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ELECTROCHEMISTRY

Adsorptive Stripping Voltammetric Determination of Antimony by Using Alizarin Red S

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Abstract: A sensitive and selective voltammetric method for determination of antimony(III) using Alizarin Red S (ARS) as complexing agent is described. The method is based on the monitoring the oxidation peak of antimony(III)-ARS complex at -520 mV in ammonium-ammonia buffer ($\text{pH} = 7.5$). The peak current was measured by scanning the potential from -700 mV versus Ag/AgCl to more positive potentials without accumulation in the presence of 1×10^{-6} mol L⁻¹ of ARS. The limit of detection (3 s) and limit of quantification (10 s) of the method were calculated from calibration curve as $1.45 \mu\text{g L}^{-1}$ and $4.8 \mu\text{g L}^{-1}$ respectively. The calibration plot for antimony(III) was linear in the range of $4.8\text{--}30 \mu\text{g L}^{-1}$. The interference of various ions was examined. Serious interference from Al(III), Fe(III), Cu(II), Pb(II), and Zn(II) was eliminated by addition of EDTA to the solution. The method was applied to drinking

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water samples. The recoveries were in the range 94%–105%. The results obtained from the developed method were compared with those from the differential-pulse anodic-stripping method and no statistically significant difference was found.

Keywords: Adsorptive stripping voltammetry, Alizarin Red S, antimony

INTRODUCTION

Antimony is mainly found in two oxidation states, Sb(III) and Sb(V), in environmental, biological, and geochemical samples. Although it has chemical and toxic properties similar to those of arsenic, its carcinogenic effect is not completely understood. However, antimony and its compounds are considered pollutants by the Agency for Toxicological Substances and Diseases Registry (ATSDR) (<http://www.atsdr.cdc.gov/mrls.html>) and the Environmental Protection Agency (EPA) in the United States and the Council of the European Communities (Quentel and Filella 2002). The US EPA maximum contaminant level (MCL) for total antimony in drinking waters is $6 \mu\text{g L}^{-1}$ and the European Union maximum admissible concentration is $5 \mu\text{g L}^{-1}$ (Quentel and Filella 2002). Therefore, accurate determination of antimony is important in various samples and matrices.

Analytical techniques employed most frequently for determination of antimony are hydride-generation atomic spectroscopic methods, including absorption spectrometry (De Campos et al. 2002; Krachler et al. 2001; Petit de Pena et al. 2001; Yersel et al. 2005), emission spectrometry (Lopez-Molinero, 2005), fluorescence spectrometry (El-Hadri et al. 2000; Cava-Montesinos et al. 2003; Fuentes et al. 2003; Sun et al. 2004; Li and Guo 2005), and mass spectrometry (Chen and Jiang 1996; Garbos et al. 2000). These techniques generally require time-consuming separation and preconcentration steps.

On the other hand, anodic stripping voltammetry (ASV) and adsorptive cathodic stripping voltammetry (AdCSV) are valuable techniques for the determination of metal ions because of their good selectivity and sensitivity, which are inherited from the deposition process. Low cost of instrumentation and speed of analysis, compared with other techniques, are their additional advantages. Both methods have been utilized for determination of antimony in various samples (Bond et al. 1998; Zhou, et al. 1998; Sander 1999; Woolever et al. 1999; Quentel and Filella 2002; Gonzalez et al. 2007).

In the ASV method (Woolever et al. 1999), Sb(III) has generally been accumulated at the hanging mercury drop electrode (HMDE) surface in

0.1 mol L⁻¹ HCl solution, and then the potential has been scanned toward positive direction. High HCl concentration (>4 mol L⁻¹) has been recommended for total antimony determination, and the speciation of antimony was carried out by subtraction.

Alternatively, several complexing agents such as morin (Zhou et al. 1998), chloranilic acid (Bond et al. 1998; Sander 1999), and pyrogallol (Gonzalez et al. 2007) have been utilized for determination of antimony by AdCSV. However, interferences from ions such as Pb(II) for the morin method, As(III) and Fe(III) (in concentration higher than 5×10^{-5} mol L⁻¹) for the pyrogallol method, and Cu(II) for both the ASV and the chloranilic acid method have been reported. Therefore, more selective methods are needed.

This paper describes a simple, sensitive, and selective voltammetric method for the determination of Sb(III) at neutral pHs. The method is based on monitoring the oxidation peak of Sb(III)-Alizarin Red S (ARS) complex adsorbed on the HMDE surface. The method differs from conventional adsorptive stripping techniques in that the anodic—but not the cathodic—stripping is employed. The proposed method was applied to drinking water samples for determination of antimony; satisfactory results were obtained.

