Simultaneous adsorptive stripping voltammetric determination of bismuth(III) and indium(III) using cupferron as complexing agent – application to environmental waters

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Department of Analytical Chemistry, Institute of Chemical Sciences, Faculty of Chemistry, Maria Curie-Sklodowska University, 20-031 Lublin, Poland A renewable mercury film silver-based electrode (Hg(Ag)FE) in combination with adsorptive stripping voltammetry is evinced as a simple and fast approach for a simultaneous quantification of indium(III) and bismuth(III) in natural water samples. In order to effectively preconcentrate and obtain signals from both analytes in one measurement, cupferron was used as a complexing agent. Optimal conditions were found to be as follows: 0.1 mol L⁻¹ acetate buffer (pH = 4.6), 3×10^{-4} mol L⁻¹ cupferron, accumulation potential of -0.1 V and accumulation time of 60 s. A linear response of In(III) and Bi(III) in a concentration range of 2×10^{-9} to 1×10^{-7} mol L⁻¹ (r = 0.9986) was gained with a detection limit of 6.5×10^{-10} mol L⁻¹ for In(III) and 8.3×10^{-10} mol L⁻¹ for Bi(III), respectively. Analytical results of the analysis of river water samples demonstrated that the elaborated voltammetric method is apposite for direct environmental water analysis.

Keywords: indium, bismuth, renewable mercury film silver-based electrode, environmental water sample, adsorptive stripping voltammetry

INTRODUCTION

Nowadays, the method of stripping voltammetry is widely used in the trace analysis of food, surface and ground water, biological materials as well as pharmaceutical preparations [1–3]. It is one of the most sensitive electrochemical methods in which the measurement is carried out in two successive stages. In the first one, called the preconcentration step, the component to be determined is collected on the electrode at a constant potential. In the second stage, called stripping, the accumulated depolarizer undergoes the electrode reaction as a result of the electrode potential change and the voltammetric curve is recorded. In the obtained voltammogram, the signal has the form of a peak the height of which is proportional to

the depolarizer concentration. As a result, the concentration of the analyte accumulated on the electrode is much higher than its concentration in the solution. Additionally, an even greater lowering of the detection limit of method is possible due to the miniaturisation of the working electrodes used for the measurement [4, 5]. Apart from a high sensitivity, the voltammetric method undoubtedly offers a lot of other benefits, such as short time of analysis, a low cost of equipment and the ease of its use as well as a low consumption of reagents used in analysis. Most importantly, no pretreatment of the real samples is required prior to the stripping voltammetry measurement, further simplifying the voltammetric procedure and allowing the direct analysis of test samples, even at the sampling site [1, 6, 7]. Interestingly, by cause of the fact that individual depolarizers undergo electrode reaction at characteristic and sufficiently different potential

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values, the voltammetric methods allow the simultaneous determination of several analytes present in a sample [1, 4–10]. Taking the advantage of the possibilities of stripping voltammetry, it was decided to develop a procedure for the simultaneous determination of two elements in one measurement. While reviewing the literature, it was noticed that in the past few years, a lot of voltammetric procedures for the determination of bismuth simultaneously with various other metals have been reported in the literature [11-13]. Until this point, bismuth has been determined simultaneously with metals such as cadmium [11], lead [12] and uranium [13], whose peaks in the voltamperograms appeared at potentials equal to -0.6, -0.5 and -0.4 V, respectively, while bismuth undergoes an electrode reaction at a potential of about -0.2 V. With each of the above elements, bismuth was determined by adsorptive stripping voltammetry (AdSV) in the form of complexes with cupferron [11-13]. Regarding the above, in order to be able to determine two elements simultaneously, it is necessary that both voltammetric signals appear at respectively different potentials, which ensure the proper resolution of both peaks. The criterion for selecting an element that could be determined in the tested sample by the AdSV method in the presence of bismuth was that this element, like bismuth, formed electroactive complexes with cupferron, giving at the same time a signal on the voltamperogram at a potential other than -0.2 V, so that the signals were properly separated. It turned out that both of these criteria are met by indium, the AdSV determination of which was most often carried out in the form of complexes with cupferron, and the In(III) signal appears at a potential of about -0.7 V [14-18]. It should also be noted that the choice of these elements is not accidental. Both bismuth and indium are valuable raw materials used on a large scale in various industries and therefore the level of their concentration in the environment should be controlled.

