



An effective analytical system based on a pulsed direct current microplasma source for ultra-trace mercury determination using gold amalgamation cold vapor atomic emission spectrometry

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ARTICLE INFO

Article history:

Received 11 October 2013

Accepted 19 December 2013

Available online 2 January 2014

Keywords:

Microplasma

Mercury determination

Miniaturization

Gold amalgamation

Atomic emission spectrometry

ABSTRACT

A novel analysis system based on a low power atmospheric pressure pulsed direct current (Pdc) microplasma is described for the determination of ultra-trace mercury in natural water by cold vapor generation atomic emission spectrometry (CV-AES). The plasma was generated with a miniaturized home-built high-voltage Pdc power supply which decreased the volume and weight of the whole experiment setup. The CV-Pdc-AES system is based on the preconcentration of mercury vapor on a gold filament trapping micro-column prior to detection that provides fast, reproducible absorption and desorption of mercury. The micro-column is produced by winding 30 μm diameter 100 m long gold filament to a small ball and then insert it into a quartz tube of 6 mm i.d., 8 mm o.d. Under the optimized experimental conditions, the new system provides high sensitivity (detection limit: 0.08 pg mL^{-1}) and good reproducibility (RSD 3.0%, $[\text{Hg}] = 20 \text{ pg mL}^{-1}$, $n = 11$). The calibration curve is linear at levels near the detection limit up to at least 200 pg mL^{-1} and the accuracy is on the order of 1–4%. The proposed method was applied to 5 real water samples for mercury ultra-trace analysis. The advantages and features of the newly developed system include high sensitivity, simple structure, low cost, and compact volume with field portable potential.

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1. Introduction

Mercury pollution in natural water is recognized as a major environment issue and health hazard for humans due to its high toxicity and its accumulative character in the biota. It has become obvious that even very low concentrations of mercury in natural water, can be accumulated by a factor of up to 10^6 in fish and aquatic mammals [1]. Drinking water, fish and fish products are the main routes of incorporation of mercury into the human body [2]. Hence, determination and monitoring of mercury in natural water are of great importance. Recent reports estimate the total mercury content in natural water: concentrations of 0.2–55 pg mL^{-1} can be found in non-contaminated rivers and lakes [3–7], values of 5–90 pg mL^{-1} can be found in rainwater [8,9], whereas contaminated water often have concentrations up to ng mL^{-1} level [10,11].

Since mercury concentrations in non-polluted water are very low, powerful techniques are required and few of them show enough sensitivity. The most commonly used ones are atomic fluorescence spectrometry (AFS) [3,4,12–14], atomic absorption spectrometry (AAS) [15–23], inductively coupled plasma atomic emission spectrometry (ICP-AES) [24–26], and inductively coupled plasma mass spectrometry

(ICP-MS) [27,28]. Preconcentration techniques, such as gold amalgamation [15,22], Liquid–liquid extraction (LLE) [29], solid phase extraction [30] and solid phase microextraction [31], are frequently used. By preconcentration and separation of the analytes prior to measurement, some of these analytical techniques showed very high sensitivity and could quantitate Hg in the sub- pg mL^{-1} range [4,5,12,16]. However, these conventional instruments have some limitations, such as being bulky, requiring significant power consumption, and having high maintenance and operational costs. Therefore, they can only be used for laboratory-based applications.

Recently, microplasmas have attracted attention as potential sources for atomic spectrometry. These microplasmas operated at atmospheric pressure, equipped with microspectrometers, have the advantages of small size, light weight, low gas and power consumption, and relatively low manufacturing cost [32–34]. In the literature, the use of different microplasma sources comprising high-frequency microplasmas such as microwave microstrip plasma (MSP) [35,36], capacitively coupled microplasma (μCCP) [7,37] and low-frequency microplasma dielectric barrier discharge (DBD) [38–41] for the purpose of determination of Hg^{2+} in solution or gaseous Hg^0 has been reported. In monitoring Hg^{2+} in the liquid phase, the cold vapor generation technique (CV) is often used to liberate Hg^0 from solution for sample introduction because of the limited capabilities of the microplasma excitation sources to evaporate water vapor originating from the gas–liquid phase reaction/

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separation process. Preconcentration is always used in real sample analysis, and the most common preconcentration method for Hg^0 is trapping on gold. The combination of DBD with mercury preconcentration on gold amalgam followed by thermal vaporization facilitated the detection of atmospheric mercury [40] or Hg^0 in stack gas [41]. Nevertheless, the use of direct current (dc) microplasma or pulsed dc (Pdc) microplasma as an excitation source in monitoring of mercury has never been reported.

Our group has long been working on microplasma technologies and devices for molecular emission spectrometry [42–44] and atomic emission spectrometry analysis [45]. In the present work, a novel analytical system for the determination of mercury in natural water by atmospheric-pressure pulsed dc microplasma coupled with the CV technique is reported. The experiment setup is low cost and the detection system is very small in size. By trapping of mercury on a gold filament preconcentrator followed by rapid thermal desorption, the detection sensitivity is greatly improved, which ensures accurate mercury detection in natural waters at the ultra-trace level. Several experimental parameters were optimized, and analytical figures of merit were determined. In addition, five real water samples were investigated by the proposed method in order to proof feasibility of the optimized procedure.