Experimental

Apparatus and Reagents

Voltammetric measurements were performed using a Radiometer Pol 150 Polarographic Analyzer in combination with an MDE 150 polarographic stand. The analyzer was controlled with Trace Master 5 software. Three electrode systems, with an HMDE as working electrode, Ag/AgCl (satd. KCl) as reference electrode and platinum wire as auxiliary electrode, were used. Hexadistilled mercury (Radiometer-Copenhagen) was used throughout the study for HMDE. An Orion 920 A pH-meter with combined pH electrode was used for pH measurement.

Titrisol ampuls were purchased from Merck for antimony and the other metal ions (1000 mg L⁻¹). Then a 1×10^{-2} mol L⁻¹ stock solution of ARS was prepared from Fluka. The working solutions were prepared daily. The other chemicals used throughout the study were of analytical grade. All of the solutions were prepared with deionized water having the resistivity of 18.2 M Ω . The drinking water samples were collected from the market in Balikesir City. The samples were stored in a refrigerator below 4°C.

Procedure

General procedure includes the transfer of 50 μL of $1 \times 10^{-4} \text{ mol L}^{-1}$ of ARS, 1 mL of 1 mol L^{-1} ammonium-ammonia buffer ($\text{pH} = 7.5$), and the required amount of Sb(III) (or water sample) into a 10 mL volumetric flask, and the mixture is filled to the mark with deionized water. The solution was transferred to the quartz voltammetric cell, and nitrogen was purged for 5 min. A new mercury drop was extruded, and the stripping was initiated immediately in the anodic direction, starting from -700 mV , by using differential pulse (DP) modulation in quiescent solutions. The instrumental conditions were the following: potential scan rate, 1 mVs^{-1} ; pulse duration, 0.02 s; pulse amplitude, 50 mV. All of the measurements were performed at room temperature. The sample analysis was performed in the presence of $10^{-4} \text{ mol L}^{-1}$ of EDTA for the elimination of interference from metal ions.

RESULTS AND DISCUSSION

Voltammetric Peak Characteristics of the Antimony(III)-ARS Complex

The DP voltammograms of the free reagent displayed an oxidation peak at -570 mV in $\text{pH} 7.5$ ammonium buffer solution (Fig. 1). Upon the addition of Sb(III) standard solution, another peak at -520 mV was observed and proportionally built up with Sb(III) concentration. It is rather unusual to obtain adsorptive anodic stripping peak; however, it is the case for complexes of few metal ions with reducible ligands. Similar behavior of free ARS and its complex with boron have already been discussed in the literature (Sahin and Nakiboğlu 2006). This can be explained in that the free ligand, which is in anionic form at this pH , is more easily oxidized by applying the potential toward anodic direction. On the other hand, the Sb(III)-ARS complex is formed and most likely adsorbed at the electrode surface during the potential scan and oxidizes at more positive potentials than that of the free ligand.

It is well known that the key point for the adsorption process is the dependence of the peak current on the accumulation potential. Fig. 2 shows that peak currents of the complex reach a maximum value in a short accumulation time of 5 s. Therefore, the massive adsorption of the free ARS on the electrode surface hinders the accumulation of the complex, as is the case for the boron complex as well. The competitive adsorption of the complex and the free ligand results in poor precision. Therefore, the accumulation time was chosen to be as short as 5 s, or