Bismuth is being exploited as a component of easily fusible multi-component alloys (e.g. Wooda and Rosego alloys). The use of this metal has been reported mainly in the cosmetology, medicine and dentistry. Currently, medicaments based on bismuth hold a strong position in the treatment of gastrointestinal diseases. Bismuth is a component of Newton's alloy in dentistry, it is also one of the ingredients of skin creams in cosmetology.

Externally applied bismuth compounds denature proteins, acting as astringent, drying and antibacterial agents [19, 20]. Moreover, bismuth nanoparticles, due to their biocompatibility, are an alternative contrast agent in diagnostics [21]. The toxicity of bismuth results mainly from exceeding the doses of bismuth salts used for therapeutic purposes. Bismuth in excessive amounts tends to be deposited in internal organs, mainly the kidneys, where it binds to a metalloprotein specific for bismuth. Significant amounts of this element have also been detected in the lungs, spleen, liver, brain, and skeletal muscles. Occupational exposure is rare and is related to the cosmetics industry, the production of drugs and chemicals [19, 20].

On the other hand, indium due to its superconducting properties at the beginning of the 21st century entered the electronics and high-tech industries. Some of indium alloys began to be used in the manufacture of electroluminescent lamps, semiconductors, transistors, infrared detectors and solar panels. Until now, indium-tin oxide is the most widely used as a thin layer covering the surfaces of liquid crystal displays (LCD) used on a large scale, especially in the production of televisions, computers, smartphones and many other electronic devices. Currently, along with the production of photovoltaic cells, it is the main and most significant use of indium in industry [22]. However, there are reports that indium tin oxide contributes to skin irritation and inflammation. Inhaling it may cause respiratory problems, while its consumption causes gastrointestinal burns. The current state of knowledge indicates that the employees of plants specialising in the processing of indium oxide are most susceptible to the harmful effects of indium. Chronic exposure to indium compounds contributes to its accumulation in the liver or spleen, leading to changes in these organs and the development of chronic diseases. Studies in rats and rabbits have shown that, following the intravenous administration of indium oxide, the animals studied simultaneously developed necrotic pneumonia, fibrosis, pulmonary hyperplasia, and hepatic and heart failure [23, 24].

According to the literature data, all methods of voltammetric determination of bismuth published so far relate to the use of the hanging mercury drop electrode (HMDE) [11–13]. On the other hand, the AdSV procedures for the determination of indium in the form of complexes with cupferron

are based on metal film electrodes, such as bismuth film electrode (BiFE) [14], lead film electrode (PbFE) [15] and renewable mercury film silver-based electrode (Hg(Ag)FE) [16], whereas the lowest limit of detection of indium equal to 1.5×10^{-10} was achieved using the Hg(Ag)FE electrode. This sensor was designed and constructed by Boguslaw Bas [25] and turned out to be an innovative approach in the field of film electrodes. Owning to its special design, this electrode allows for the simultaneous use of all the advantages of mercury electrodes and at the same time minimising the contact with toxic mercury. The excellent analytical properties of the above electrode are evidenced by its numerous uses in many research works described in the literature [26–30]. Therefore, in this paper, it was decided to describe a newly developed procedure for the simultaneous determination of bismuth and indium using the Hg(Ag)FE electrode.

EXPERIMENTAL

Instrumentation

All voltammetric measurements were performed with an Autolab PGSTAT 10 analyzer (Utrecht, The Netherlands) connected to a personal computer handled by the GPES 4.9 software created by Eco Chemie, the Netherlands. The electrode stand was made up of a mercury film silver-based electrode (Hg(Ag)FE) as a working electrode, an Ag/AgCl (saturated KCl solution) reference electrode, and a Pt auxiliary electrode. All measurements were conducted using a 10 mL quartz cell. An Orion Star A211 pH benchtop meter (Thermo Scientific, Waltham, MA, USA) was utilised to measure the pH values of the solutions.

