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Rapid determination of indium in water samples using a portable solution cathode glow discharge-atomic emission spectrometer

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ABSTRACT

A novel method for fast determination of indium in water was established using a homemade solution cathode glow discharger coupled with a portable fiber optical spectrometer. The instrumental parameters and operation conditions which may remarkably influence the analytical performance including the analytical emission line, the solution acidity, etc. were optimized. The resolution of the instrument based on the peak width at half height at 451 nm for 2.0 mg/L indium solution was about 1.1 nm. Under the optimized parameters and conditions, the limit of detection (LOD) was 0.032 mg/L calculated by $3SD_{BLANK}/k$ (n = 11). Moreover, the analytical stability was inspected by 7 parallel tests of 0.5 mg/L indium standard solution, and the relative standard deviation (RSD) was below 5%. The interferences of potential co-existing metal ions on indium determination were inspected and no significant interferences were observed. This method was applied to the analysis of real water samples available in Beijing and the spiked recoveries were in the range of 94.0%–103.6%, which proved the feasibility of indium determination of real samples. Besides, this method can satisfy the demands of field tests due to the simple and small-sized instrument and less consumption of reagent.

1. Introduction

Indium, which is known as a heavy-metal element, is now widely used in different fields, including alloy, semi-conductor, biomedicine, etc. [1–5]. Meanwhile, the risk of indium pollution tends to be enhanced accordingly as the release of indium into the environment is remarkably increasing due to the continuously growing indium consumption [6]. Moreover, indium compounds are proved to be with certain toxicity and damages to health will be caused if the income amount exceeds a certain degree, especially for tooth decay, pain in joints and the germinal and inherit system [7]. Thus, it makes significant sense to monitor the indium content level of environmental water and industrial effluents to prevent from indium pollution.

For the time being, the methods for indium determination have already been reported in literature, including atomic spectrometry [8–10], inorganic mass spectrometry [11] and electrochemical technique [12–14]. Generally speaking, flame atomic absorption spectrometry (F-AAS) is widely used in indium determination. However, the limit of determination is somewhat unsatisfactory, and the use of fuel gas is unavoidable. As a high sensitivity technique, inductively coupled plasma-mass spectrometry (ICP-MS) is suitable for ultra-trace indium determination, yet the instrumental and analytical cost is high and the apparatus is cumbersome. Furthermore, all the atomic spectrometry and ICP-MS techniques above can't work without gas cylinders so that instrumental portability tends to be difficult. As for electrochemical methods for indium detection, field test can be realized while the daily treatment or replacement of the electrodes is tedious and the memory effect is remarkable.

As a novel technique for metal elemental analysis, solution cathode glow discharge atomic emission spectrometry (SCGD-AES) has been successfully applied in metal elemental quantitative analysis, such as Pb, Cu, Li, Na, etc. and showed favorable analytic performance [15–22]. As for the solution cathode discharge system, the solution is the cathode while a metal electrode is employed as the anode. The plasma will come into being due to the gas ionization when a high voltage is applied between the two electrodes. The solution is volatilized and the elements dissolved in the solution will enter the plasma, followed by the elemental atomization and exciting process. Thus, the characteristic emission spectrum is ultimately formed.

In our previous work, the method for thallium determination by SCGD-AES was established and favorably verified in real samples [23]. However, the study on indium analysis has never been involved in the above literatures. In this paper, a novel method for indium determination using a portable SCGD equipment coupled with a fiber optical spectrometer is established. This method is feasible for field test as the dependence on gas cylinders for traditional atomic spectrometry

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Fig. 1. Diagram of SCGD cell with discharge on.

techniques is cast off and the instrument is with excellent portability due to the ideally small size and weight. Moreover, the home-made SCGD works under the normal pressure and this method is friendly to environment as only dilute acid is demanded in the test process, by which pollution brought from large amount of reagents can be effectively reduced.

2. Experimental

2.1. Reagents

The nitric acid was of guarantee grade (G.R., from Beijing Fine Chemicals Ltd.), Indium standard stock solution (1000 $mg \cdot L^{-1}$) was acquired from national standard materials research center. The calibration solutions (0.2 mg/L–5.0 mg/L) were prepared by stepwise dilution of the standard stock solution with 1% (V/V) HNO_3. Ultra pure water was produced by a Millipore ultra pure water system and the electrical resistivity is above 18.2 $M\Omega \cdot cm$. Besides, all the glassy wares were immersed in the 10% HNO_3 for 24 h, washed clean with ultra pure water and finally dried for standby application.