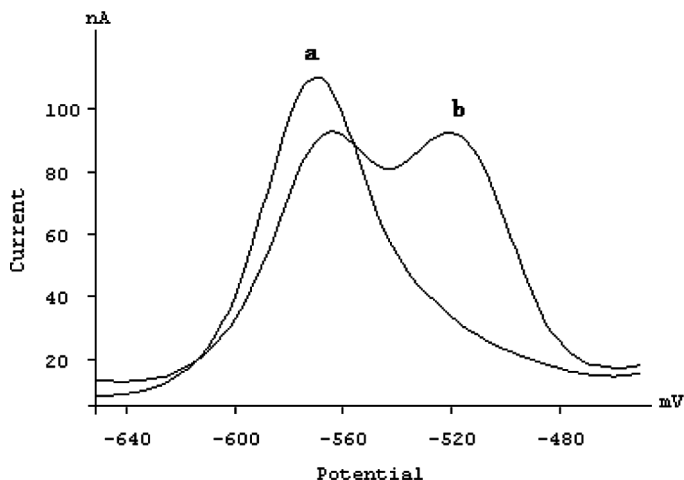


Figure 1. DP voltammograms of (a) $5 \times 10^{-7} \text{ mol L}^{-1}$ ARS and (b) addition of Sb(III) to be 15 ng mL^{-1} Sb(III) in 0.1 mol L^{-1} ammonium buffer at $\text{pH}=7.5$. Other conditions; $t_{\text{acc}}=0 \text{ s}$, $E_{\text{acc}}=-600 \text{ mV}$, pulse amplitude: 50 mV , scan rate: 1 mV s^{-1} .

the measurements were made by scanning the potential without time for deposition.

Fig. 3 shows the dependence of the peak current on the accumulation potential for 5 s accumulation times. Each scan was initiated from

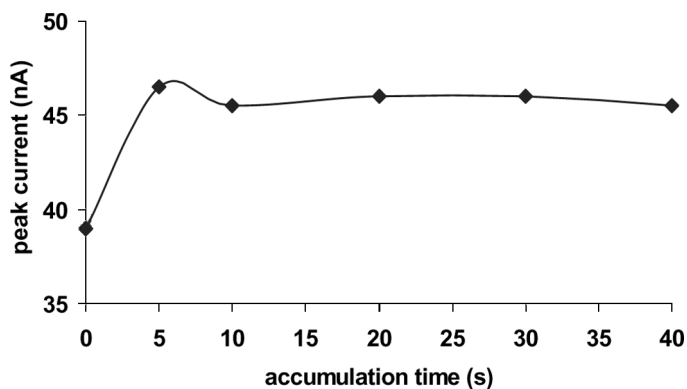


Figure 2. The effect of accumulation time on peak current. Conditions: $0.05 \mu\text{g mL}^{-1}$ Sb(III), $C_{\text{ARS}}: 1 \times 10^{-6} \text{ mol L}^{-1}$, $\text{pH}=7.5$, $E_{\text{acc}}=600 \text{ mV}$, pulse amplitude: 50 mV , scan rate: 10 mV s^{-1} .

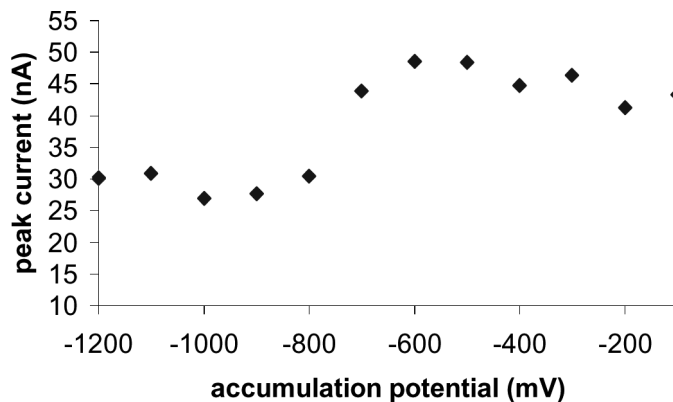


Figure 3. The effect of accumulation potential on peak current. Conditions: $50 \mu\text{g L}^{-1}$ Sb(III), $C_{\text{ARS}}: 1 \times 10^{-6} \text{ mol L}^{-1}$, $\text{pH} = 7.5$, $t_{\text{acc}}: 5 \text{ s}$, pulse amplitude: 50 mV , scan rate: 10 mV s^{-1} .

-700 mV in order to maintain the same scanning time, because it determines the extent of the accumulation. As shown in Fig. 3, the peak current gives a plateau from -700 mV and remains relatively constant toward positive potential. For this reason, -700 mV was chosen as the accumulation potential.

Effect of ARS Concentration

The concentration ratio of the ligand to Sb(III) is critical for the quantitative analysis because the free ligand competes with the complex for active sites of the electrode surface. Fig. 4 shows the voltammograms for various concentrations of ARS in the presence of $30 \mu\text{g L}^{-1}$ Sb(III) in 0.1 mol L^{-1} of ammonium-ammonia buffer at $\text{pH} 7.5$. It is clearly seen that the free ARS peak seriously interferes with the complex peak and the high ratio of free ligand to metal ion concentration badly affects the resolution and therefore the sensitivity. For this reason, the concentration of ARS was selected as $5 \times 10^{-7} \text{ mol L}^{-1}$ for subsequent measurements.