The Hg(Ag)FE electrode is designed in such a way that the mercury layer can be refreshed before each measurement without the need for electrochemical cleaning. The refreshing of the mercury layer is achieved by inserting a silver wire, on which the mercury layer is formed, into the electrode. Through this stage, the silver wires crossed with especial O-rings and there was a precise wiping. Then, the silver wire was ejected from the electrode body through the mercury chamber, which resulted in the formation of a mercury layer [31]. Therefore, when using the Hg(Ag)FE electrode, electrochemical cleaning is not necessary, unlike

in the case of film electrodes formed electrochemically on a suitable electrode substrate.

Chemicals

Each of the chemical reagents utilised was of analytical reagent grade or Suprapur. For all purposes, the deionised water obtained from Milli-Q system purification (Millipore, UK) was used. Acetate buffer was prepared from Suprapur CH₃COOH and NaOH acquired from Merck. To prepare 1 mol L⁻¹ the acetate buffer of pH of 4.60, 57.1 mL of 17.5 mol L-1 CH₃COOH was added to approximately 800 mL of deionized water. Next, 10 mol L⁻¹ NaOH was added to obtain a post-mixing pH value of 4.60. In the last stage, the resulting solution was filled with deionised water to a volume of 1 L. Cupferron (benzenamine, N-nitrosophenylhydroxylamine ammonium salt) used as a complexing agent was received from Merck (Darmstadt, Germany). A solution of 1×10^{-2} mol L⁻¹ of a complexing agent was prepared daily by dissolving 0.0155 g of this chemical in 10 mL deionized water. The stock solutions of Bi(III) and In(III) nitrates at a concentration of 1 g L⁻¹ were purchased from Fluka (Buchs, Switzerland). The working solutions of the above metal ions at concentrations of 1×10^{-4} and 1×10^{-5} mol L⁻¹ were prepared by dilution of the stock solution in 5% HNO₃ as required.

Standard DP-AdSV procedure of measurement

The standard measurement was carried out using a differential pulse adsorptive stripping voltammetry (DP-AdSV) procedure. A given volume of the water sample (natural or synthetic with the known concentration of Bi(III) and In(III)) was moved to the voltammetric cell together with 1 mL 1 mol L⁻¹ of acetate buffer (pH 4.6) and 300 μL 1×10^{-2} mol L⁻¹ cupferron. Subsequently, the prepared solution was made up to 10 mL with distilled water to give the concentration of a supporting electrolyte and a complexing agent equal to 0.1 and 3×10^{-4} mol L⁻¹, respectively. The quantitative determination of In(III) and Bi(III) concentrations in the solution prepared as above was performed in the following two steps. In the first step, the Hg(Ag) FE electrode was polarised with a constant potential value of -0.1 V vs Ag/AgCl for 60 s. Over this time, In(III) and Bi(III), both in the form of complexes with cupferron, were simultaneously accumulated on the working electrode while stirring the solution

at a speed of 600 rpm. It is a non-faradaic process, it is the result of adsorption of the In(III)-cupferron and Bi(III)-cupferron complexes onto the electrode surface. Next, the stirring was stopped and the solution was left to settle for 5 s. After that, a differential pulse stripping voltammogram was recorded in the quiescent solution, by applying a negative-going potential scan from -0.00 to -0.80 V, with a potential scan rate of 5 mV s⁻¹ and a pulse height of 70 mV. The intensities of the received peaks on the voltammogram were directly proportional to the concentration of Bi(III) and In(III) in the sample. The measurements were carried out from solutions not deaerated

Sample preparation

Natural water samples collected from the Bystrzyca River and Lake Zemborzyce were filtered using a 0.45 µm Milipore membrane filter. Both collected samples were stored in polypropylene bottles in a refrigerator at a temperature of approximately 4°C.

