



# Electrochemical determination of bismuth with use of a Bi(III)-cupferron complexation system and elimination of interferences connected with the presence of organic substances in natural samples

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## ABSTRACT

A sensitive and selective method for the determination of bismuth based on adsorptive stripping voltammetry using cupferron as a complexing agent is presented. The effect of various parameters such as concentration of cupferron, the pH of the supporting electrolyte, the accumulation potential and time on the sensitivity was studied. The optimal analytical conditions were found to be cupferron concentration of  $1 \times 10^{-4} \text{ mol L}^{-1}$ , an acetate buffer of pH = 3, accumulation potential of 50 mV. The calibration graph is linear from  $2 \times 10^{-9}$  to  $2 \times 10^{-7} \text{ mol L}^{-1}$  for accumulation time of 30 s. The influence of interfering substances such as surfactants and humic substances present in the matrices of natural samples on the Bi(III) signal was examined. Additionally, in this work a procedure for removing these disturbing organic substances due to their adsorption on Amberlite XAD-7 resin was successfully evaluated. The procedure was applied to determine bismuth in natural water samples.

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## 1. Introduction

Bismuth compounds have been used as reagent in organic synthesis, in semiconductors and also in cosmetic preparation such as creams and hair dyes [1,2]. Some of its colloidal salts, due to their antiseptic, astringent and diuretic properties, have important applications in pharmaceutical preparations as anti-ulcer, antibacterial and radiotherapeutic agents [1]. Bismuth is also used in alloys and metallurgical additives and in recycling of uranium nuclear fuels [3,4]. However, a number of toxic effects on humans have been attributed to bismuth compounds, such as nephropathy, osteoarthropathy, hepatitis, and neuropathology [5]. These cases underscore the necessity for methods to determine bismuth for pollution control.

The determination of a trace concentration of bismuth in environmental samples requires powerful techniques and only a few techniques have sufficient sensitivity. Among the methods which have been used for bismuth determination are inductively-coupled plasma atomic emission spectrometry (ICP-AES) [6,7], inductively coupled plasma mass spectrometry (ICP-MS) [8,9], flame atomic absorption spectrometry (FAAS) [10,11], hydride generation atomic absorption spectrometry (HGAAS) [12,13] and spectrophotometry [14]. The advantages of these methods are excellent sensitivity, good selectivity and a wide linearity range, but on the other hand, they require expensive instruments, so they may be prohibitive to

many laboratories. Moreover, due to the presence of bismuth at low levels in environmental samples, its separation from other elements present and also the use of a preconcentration is usually necessary.

Electrochemical stripping procedures such as potentiometric stripping analysis [15], anodic stripping voltammetry [16–18] and adsorptive anodic stripping voltammetry [19] have been described for the determination of bismuth. The benefits of these methods are simple and cheap apparatus featuring fast response and high sensitivity.

The main aim of this work was to set a simple and sensitive electrochemical method for the determination of trace amounts of bismuth in natural water samples. The proposed procedure is based on adsorptive cathodic stripping voltammetry (AdCSV) at a hanging mercury drop electrode (HMDE). This technique is carried out in two steps. The first step is based on adsorptive accumulation of a complex of metal ion with added specific ligand onto a hanging mercury drop electrode. The second step is an electrochemical reduction of the adsorbed metal complex and a voltammetric scan to determine the amount of the metal ions studied. This technique is a favorite among different electrochemical methods because of easy speed of operation, excellent sensitivity and precision. Recently, a few adsorptive stripping voltammetry (AdSV) procedures have been reported for the determination of bismuth at a mercury drop electrode [20–27]. In these methods many ligands such as PDA [20], Alizarine S [21], TPN [22], nuclear fast red [23], chromazolum-S [24], morfin [25], substituted catechols [26] and EDTA [27] have been used for determination of bismuth. What is

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more, although a number of procedures for Bi(III) determination have been proposed, only in some procedures the interferences of organic matter were taken into account [28]. The most common interfering compounds are surfactants and humic substances. Surface-active substances are produced worldwide in large amounts because of their various applications in domestic and industrial detergents, cleaning and cosmetic products [29,30]. They are known as interferents and are easily adsorbed at the HMDE surface, which cause fouling or passivation of the electrode and disturb the voltammetric signal of the determined metal [31,32]. No procedures are proposed for bismuth voltammetric determination, where the effect of interferences has been precisely examined and then eliminated.

In this study, a novel voltammetric method is reported for the determination of bismuth by using cupferron as a complexing agent. The effects of interferents such as surfactants and humic acids were examined and, as was assumed, these compounds caused significant suppression in the AdSV response. In some voltammetric procedures the adsorptive properties of Amberlite XAD-7 resin [33] were successfully applied for elimination of interferences from organic matter [34,35]. Thus, to develop the elimination of interferences of surfactants and HA in bismuth determination, this polymeric resin was effectively exploited.

The proposed method was successfully applied for Bi(III) determination at trace levels in natural water samples. The results provide sufficient evidence for a high feasibility of the proposed method for determining Bi(III) in natural water samples.

## 2. Experimental

### 2.1. Instrumentation

The voltammograms were recorded using an  $\mu$ Autolab analyzer (Utrecht, The Netherlands). The electrochemical cell consisted of hanging mercury drop electrode – HMDE (MTM-ANKO Cracow, Poland) as a working electrode, Ag/AgCl (saturated NaCl) as a reference electrode and a platinum wire as an auxiliary electrode.