Effect of pH and Supporting Electrolyte

The effect of pH on the peak current of Sb(III)-ARS complex was studied in the range of 5.5 and 8.5, because no signal related to the complex was obtained below $\text{pH} 5.5$. The peak current gives a maximum at $\text{pH} 7.0$

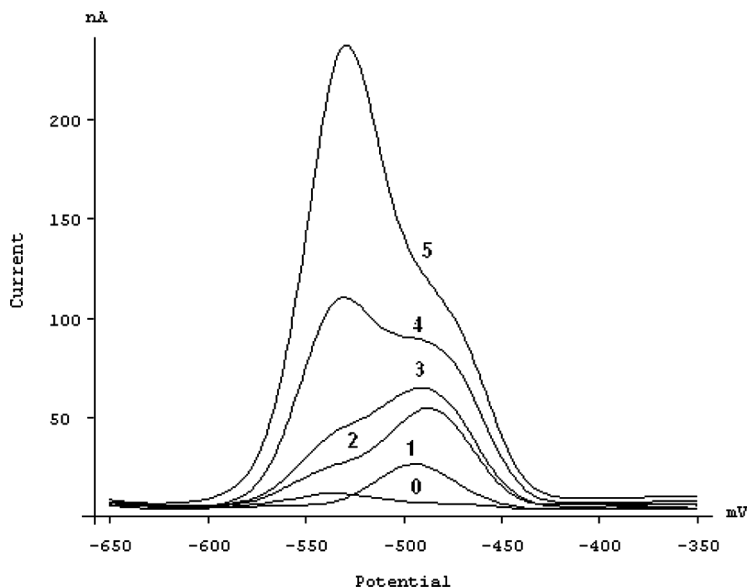


Figure 4. DP voltammograms obtained for (0) $2 \times 10^{-7} \text{ mol L}^{-1}$ ARS in 0.1 mol L^{-1} of ammonium-ammonia buffer at pH 7.5, (1) upon addition of Sb(III) standard to be $30 \mu\text{g L}^{-1}$, and then the ARS concentration was increased to (2) 4×10^{-7} , (3) 8×10^{-7} , (4) 1.2×10^{-6} , (5) $1.4 \times 10^{-6} \text{ mol L}^{-1}$. Other parameters; $E_{\text{acc}} = -600 \text{ mV}$, $t_{\text{acc}} = 5 \text{ s}$, scan rate: 5 mV s^{-1} .

(Fig. 5). A negative shift in the peak potential was observed for both the free ARS and Sb(III)-ARS complex as the pH increases. On the other hand, the peak current of free ARS decreases at higher pHs and results in better peak resolution. Additionally, various electrolytes at 6.5–7.5 pH ranges were tested as supporting electrolytes (ammonium acetate, phosphate buffer, ammonium acetate-phosphate buffer, ammonium-ammonia buffer, sodium acetate, potassium perchlorate). Among them, both the free ligand and the complex peaks overlap in phosphate buffer, resulting in poor resolution. The best resolution was observed in 0.1 mol L^{-1} ammonium-ammonia buffer (pH = 7.5) and was thus selected for subsequent experiments.

Effect of Stripping Mode and Scan Rate

Linear sweep, DP, and square wave (SW) were studied to determine the stripping mode. The lowest background and the highest peak current, in addition to better peak resolution, were obtained in the case of DP mode.

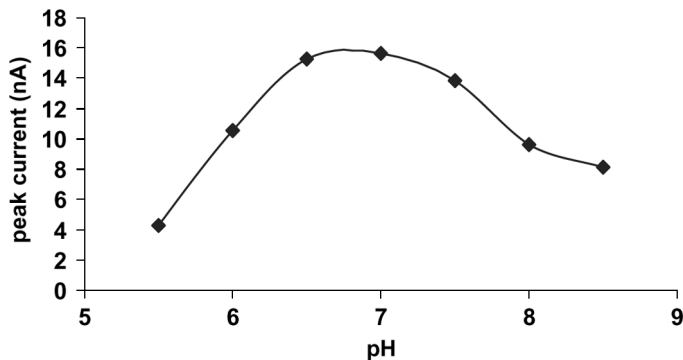


Figure 5. The effect of pH on the peak current. Conditions: $30 \mu\text{g L}^{-1}$ Sb(III), $C_{\text{ARS}} = 5 \times 10^{-7} \text{ mol L}^{-1}$, $t_{\text{acc}} = 0$, $E_{\text{acc}} = -700 \text{ mV}$, pulse amplitude: 50 mV , scan rate 10 mV s^{-1} .

