in water has become a problem of global concern [1]. Of all the heavy metals, Pb(II) is important to consider in terms of food chain contamination because of its toxicity [2,3]. Pb(II) ions causes both acute and chronic poisoning, exhibits adverse effects on the kidney, liver, heart, vascular and immune system. Moreover, Pb(II) exposure causes chromosome aberration, skin allergy, cancer and birth defects [4,5]. Hence, there is a great demand to develop a simple and reliable method for the determination of the level of lead in water on-site.

In this perspective a number of methods have been developed for the determination of Pb(II) the well-known high-performance spectrometric instruments, such as atomic absorption spectrometry (AAS) [6], atomic fluorescence spectrometry (AFS) [7], inductively coupled plasma atomic emission spectrometry (ICP-AES) [8] and inductively coupled plasma mass spectrometry (ICP-MS) [9] can perform fast, sensitive, and accurate lead determinations. However, these methods are generally performed at laboratories, requiring complicated equipment (e.g., high power, large inert or special gases, high vacuum). These disadvantages limit their applications

primarily to laboratory settings, and prohibit their use for rapid analyses under field conditions. Therefore, it is of much significance to develop compact and low-cost instruments [10], for in situ monitoring of Pb contamination in water.

In recent years micro-plasmas [11–15], embedded in portable analytical instrumentation, such as corona discharge, glow discharge, capacitively coupled plasma, and dielectric barrier discharge (DBD) [16] received increased attention for the development of portable instruments for elemental analysis because of their small size, low power consumption, cost effectiveness, and reduced inert gas requirement. The unique advantages of DBD include simple construction, low temperature and power consumption, and atmospheric operation [14]. However, direct introduction of liquid sample into the DBD micro-plasma for analytical atomic spectrometry can be a problem for its lowered excitation capability or can even extinguish it. Tombrink et al. [17] first introduced direct liquid analysis by using a capillary DBD, and Zhu et al. [18] developed a liquid-film DBD (LFDDBD). In their studies, they solved the liquid sampling problem to some extent. Nevertheless, residual or concomitant moisture and matrix from liquid sample introduction still restrict the analytical performance of DBD micro-plasma for AES. The vapor generation (cold vapor and hydride generation) was mostly employed in DBD-AES for the sample introduction since it improves the sample introduction efficiency and reduces the introduction of water. However, only a limited number of elements is applicable to

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CVG and the reported research mainly focused on mercury, arsenic and some nonmetals [11,15,19].

As well known, electro-thermal vaporization (ETV) has long been considered as an excellent sample introduction technique for analytical atomic spectrometry. It offers several advantages, including improved sample introduction efficiency, efficient separation matrix, requirement of small amounts of sample, and the capability to perform direct analysis of slurries and solids [20–27]. Most works on ETV employ graphite furnaces as electro-thermal vaporizers [22,24,26–28]. However, it requires heavy power supplies and water cooling systems, so they are seldom used in portable instrumentation [29]. A simpler and less expensive alternative is to use tungsten coil, which can be extracted from commercially available stage light bulbs. Because of their low mass and the specific heat of tungsten, tungsten coils (WC) present a faster heating–cooling regimen, which requires no complex bulky chillers. In addition, a simple low power source is enough to efficiently vaporize samples. Another advantage of WC vaporizers when compared to graphite furnaces is that carbon species are not formed from atomizer surface vaporization, which minimizes the formation of carbides [30,31]. In the past studies, tungsten coil has been widely used as atomizers in atomic absorption and atomic fluorescence spectrometry [32,33]. Meanwhile, it has also been employed as electro-thermal vaporizers in atomic emission [10,34,35], as for inductively coupled plasma [21,36] and microplasma [37–39]. For years, to the best of our knowledge, there are

no reports on the hyphenation of ETV with DBD microplasma emission source. Until more recently, Hou et al. [40] firstly coupled W-coil ETV with DBD-AES and applied it to the determination of Hg, Zn, and Cd.