RESULTS AND DISCUSSION

Dependence on supporting electrolyte composition

As has been shown in scores of studies devoted to the voltammetric determination of various elements, cupferron is capable of forming electrochemically active complexes with numerous metal ions in an acidic medium [11-18, 32-35]. When reviewing only the procedures for the determination of bismuth and indium, we noticed that regardless of whether a given procedure was dedicated to the determination of Bi(III) or In(III), the optimal environment for the determination of each of the above-mentioned metal ions was provided by an acetate buffer [11-18]. Thus, at the beginning of our research, our attention was paid to pre-accomplish the simultaneous determination of Bi(III) and In(III) in 0.1 mol L⁻¹ acetate buffer at pH varied from 3.0 to 5.3. In the case of indium, the signal remained unchanged in the range from 4.6 to 5.3, whereas for lower values it gradually decreased. The pH value had no significant effect on the bismuth peak height, which remains practically constant in the tested pH range, with a minimal decreasing tendency above pH = 4.6. Taking into account the above details as well as good buffering capacity, a pH value of 4.6 was chosen as the optimum one.

Dependence on cupferron concentration

Since this voltammetric procedure is based on the accumulation of both analytes on the working electrode as complexes with a chelating agent, we expected that changing the cupferron concentration could have a big effect on the sensitivity of the bismuth and indium determination. Therefore, the behaviour of the both signals as a function of cupferron concentration was investigated. The concentration of cupferron varied from 1×10^{-5} to 6×10^{-4} mol L⁻¹ in the presence of 1×10^{-8} mol L⁻¹ of each analysed metal ion. This dependence is presented in Fig. 1 and the following conclusions are drawn from it. If the cupferron concentration varies within a range of 1×10^{-5} to 1×10^{-4} mol L⁻¹, the intensity of the indium peak current tends to increase, whereas in the case of cupferron concentrations of 1×10^{-4} to 6×10^{-4} mol L⁻¹, the signal is noticed to show downward trends. At the same time, the higher the concentration of the complexing agent in the measuring cell, the higher the signal obtained from bismuth. Based on this, the cupferron concentration of 3×10^{-4} mol L⁻¹ was adopted as the most advantageous for subsequent research.

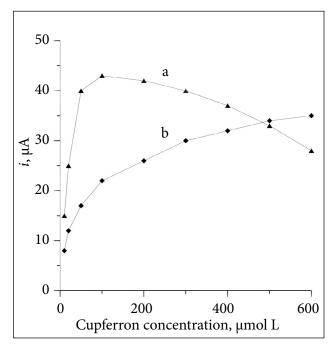


Fig. 1. The curves of ln(III) signal intensity (a) and Bi(III) signal intensity (b) depending on the complexing agent concentration. The fixed composition of solution: 1×10^{-8} mol L⁻¹ ln(III) and Bi(III), 0.1 mol L⁻¹ acetate buffer (pH = 4.6). Accumulation potential/ time -0.1 V/60 s

Accumulation potential and time conditions

The impact of the accumulation potential on the peak currents over a scope of -0.2 to 0.2 V for 1×10^{-8} mol L⁻¹ In(III) and Bi(III) indicated that the both voltammetric signals were almost unchanged over the entire range. The difference is that for a more positive potential, a slight tendency to decrease is noticeable for indium and at the same time an inconsiderable tendency to increase is noticeable for bismuth. In accordance with the above, the accumulation potential equal to -0.1 V was chosen for subsequent experiments, as the most appropriate for both metal ions (Fig. 2).