On the other hand, it was observed that the potential scan rate is a critical parameter affecting the peak current for both free ARS and Sb(III)-ARS complex, because it also determines the extent of complex adsorption during the scan. Figure 6 has shown that peak currents, especially the complex peak current, decrease dramatically with the increase of scan rate from 1 to 5 Mv s^{-1} and then continue to decrease gradually

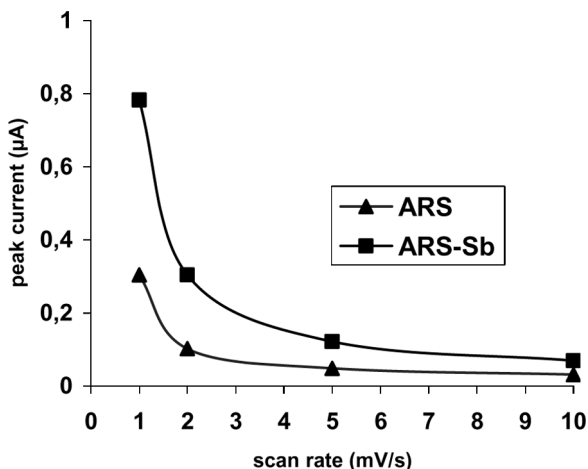


Figure 6. The effect of scan rate on the peak current. Conditions: $50 \mu\text{g L}^{-1}$ Sb(III), $C_{\text{ARS}} = 1 \times 10^{-6} \text{ mol L}^{-1}$, $t_{\text{acc}} = 0$, $E_{\text{acc}} = -700 \text{ mV}$, pulse amplitude: 50 mV , 0.1 mol L^{-1} , ammonium-ammonia buffer (pH = 7.5).

for higher scan rates. The time passed during the scan should be justified in a range long enough for the adsorption of the complex, but not too long for the massive adsorption of the free ligand.

The proper selection of the accumulation time or scan rate results in good resolution and better sensitivity for the determination method. Thus, the scan rate was selected as 1 mV s^{-1} .

Calibration Plot, Detection Limit, and Repeatability

Under the optimum conditions obtained, the voltammograms shown in Fig. 7 were recorded for construction of a calibration curve for antimony(III) determination. The curve is linear in the concentration range of $4.8\text{--}30 \mu\text{g L}^{-1}$ ($39\text{--}250 \text{ nmol L}^{-1}$). The equation of regression line was calculated as $I_p = 3.94(\pm 0.53)C_{\text{Sb}} + 1,19(\pm 3.45)$, where I_p is the peak current (in nA) and C_{Sb} is the concentration of antimony(III) (in $\mu\text{g L}^{-1}$).

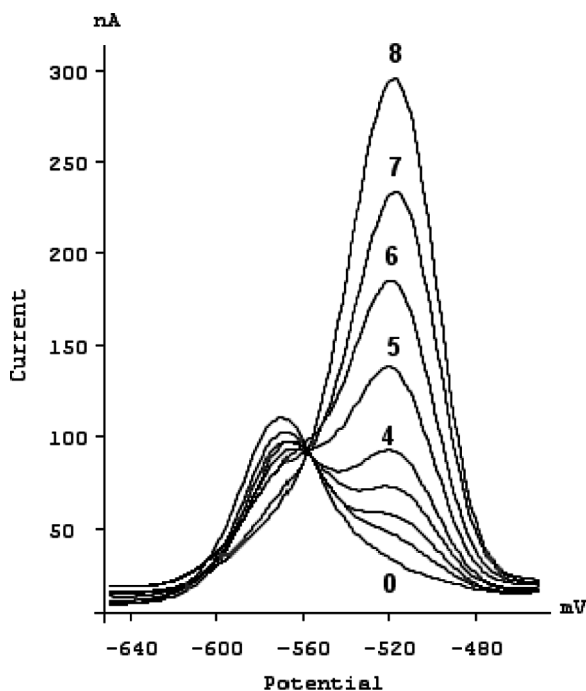


Figure 7. Voltammograms obtained for (0) $5 \times 10^{-7} \text{ mol L}^{-1}$ ARS + 0.1 mol L^{-1} ammonium buffer, and upon addition of Sb(III) standard solution to be (1) 3, (2) 6, (3) 9, (4) 15, (5) 30, (6) 40, (7) 60 and (8) $90 \mu\text{g L}^{-1}$.

