

A new and simple procedure for the polarographic speciation and determination of trace inorganic As(III) and As(V) species

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A new and simple differential pulse polarographic method was developed for the trace determination of As(III) and As(V) species in the same sample. Among several electrolytes, 0.1 M HCl was found to be the most suitable. As(III) had 3 peaks in this medium, and the peak at -0.82 V was chosen because of its peak shape and response to standard additions. In this medium, the limit of detection for As(III) was found to be 1.5×10^{-7} M. Since As(V) is not electroactive, it had to be reduced first into As(III). For this purpose, KI in an acidic and hot medium was found to be suitable. For As(V), the limit of detection was 1.0×10^{-7} M. The interference effects of the most common cations, such as Fe(III), Cu(II), Pb(II), and Zn(II), and of anions such as sulfate, chloride, and nitrate ions, were investigated. None of these ions had an overlapping peak with the peaks of As(III) in this medium. However, it was found that only Fe(III) could oxidize As(III). This creates problems in As(V) determination, since As(III), which was formed from the reduction of As(V), will be oxidized by Fe(III) and As(V) determination will not be correct. This problem was eliminated with KI, added for the reduction of As(V). As(III) and As(V) concentrations for a sample containing 1×10^{-5} M As(III) and 1×10^{-5} M As(V) could be determined with high accuracy. This method was applied to the tap water of Ankara.

Key Words: Arsenic speciation, differential pulse polarography, determination, elimination of interference, arsenic in tap water

Introduction

The danger of environmental pollution increases with the increase of toxic elements in the air, soil, and water. Arsenic is known as a very toxic element; its toxicity depends on its chemical form, its oxidation state, and its

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solubility in biological media. Arsenic exists in various states, with As(III) being the most toxic, followed by the more stable As(V) form and various organic derivatives. This element is classified as a class 1 human carcinogen by the World Health Organization (WHO) and it has been associated with skin, lung, bladder, kidney, and liver cancers.¹ The clinical manifestations of chronic arsenic poisoning are color changes in the skin, high blood pressure, cardiovascular diseases, and diabetes.

Arsenic is present in most natural materials at usually very low concentrations and, therefore, highly sensitive methods are required for its determination. Voltammetric, spectroscopic, chromatographic, and hyphenated methods are used. Among these, atomic absorption spectrophotometry (AAS),² graphite furnace atomic absorption spectrophotometry (GFAAS),^{3,4} hydride generation atomic absorption spectrometry (HGAAS),⁵⁻⁹ high-performance liquid chromatography (HPLC),^{10,11} and high-performance liquid chromatography-inductively coupled plasma-mass spectrometry (HPLC-ICP-MS)¹²⁻¹⁴ can be mentioned. For the determination of arsenic in various white wine samples, the species were first separated with ion exchange chromatography, then reduced and determined using AAS. The total arsenic was found to be 2.1-14.6 $\mu\text{g/L}$.² On the other hand, when arsine generation and gas-phase molecular absorption spectrometry were used, the limit of detection (LOD) for As(III) and As(V) was found to be 0.5 $\mu\text{g/mL}$ and 3 $\mu\text{g/mL}$, respectively.¹⁵ With the use of inductively coupled plasma-atomic emission spectrometry (ICP-AES), the LOD was found to be 0.1 $\mu\text{g/L}$ for As(III) and As(V).¹⁶

Electrochemical methods are primarily preferred for trace analysis and speciation analysis, because of their high selectivity and very low detection limits, and because they do not require tedious extraction or preconcentration procedures. As the redox character has an important role in most speciation analysis, electrochemical methods are especially used for speciation studies due to their significance in redox reactions.

