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On the coupling of hydride generation with atmospheric pressure glow discharge in contact with the flowing liquid cathode for the determination of arsenic, antimony and selenium with optical emission spectrometry



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ABSTRACT

The miniaturized atmospheric pressure glow discharge (APGD) sustained between a liquid flowing cathode and a He nozzle jet anode was combined with hydride generation (HG) to improve the performance of the determination of As, Sb and Se with optical emission spectrometry (OES). As(III), Sb(III) and Se(IV) species were converted into volatile hydrides in the reaction with NaBH₄ and right after that they were delivered to the near-anode region of APGD through the nozzle. The transport efficiency of As, Sb and Se to the discharge was several times higher, while intensities of atomic emission lines of As, Sb and Se were improved 3 orders of magnitude (as compared to intensities acquired for the near-cathode region in a APGD system with a typical introduction of analytes through sputtering of the flowing liquid cathode). The effect of the concentration of NaBH₄ and HCl in a sample solution, the discharge current, the flow rate of He carrier/jet-supporting and He shielding gases on the emission yield coming from As, Sb, Se, He and H atomic lines and OH and N₂ band heads as well as the electron number density was thoroughly studied. Under compromised conditions, limits of detection (3σ criterion) of As, Sb and Se were respectively 4.2, 1.2 and 3.1 μg L⁻¹. Usefulness of the method was confirmed by the analysis of Sniadecki and Marchlewski highly mineralized spring waters (Kudowa Zdroj, Poland) on the content of As, Sb and Se. Recoveries of elements added to these spring waters were within 90.3–103.7% proving good accuracy of the HG–APGD–OES method.

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1. Introduction

New miniaturized radiation sources developed for optical emission spectrometry (OES) bring numerous advantages, e.g., simple design, high reliability and low operating costs related to energy and gas consumption. They sheared a considerable interest and are the subject of a growing number of works [1–3]. Among developed and studied in recent years miniaturized excitation sources there are dielectric barrier discharge (DBD), electrolyte as cathode glow discharge (ELCAD), liquid sampling atmospheric pressure glow discharge (LS–APGD), solution cathode glow discharge (SCGD), capacitively coupled plasma (CCP) and microstrip plasma (MSP) [4–18]. In general, these new excitation sources enable efficient excitation of elements with relatively low excitation potentials, i.e., alkali and alkaline earth elements and some of transition metals. Limits of detection (LODs) of elements usually

vary from several to tens micro gram per liter and because of that miniaturized excitation sources are commonly used for their quantification in different samples [4–16]. On the other hand, mentioned radiation sources are rarely utilized for the determination of elements with high excitation potentials, e.g., As, Sb or Se, with OES. For instance, the suitability of DBD used as an atomizer for determining As, Sb and Se with atomic absorption spectrometry (AAS) and atomic fluorescence spectrometry (AFS) has already been reported for several years [19–22]. Use of the same discharge as the excitation source in determination of As by OES has been reported just recently [23]. Also other miniaturized excitation sources, e.g., SCGD [15,16], CCP [24,25] or MSP [26,27] were used for the determination of As, Sb or Se with OES just a few times.

In our recent paper [28], a successful coupling of atmospheric pressure glow discharge (APGD) generated between a small-sized liquid flowing cathode (LFC) and a He jet with cold vapor generation (CVG) of Hg has been demonstrated. The proposed approach led to the improvement of the LOD of Hg by 4 orders of magnitude (as compared to a corresponding APGD system with

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conventional sputtering of Hg from a LFC solution). It was determined that the observed enhancement was mostly due to introduction of Hg vapor to the near-anode region of APGD. Because of a lower saturation of this region with H₂O vapor and the presence of a large population of high-energy electrons, more favorable conditions for excitation of Hg atoms were provided.

To improve the analytical characteristic of APGD operated between the He nozzle jet and the LFC for the sensitive determination of As, Sb and Se with OES, it was decided to combine it with hydride generation (HG) of As, Sb and Se. It was expected that HG would enhance the transport efficiency of analytes to the discharge and enable introduction of uncharged volatile hydrides to the near-anode region of APGD, where the concentration of water vapor would be lower. At first, APGD with a conventional introduction of elements to the discharge through sputtering of the surface of the LFC was used and analytical figures of merit of APGD–OES were evaluated. Subsequently, APGD was combined with the HG system and volatile covalent hydrides of As, Sb and Se were delivered to the discharge through the He nozzle jet anode. The effect of different working conditions on detectability of As, Sb and Se was thoroughly studied. Under compromised conditions, selected analytical figures of merit were assessed and the proposed HG–APGD–OES method was applied for the analysis of two healing mineralized waters from Sniadecki and Marchlewski springs (Kudowa Zdroj, Poland). The accuracy of the method and results of the element analysis were confirmed by spiking water samples with As, Sb and Se and determination of the amount of recovered elements.