### 2.2. Reagents and solutions

All solutions were prepared from triply distilled water. A working standard solution of  $1 \times 10^{-5} \text{ mol L}^{-1}$  Bi(III) was prepared from a dilution of  $1 \text{ g L}^{-1}$  Bi(III) standard solution (Merck) as required. A  $1 \times 10^{-4} \text{ mol L}^{-1}$  cupferron stock solution was prepared weekly by dissolving 0.0155 g of the reagent in water in a 10 ml voltammetric flask. A solution of  $1 \text{ mol L}^{-1}$  of the acetate buffer (pH = 3) was prepared from Suprapur  $\text{CH}_3\text{COOH}$  and NaOH obtained from Merck. Triton X-100, sodium dodecyl sulfate (SDS), cetyltrimethylammonium bromide (CTAB) were purchased from Fluka (Buchs, Switzerland). Humic acid sodium salt (HA) was obtained from Aldrich. Amberlite XAD-7 was obtained from Sigma, washed four times with triply distilled water and dried up at temperature of  $50^\circ\text{C}$ . Metal ion solutions were prepared by dissolving appropriate amounts of their salts in triply distilled water.

### 2.3. The DP-AdCSV procedure of bismuth determination

Standard experiments were performed using differential pulse adsorptive cathodic stripping voltammetry (DP-AdCSV) in the following manner. The supporting electrolyte (10 mL), containing 1 mL of  $1 \text{ mol L}^{-1}$  acetate buffer pH = 3, 100  $\mu\text{L}$  of  $1 \times 10^{-2} \text{ mol L}^{-1}$  cupferron, different concentrations of Bi(III) and an adequate volume of triply distilled water was transferred into the voltammetric cell and purged with pure nitrogen for 5 min. The potential of 50 mV was applied for 30 s to a fresh mercury drop while the

solution was stirred. Following the accumulation period, the stir was stopped and after 5 s (equilibrium time) the differential pulse voltammogram was recorded by applying the potential scan from 0 to  $-0.4 \text{ V}$  with a  $20 \text{ mV/s}$  scan rate and a  $-50 \text{ mV}$  amplitude.

## 3. Results and discussion

### 3.1. Optimization of analytical parameters

Various parameters affecting the height and shape of the adsorptive stripping peak were examined in order to obtain the optimal conditions for bismuth (III) determination.

#### 3.1.1. Effect of cupferron concentration

The concentration of cupferron has a great impact on the bismuth peak current, so the influence of cupferron added to a sample solution containing  $1 \times 10^{-7} \text{ mol L}^{-1}$  of Bi(III) and  $0.1 \text{ mol L}^{-1}$  of acetate buffer, in the range from  $1 \times 10^{-7}$  to  $2 \times 10^{-4} \text{ mol L}^{-1}$  in the standard conditions was examined. The peak of bismuth appeared at a concentration of cupferron equal to  $2 \times 10^{-6} \text{ mol L}^{-1}$  and increased with the increasing concentration of cupferron. However, a concentration higher than  $1 \times 10^{-4} \text{ mol L}^{-1}$  did not cause significant changes in the peak height. Thus cupferron concentration of  $1 \times 10^{-4} \text{ mol L}^{-1}$  was adopted as the optimum concentration for further experiments.

#### 3.1.2. Effect of pH

The influence of the pH on the stripping process, in a solution containing  $1 \times 10^{-7} \text{ mol L}^{-1}$  of bismuth,  $1 \times 10^{-4} \text{ mol L}^{-1}$  of cupferron and  $0.1 \text{ mol L}^{-1}$  of acetate buffer was examined in the studied pH range from 3 to 6.4. In this case a variation of the pH in the whole examined range did not cause a significant change of the bismuth voltammetric signal. Taking into consideration the most convenient peak shape (the peak is the narrowest and most symmetric), the pH = 3 was selected for the next experiments.

#### 3.1.3. Effect of accumulation potential and time

The dependence of the voltammetric peak current on the accumulation potential was examined over the potential range from  $0.2 \text{ V}$  to  $-0.2 \text{ V}$ , using a solution containing  $1 \times 10^{-7} \text{ mol L}^{-1}$  of bismuth,  $1 \times 10^{-4} \text{ mol L}^{-1}$  of cupferron and  $0.1 \text{ mol L}^{-1}$  of acetate buffer. As shown in Fig. 1, the peak current slightly increased with

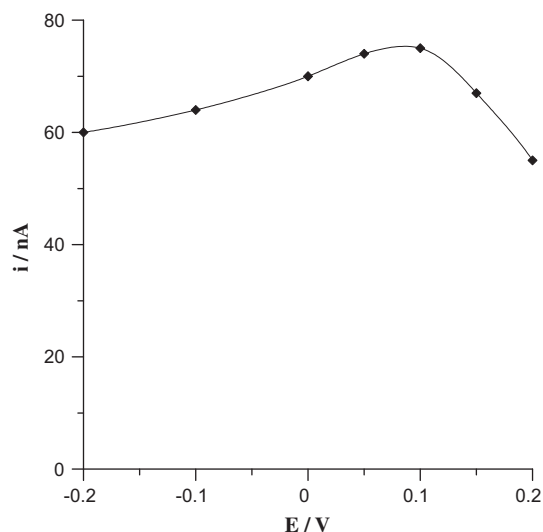


Fig. 1. The influence of the deposition potential on the peak current of  $5 \times 10^{-8} \text{ mol L}^{-1}$  Bi(III). Accumulation time 30 s.

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