As(III) determination was conducted using a catalytic hydrogen wave that was obtained with a bismuth film electrode.¹⁷ In another study, the As(III) and total arsenic in a natural water source and mineral water could be determined with cathodic stripping voltammetry, with 40 s of deposition using a hanging mercury electrode.¹⁸ The As(III) and As(V) contents in soil were determined with cathodic stripping voltammetry. The reduction of As(V) into As(III) was conducted with ascorbic acid¹⁹ and 3 different extractants were tested. As, Se, Cu, Pb, Cd, Zn, and Mn levels in marine environmental matrices were determined using anodic and cathodic stripping voltammetry.²⁰ Matrix exchange differential pulse stripping voltammetry was also used for the determination of total arsenic in gold samples.²¹ Cathodic stripping voltammetry with a hanging mercury drop electrode was used for the determination of As(III) and As(V) in sea water samples.²² Using a gold electrode with differential pulse anodic stripping voltammetry, trace As(III) levels of natural waters and of biological samples were determined. The detection limit was 0.15 $\mu\text{g/L}$ for As(III).²³ The hydrogen catalytic wave formed in the presence of Mo(VI) and As(III) could be used for the determination of from 10^{-6} to 10^{-8} M As(III) using differential pulse polarography (DPP).²⁴ The detection limit was 2×10^{-9} M. This method was used for the determination of arsenic in a beer sample.

In most of the spectrometric speciation methods mentioned above, procedures such as extraction, separation, and enrichment methods are needed and the LOD is not as low as the LOD obtained with electroanalytical methods. On the other hand, electrochemical methods are more selective and inexpensive. Each species can be determined separately, while only the total quantity can be determined with most of the spectrometric speciation methods mentioned above. The results obtained with DPP are very reproducible because, with the use of a dropping mercury electrode (DME), the behavior of the electrode is independent of its history.

As can be seen from a literature survey, for the determination of As(V) in the presence of As(III) using voltammetric analysis, As(V) must be reduced to As(III) with a reducing agent. However, the possible interference of Fe(III), which is usually present in natural materials, is not taken into account. If Fe(III) is present, it will oxidize As(III) into As(V) once more, and thus its determination will not be correct. In this work, this interference was also investigated.

The aim of this work was to establish a new and rapid method for the speciation and determination of trace As(III) and As(V), without a need for separation or enrichment procedures.

Experimental

Apparatus

A polarographic analyzer (PAR 174 A; PAR, Princeton, NJ, USA) equipped with a PAR mercury drop timer was used. The drop time of the electrode was in the range of 2-3 s (2.4 mg s^{-1}). A Kalousek electrolytic cell with a saturated calomel electrode (SCE), separated by a liquid junction, was used in the 3-electrode configuration. The counter electrode was platinum wire. The polarograms were recorded with a Linseis X-Y recorder (LY 1600; Selb, Germany) under the conditions of a drop life of 1 s, a scan rate of 2 mV s^{-1} , and a pulse amplitude of 50 mV. High-purity nitrogen (99.999%) was used to expel the oxygen present before each measurement. A Cr(II) solution was used in gas washing bottles to reduce any trace oxygen in the nitrogen gas.

Reagents

All chemicals used were of analytical-reagent grade (proanalysis). Triply distilled water was used in the preparation of all solutions. Solutions of 10^{-3} M and more dilute solutions were prepared before every use in order to avoid the aging of the solution. In order to expel the oxygen present in the polarographic cell, 99.999% pure nitrogen was passed through the solution.

The mercury used in the dropping mercury electrode was obtained from Merck (Darmstadt, Germany). Contaminated mercury was cleaned by passing it successively through dilute HNO_3 (3.0 M) and water columns in the form of fine droplets by using a platinum sieve. This mercury was then washed in the same way until no acidic reaction was observed. The collected mercury was dried between sheets of filter paper. Before use, a DPP polarogram of this mercury was recorded in order to confirm the absence of impurities. As can be seen, the mercury used was not lost and could be used hundreds of times without causing any pollution.

Preparation of As(III) and As(V) solutions

Dilute solutions of 1×10^{-3} , 1×10^{-4} , and 1×10^{-5} M were prepared before every use in order to avoid the aging of the solution.