2. Experimental

2.1. Instrumentation

A scheme of the system for miniaturized APGD generated between the He nozzle jet anode and the small-sized LFC is presented in Fig. 1. The LFC compartment consisted of a graphite tube (OD/ID 6.0/4.0 mm) with an inserted quartz tube (OD/ID 4.0/2.0 mm) in such a way that its edge was 5 mm below the edge of the graphite tube. A 0.1 mol L⁻¹ HCl solution was delivered through the quartz tube using a 2-channel LabCraft Hydris 05 peristaltic pump (France) at a flow rate of 1.8 mL min⁻¹. The overflowing solution was drained out of the cathode compartment with the same peristaltic pump. A flow of He was introduced through a stainless steel nozzle (length 35 mm, OD/ID 1.20/0.75 mm) using a Tylan General (CA, USA) FC-2900 flow controller and a RO-28 digital flow meter. The nozzle and the graphite-quartz tube were vertically oriented with a 6 mm gap between them. A Pt wires attached to the graphite tube and the nozzle were used for providing the electric contact. A stable discharge was maintained during flow of He through the nozzle and after applying a potential of

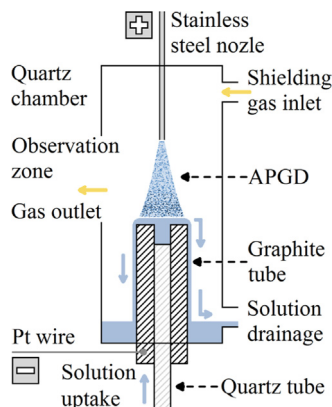


Fig. 1. Experimental set-up of APGD generated between a He jet and a small-sized flowing liquid cathode (not in scale).

1000–1500 V from a Dora (Poland) HV dc power supply. This resulted in a flow of the current of 20–50 mA, additionally stabilized by a 5 k Ω ballast resistor connected in a series. The HV dc power supply was equipped with a built-in voltmeter and an ammeter that were used to measure the conductivity of the discharge. To prevent APGD from penetration of ambient air, a semi-closed quartz chamber was used to protect the cathode compartment and the nozzle. A flow of He shielding gas, controlled by a rotameter, was additionally applied. The He flow leaked from the chamber through an elliptical hole (diameters 8 and 5 mm), the same that acted as an observation window.

A continuous flow HG system has already been described in details in our recent paper [28]. Volatile hydrides of As, Sb and Se along with H₂ were swept into the near-anode region of APGD through the stainless steel nozzle. To improve gas–liquid separation, an additional glass coil (length 70 cm, dead volume 9 mL) was inserted just before the nozzle.

The emitted radiation from near-cathode (APGD) or near-anode (HG–APGD) regions of the discharge was detected using Triax 320 spectrometer (Horiba-Jobin Yvon, France). The spectrometer was equipped with a holographic grating (1200 grooves per mm) and had the resolution of 0.06 nm. Emission spectra of the discharge were acquired with a 0.01 nm step and an integration time of 0.1 or 0.5 s. Profiles of emission lines were recorded in \sim 1.0–1.5 nm spectral windows. Further details on the optical detection arrangement are described in the paper [28].

2.2. Reagents and samples

Double-distilled water was used during research. He (99.999% grade) was supplied by Air Products (Poland). All reagents were of analytical grade or better. Stock (1000 mg L⁻¹) standard solutions of As(III), Sb(III) and Se(IV), NaOH and NaBH₄ were supplied by Sigma-Aldrich (Germany). Mixed standard solutions of As, Sb and Se were prepared by diluting stock standard solutions. To stabilize the reducing agent, solid NaBH₄ was dissolved in a 0.1% (m/v) NaOH solution. A NaBH₄ solution was freshly prepared and filtered through a cellulose filter paper. A concentrated HCl solution (37%, m/m), from Avantar Performance Materials (Poland), was used to acidify all sample solutions. In addition, a 6.0% (m/m) solution of thiourea and a 12% (m/m) solution of ascorbic acid (Avantar Performance Materials, Poland) were used to pre-reduce As and Sb from their pentavalent to trivalent oxidation states.

Samples of Sniadecki and Marchlewski spring waters (from Kudowa Zdroj, Poland) were collected into 0.5 L pre-cleaned polyethylene terephthalate (PET) bottles. After sampling, they were immediately acidified with HCl to pH 1.0. Before the analysis, samples of spring waters were subjected to appropriate pre-reduction procedures. In case of As and Sb, solutions of ascorbic acid (2.0 g), thiourea (4.0 g) and concentrated HCl (24 g) were added to 15-g samples to reach final concentrations of 0.5, 0.5 and 20% (m/m), respectively. In case of Se, 15-g samples were acidified with concentrated HCl (17 g) and heated in a water bath for 30 min to pre-reduce Se(VI) to Se(IV) ions. Then, sample solutions were diluted with water to 32 g. A final concentration of HCl in these solutions was also 20% (m/m). For a recovery study, samples of spring waters were spiked with known amounts of As, Sb and Se so that final concentrations of these elements were increased by 100, 15 and 30 μ g L⁻¹, respectively. All samples were prepared in triplicate, respective blank samples were run and considered in final results. For calibration of HG–APGD–OES, standard solutions were used within the following concentrations ranges: 50–500 μ g L⁻¹ of As in 20% (m/m) HCl with added ascorbic acid and thiourea (both at 0.5%, m/m), 10–500 μ g L⁻¹ of Sb in 20% (m/m) HCl with added ascorbic acid and thiourea (both at 0.5%, m/m) and 20–500 μ g L⁻¹ of Se in

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