The correlation coefficient was 0.997 (for $n=7$). The limit of detection (3 s) and the limit of quantification (10 s), calculated from the data for the calibration curve, were $1.45 \mu\text{g L}^{-1}$ (12 nmol L^{-1}) and $4.8 \mu\text{g L}^{-1}$ (39 nmol L^{-1}), respectively. The relative standard deviation was found as 6.7% for the $5 \mu\text{g L}^{-1}$ antimony(III) level ($n=10$).

Analytical Application

The developed method was applied to the determination of antimony(III) in tap water and bottled water samples obtained from local markets. Antimony(III) was not detected in the samples. Recovery studies of the proposed method were performed in the spiked samples. The recoveries were in the range 94%–105%. The results were also checked using ASV as a reference technique (Quentel and Filella 2002). All of the results obtained are shown in Table 1. Statistical evaluation of the results shows good agreement between the mean concentrations obtained by the two methods in a 95% confidence level.

Interferences

Under the optimum conditions, possible interferences of various ions were examined, and studies revealed that As(III), B(III), and Cd(II), did not interfere. On the other hand, Al(III), Fe(III), Cu(II), and Zn(II),

Table 1 Recoveries of antimony(III) spiked in water samples and comparison of the results obtained from the proposed method and ASV method

Water samples	Sb(III) Added ($\mu\text{g L}^{-1}$)	Sb(III) found by proposed method ($\mu\text{g L}^{-1}$)	Sb(III) found by ASV method ($\mu\text{g L}^{-1}$)	R (%)
Top water	–	nd	–	–
Top water	5.0	4.9 ± 2.3	–	97
Top water	10.0	9.4 ± 0.8	–	94
Top water	25.0	24.1 ± 2.3	–	96
Bottled water	–	nd	–	–
Bottled water	5.0	5.2 ± 0.8	3.81 ± 0.9	104
Bottled water	10.0	10.1 ± 1.0	11.2 ± 2.1	101
Bottled water	25.0	26.3 ± 3.1	28.4 ± 3.1	105

n.d.: not detected.

Table 2 Interference study for the determination of antimony(III)

Interfering ions	Tolerable ratio A ^a (M ⁿ⁺ /Sb ³⁺)	Tolerable ratio B ^b (M ⁿ⁺ /Sb ³⁺)
B ³⁺	10	10
Cu ²⁺	2	40
Cd ²⁺	10	20
Pb ²⁺	2	20
Zn ²⁺	2	20
As ³⁺	20	20
Al ³⁺ , Fe ³⁺	not tolerable	15

^aThe tolerable ratio was calculated for a $\pm 5\%$ error in the peak height. A solution containing $10\ \mu\text{g L}^{-1}$ of antimony(III) was used for throughout the interference studies.

^bThe tolerable ratio calculated in the presence of $1 \times 10^{-4}\ \text{mol L}^{-1}$ of EDTA.

which form a complex with the ARS, caused a decrease in peak current, whereas Pb(II) increased the signal because of overlapping its peak with the peak of Sb-ARS complex. The interference from all of the metal ions was eliminated by the addition of a small amount of EDTA into the cell solution. The results are summarized in Table 2.

CONCLUSIONS

The proposed method describes an alternative, simple, sensitive, selective, and accurate voltammetric method using ARS as complexing agent for the determination of Sb(III) in neutral pHs. The method is based on monitoring the oxidation peak of Sb(III)-ARS complex adsorbed on the HMDE surface. The method differs from conventional AdCSV techniques because anodic stripping is employed. This is another application of the adsorptive ASV method with the same ligand (Sahin and Nakiboğlu 2006). The proposed method also shows that the metal ions can be determined not only by AdCSV but also by adsorptive ASV.

In addition, the solution characteristics of the method are also different from the conventional methods because the ligand tends to adsorb strongly on the HMDE surface. The surface excess of the ligand completely covers the active surface area of the electrode, and competitive adsorption of the complex and the free ligand results in poor precision. Therefore, the deposition step was omitted, and, because the extent of adsorption was determined by scan rate, it was justified in a range to ensure sufficient time for the adsorption of the complex but not too long for the massive adsorption of the free ligand.

In conclusion, the proposed method offers a fast and more selective way for determination of Sb(III) in the presence of metal ions that can form complexes with ARS. The interference of Al(III), Fe(III), Cu(II), Pb(II), and Zn(II) was eliminated by addition of EDTA. The method is applicable, with satisfactory results, to drinking water samples for determination of antimony.

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