2.2. Sample preparation

The clearness of the water samples should be pre-judged before the follow-up treatment. If the samples are clear enough, direct determination is feasible after only an acidification operation. To say concretely, 1 mL HNO_3 is added into each 100 mL water sample to ensure the acidity consistent with the standard solutions. As for the muddy samples, the acidification is implemented only after filtration with 0.45 μm filter membranes-aquo system. The blanks and spiked samples were prepared in parallel.

2.3. Apparatus

A home-made solution cathode glow discharge cell similarly as reported in our former study [23] was further employed except for certain improvement. The SCGD cell with discharge on was as showed in Fig. 1 while the detailed SCGD-AES configuration was as showed in Fig. 2.

Generally speaking, the anode is a tungsten pin (20 mm long and 1 mm diameter) and the solution plays the role of cathode. When the gap between the two electrodes is applied a high voltage (500–1000 V) with a high voltage supply module (DW-P102 - 100C5D, Beijing Yuan Bo Sheng Electronic Technology Co., Ltd.), the gas in the gap is ionized and the plasma is generated. The solution is progressively gasified and enters the plasma so that indium ions dissolved in the solution together enter the plasma and are atomized. Then the ground-state indium atoms are excited and give out the characteristic spectrum determined by a fiber-optical spectrometer (HR4000, Ocean Optics, Inc.). The whole apparatus is portable due to the small size and weight.

In this work, a 6-way valve is adopted for the sampling module as showed in Fig. 1. In the loading process, the sample solution is pumped into the storage coil from 1, and fills in the storage coil between 3 and 6 while the surplus solution flows out as waste liquid through 2.

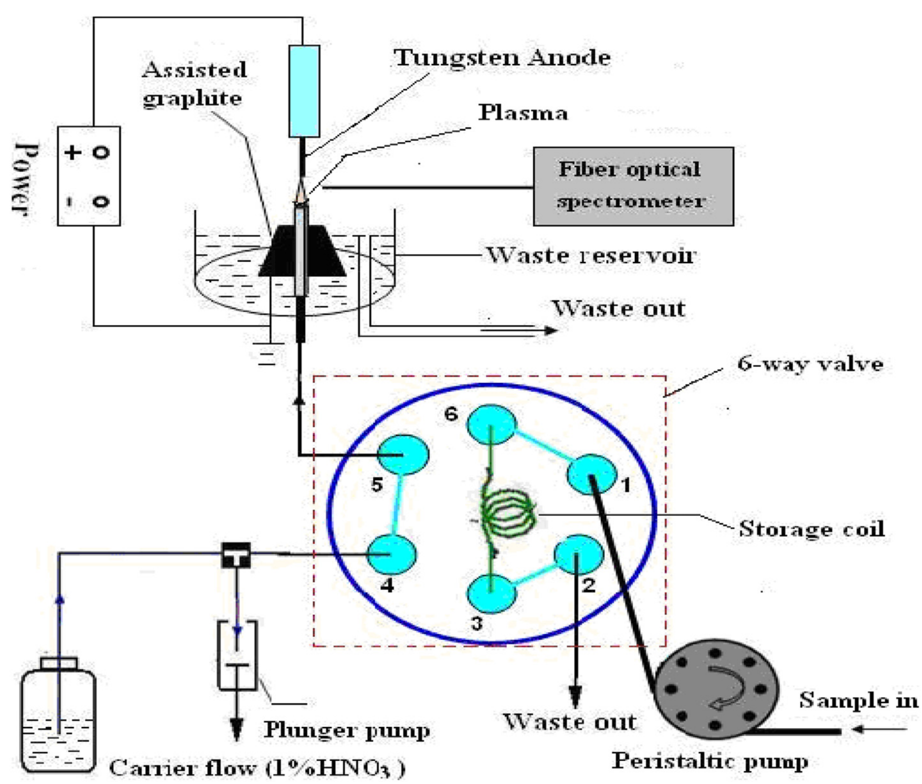

Fig. 2. The configuration of the SCGD-atomic emission spectrometry device.