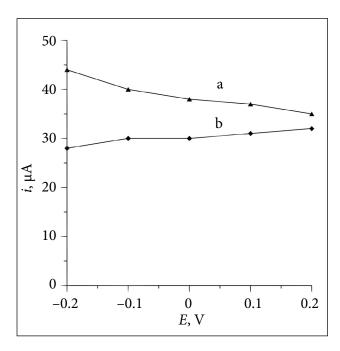


Fig. 2. The curves of ln(III) signal intensity (a) and Bi(III) signal intensity (b) depending on the accumulation potential. The fixed composition of solution: 1×10^{-8} mol L⁻¹ ln(III) and Bi(III), 0.1 mol L⁻¹ acetate buffer (pH = 4.6) and 3×10^{-4} mol L⁻¹ cupferron. Accumulation time: 60

The influence of accumulation time was studied by polarising the electrode to a potential value of – 0.1 V over the time from 0 to 150 s. The experimental data exhibits that the intensity of the Bi(III) signal successively increases with increasing the accumulation period. On the other hand, the In(III) peak current increases with the increase of the accumulation time up to 60 s, reaching a maximum value at that time, and then a gradual decrease is observed (Fig. 3). This second dependence confirmed our belief that the optimal time in which

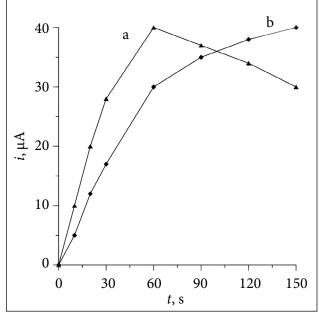


Fig. 3. The curves of ln(III) signal intensity (a) and Bi(III) signal intensity (b) depending on the accumulation time. The fixed composition of solution: 1×10^{-8} mol L⁻¹ ln(III) and Bi(III), 0.1 mol L⁻¹ acetate buffer (pH = 4.6) and 3×10^{-4} mol L⁻¹ cupferron. Accumulation potential: -0.1 V

the complexes of both metal ions should be accumulated on the working electrode in this procedure should be 60 s.

Calibration data

Analytical performance for the simultaneous determination of indium and bismuth was assessed under the optimised conditions: 0.1 mol L-1 acetic buffer (pH = 4.6), 3×10^{-4} mol L⁻¹ cupferron, preconcentration potential -0.1 V, and preconcentration time 60 s. Linear calibration curves were gained in concentration ranges of 2×10^{-9} to 1×10^{-7} mol L⁻¹ for the simultaneous presence of In(III) and Bi(III) in the solution. The dependence of the peak currents of In(III) and Bi(III) on their concentration is described by the following equations: y = 3.7x + 3.5(R = 0.9986) for In(III) and y = 2.6x + 3.3 (R = 0.9986)for Bi(III), where y and x are the peak current (μ A) and concentration (nmol L⁻¹), respectively. The calibration curves are presented in Fig. 4. The detection limits estimated from three times the standard deviation at low In(III) and Bi(III) concentrations and the accumulation time 60 s were about 6.5×10^{-10} and 8.3×10^{-10} mol L⁻¹, respectively. The relative standard deviations (RSD) from five determinations at the concentrations 3×10^{-9} mol L⁻¹ of In(III) and Bi(III) were 3.9% and 5.0%, respectively.

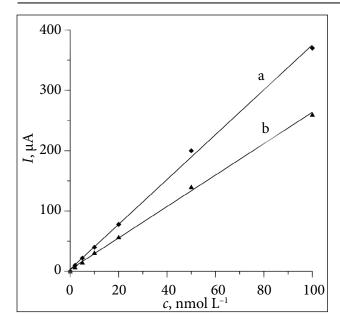


Fig. 4. The calibration curve for ln(III) (a) and Bi(III) (b). Composition of the solution: $0.1 \text{ mol } L^{-1}$ acetate buffer (pH = 4.6), $3 \times 10^{-4} \text{ mol } L^{-1}$ cupferron. Accumulation potential/ time: -0.1 V/60 s

Study of the mutual influence of both analytes

In the case of the simultaneous determination of two elements, it is very important to check that the increase in concentration of one of the elements does not affect the signal magnitude of the other element. To prove that no such interference occurs in the simultaneous determination of Bi(III) and In(III), the following experiments were carried out. A fixed concentration of Bi(III) of 1×10^{-8} mol L^{-1} was chosen and its signal recorded in the presence of higher concentrations of In(III) such as 1×10^{-8} , 2×10^{-8} and 3×10^{-8} mol L^{-1} was measured, and