In the present study, the feasibility of coupling W-coil ETV with DBD-AES for directly Pb determination was demonstrated. A tungsten coil extracted from commercially available stage light bulb was used to vaporize samples without any previous sample treatment. The analyte vaporized from the tungsten coil ETV was transported to the DBD source for excitation, and then the lead could be measured by atomic emission spectrometry. Several experimental parameters were optimized, and analytical figures of merit were evaluated. Both the WC ETV device and DBD system are simple, easily fabricated, and low cost of operation, it provided a possibility to design a portable, miniature, and mobile AES instrument, especially for field analysis of Pb with low-power supply.

2. Experimental

2.1. Instrumentation

The W-coil ETV–DBD-AES system consisted of an ETV generation unit for sample introduction and a DBD atomic emission system for Pb excitation. For ETV unit, the tungsten filament extracted from a commercially available halogen display lamp

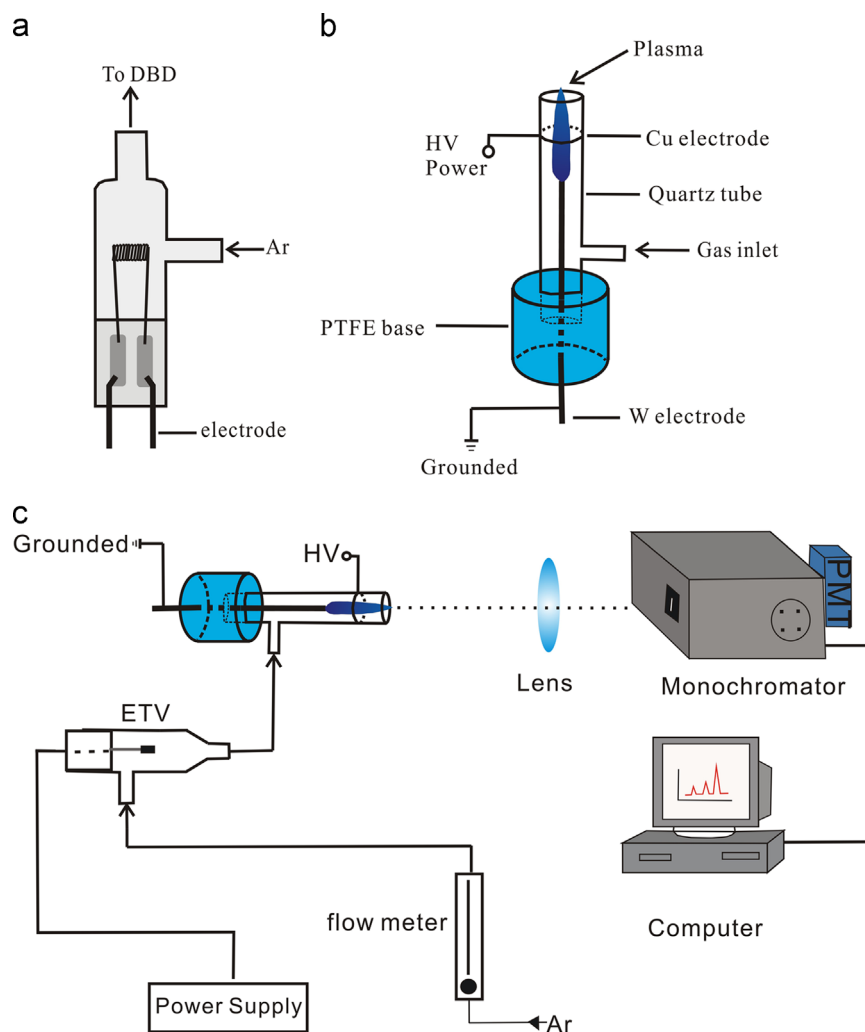


Fig. 1. The schematic diagram of the equipment. Vaporizer (a) and the DBD setup (b) and the whole instrument (c).

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