2. Experimental

2.1. Instrumentation

2.1.1. Fabrication of the microplasma mercury detector

A schematic diagram of the microplasma cell is shown in Fig. 1(b). The cube-shaped discharge device ($1.9 \text{ mm} \times 9 \text{ mm} \times 20 \text{ mm}$) was fabricated using ceramic chips which were bonded by epoxy. The plasma channel (0.6 mm height, 0.75 mm width), was in the middle of the

device and was equipped with a port for gas input on one side. Two platinum sheet electrodes ($\sim 0.75 \text{ mm} \times 1 \text{ mm}$) were placed face to face on the upper and lower inner surface of the plasma channel, 3 mm away from the plasma outlet. The distance between the electrodes was 0.6 mm , which defined the plasma thickness. The total discharge volume was $\sim 450 \text{ nL}$. A pulsed dc voltage was applied to the electrodes for atmospheric pressure plasma generation. The length of the plasma afterglow ejecting out of the discharge chamber is about 1 to 3 mm , depending on the discharge current and argon flow rate.

2.1.2. The pulsed dc power supply

In order to reduce both the weight/volume of the device and the power consumption in the plasma discharge, a small home-built pulsed direct current (Pdc) power supply was used to generate high voltage to ignite the microplasma. The dc pulsed power supply was fabricated by means of chopping the output of the dc power supply with Intelligent Power Module (IPM) and elevating the voltage by a transformer, as shown in Fig. 1(c). A low voltage from a 12 V dc power supply or alkaline batteries was applied to the transformer, and a high-voltage output of up to 1500 V was obtained during charging and discharging cycles. The current from the power supply could be up to 9 mA and could be changed by changing the voltage from the power supply. The pulsing characteristics could be varied in frequency (300 – 1500 Hz) and duty cycle ($9:1$ – $1:1$). The waveform of current and voltage input could be measured with a digital oscilloscope (Rigol DS2202, maximum sampling rate 2 GSa/s) if necessary. In order to reduce the interferences of discharge pulses on the detection system and other instruments, the high-voltage pulse power supply is placed in a $14 \text{ cm} \times 10 \text{ cm} \times 5 \text{ cm}$ shielding box. The total weight of the pulsed dc power supply is 1.1 kg . A stable pulsed dc microplasma source could be generated with this power supply.

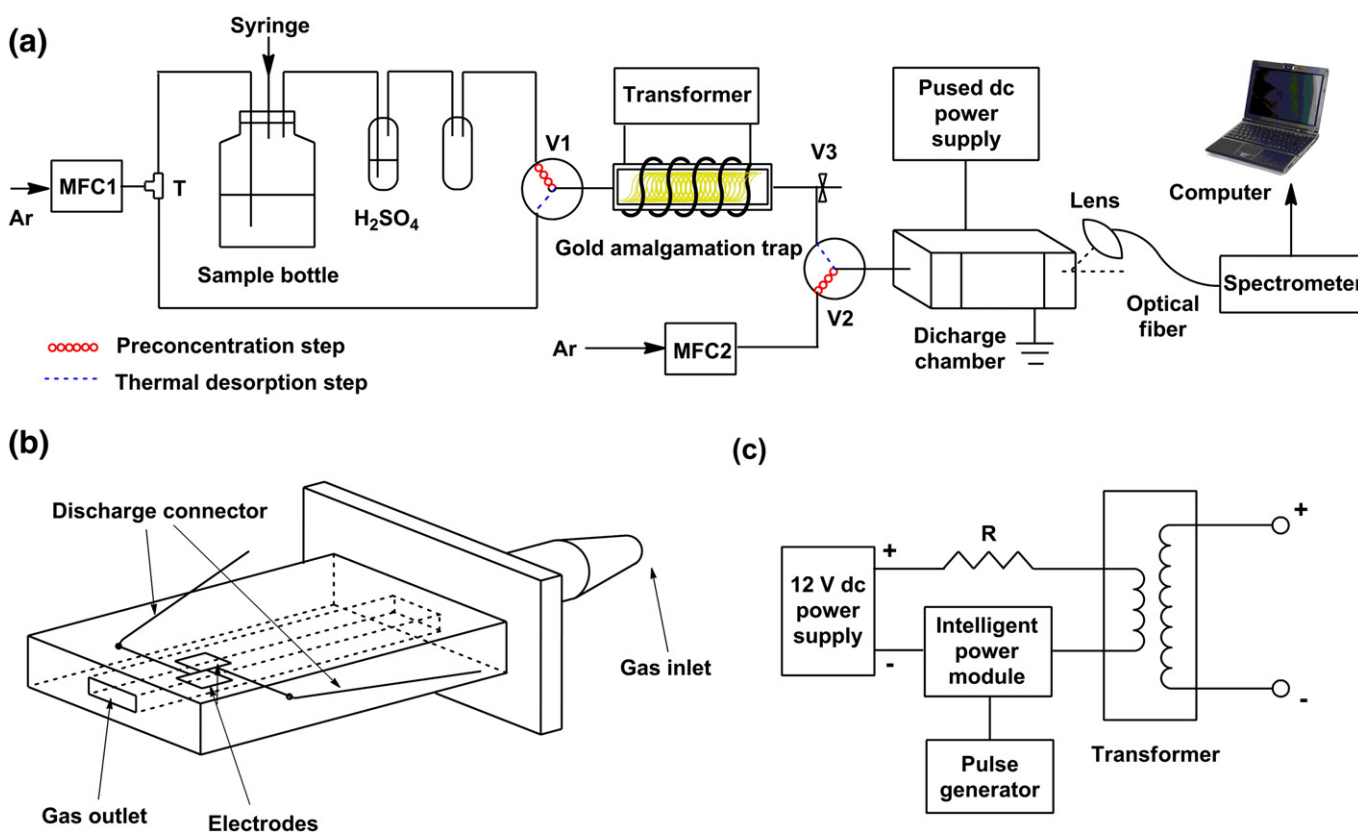


Fig. 1. Schematic diagram of (a) instrumental setup. MFC: mass flow controllers, V1 and V2: three-way valves; V3: valve; T: tee. (b) plasma cell. (c) pulsed dc power supply.

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