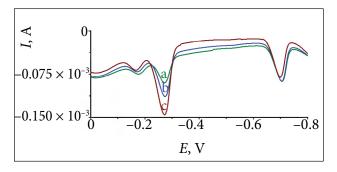


Fig. 5. The AdSV voltammograms recorded for a constant concentration of ln(III) equal to 1×10^{-8} mol L⁻¹ in the presence of increasing Bi(III) concentrations, such as 1×10^{-8} mol L⁻¹ (a), 2×10^{-8} mol L⁻¹ (b) and 3×10^{-8} mol L⁻¹ (c). The fixed composition of solution: 1×10^{-8} mol L⁻¹ ln(III), 0.1 mol L⁻¹ acetate buffer (pH = 4.6) and 3×10^{-4} mol L⁻¹ cupferron. Accumulation potential/ time: -0.1 V/ 60 s

then solutions of a fixed concentration of In(III) and different concentrations of Bi(III) were prepared identically. In both cases, it was found that both the Bi(III) and In(III) signals did not change in the presence of higher concentrations of the other accompanying element. Figure 5 shows an example of voltammograms recorded for a constant indium concentration in the presence of increasing Bi(III) concentrations.

Real sample analysis

In order to show the applicability and reliability of the recommended procedure for real water samples, water samples collated from the Bystrzyca River and Lake Zemborzyce were analysed. The voltammograms recorded for them did not exhibit any signals of In(III) and Bi(III), so the analysed samples were spiked with these metal ions at different concentration levels. Three replicate determinations using the standard addition method gave the average recovery values between 95.9 and 101.2% for 5×10^{-9} and 1×10^{-8} mol L⁻¹ In(III) with the relative standard deviation between 5.0 and 6.2%, and 97.3 and 103.5% for 5×10^{-9} and 1×10^{-8} mol L⁻¹ Bi(III) with the relative standard deviation between 4.8 and 6.3%, respectively. The examples of voltammograms recorded during the analysis of a water sample from the Bystrzyca River are presented in Fig. 6.

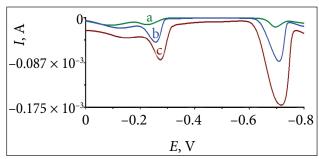


Fig. 6. The AdSV voltammograms recorded during the analysis of a water sample from the Bystrzyca River: (a) voltammogram for water from the Bystrzyca River spiked with 5×10^{-9} mol L⁻¹ Bi(III) and 5×10^{-9} mol L⁻¹ In(III); (b) as (a) $+1\times 10^{-8}$ mol L⁻¹ Bi(III) $+1.5\times 10^{-8}$ mol L⁻¹ In(III); (c) as (a) $+1.5\times 10^{-8}$ mol L⁻¹ Bi(III) $+3\times 10^{-8}$ mol L⁻¹ In(III)

CONCLUSIONS

The first elaborated adsorptive voltammetric procedure for a simultaneous determination of In(III)

and Bi(III) was evinced in this paper. The renewable mercury film silver-based electrode (Hg(Ag)FE) and cupferron as a chelating agent were found to be the right approach to the simultaneous determination of these metal ions. When using the Hg(Ag) FE electrode, there is no need to deoxidise the solution, electrochemical cleaning is not necessary and at the same time, the use of this electrode is more friendly to the laboratory environment than in the case of using the HMDE electrode. To prove the practical applicability of the proposed procedure it was successfully applied to the quantification of indium and bismuth in environmental water samples. The above-described procedure looks promising and can be recommended for monitoring the water quality of environmental waters.

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VIENALAIKIS ADSORBCINIS BISMUTO(III) IR INDŽIO(III) VOLTAMPEROMETRINIS NUSTATYMAS NAUDOJANT VARFERONĄ KAIP KOMPLEKSUS SUDARANTĮ REAGENTĄ – TAIKYMAS APLINKOS VANDENYSE