0.1 M As(III) solution: 0.65 g of NaAsO_2 was dissolved in distilled water and then diluted to 50 mL.

0.1 M As(V) solution: 1.56 g of $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ was dissolved in distilled water and then diluted to 50 mL.

Preparation of acetate electrolyte

For the 1.0 M HAc/Ac⁻ buffer, 6.0 g of solid NaOH was washed with water and then added to 57.2 mL of 1.0 M acetic acid. The solution was then diluted to 1000 mL. The pH was adjusted to the desired value using a pH meter.

Reduction of As(V)

For the reduction of As(V) into As(III), 10 mL of 1.0×10^{-3} M As(V) and 0.5 M HCl and about 0.83 g of KI (0.5 M) was added to a volumetric flask and mixed so that the KI was dissolved completely. This sample solution was then kept warm in a water bath at about 90 °C for 30 min. The solution became pale yellow because of the iodine that formed.

Results and discussion

The aim of this work was to determine trace As(III) and As(V) concentrations in the same sample solution. Since As(V) is not electroactive, both of these ions cannot be determined from one single polarogram. Since As(III) can be determined with high accuracy, As(V) must be reduced into As(III) first and then determined.

Electrochemical behavior of As(III) in various electrolytes

The electrochemical behavior of As(III) was studied in our previous work²⁵ in an acetic acid medium, both in the presence and absence of EDTA, at pH values of 4.0, 6.0, and 8.0. It was found that the peaks of As(III) in this medium were not suitable for its determination. Since the reduction of As(V) takes place in acidic solution, the electrochemical behavior of As(III) has been studied mostly in acidic solutions such as 1 M HAc/Ac⁻ (pH = 2.0), 0.1 M HClO₄, 0.1 M H₃PO₄, and 0.1 M HCl. As given below, the most suitable electrolyte for As(III) peaks was chosen according to its shape, separability from other peaks, and response to standard additions.

In a medium of 1 M HAc/Ac⁻ (pH = 2.0), 2 peaks for As(III) were observed. The peak at -0.72 V was sharp and the peak at -0.86 was broad, as seen in Table 1. Neither peak responded well to standard As(III) additions in this medium.

Table 1. The behavior of 1.0×10^{-5} M As(III) in various electrolytes (n = 3).

Medium	Peak potentials E (V)	Peak heights (mm)	Peak shapes
1 M HAc/Ac ⁻ (pH = 2.0)	-0.72 and -0.86	38 ± 3 and 27 ± 4	sharp/broad
0.1 M HClO ₄	-0.60, -0.68, and -0.77	32 ± 2, 43 ± 2, and 32 ± 3	broad/sharp/broad
0.1 M H ₃ PO ₄	-0.62, -0.72, and -0.86	31 ± 3, 44 ± 1, and 26 ± 2	broad/sharp/broad
0.1 M HCl	-0.61, -0.68, and -0.82	24 ± 2, 37 ± 1, and 24 ± 1	broad/sharp/broad

In 0.1 M HClO₄, 0.1 M H₃PO₄, and 0.1 M HCl media, As(III) had 3 peaks. The first and the third

showed proportional increments to standard additions, but the second did not. In our further studies, we showed that this second peak was an adsorption peak.

Because of the larger peak separation between the peaks, it was decided to use the third peak for the determination of As(III). This peak was observed at -0.77 V in 0.1 M HClO_4 , at -0.86 V in 0.1 M H_3PO_4 , and at -0.82 V in 0.1 M HCl . The results obtained for peak heights and shapes for 3 separate measurements are summarized in Table 1. HCl was taken as the most suitable medium because it had the most reproducible response for standard additions.

Determination of As(III)

As can be seen from Figure 1, the polarogram of 1.0×10^{-5} M As(III) in 0.1 M HCl had 3 peaks, at -0.61 V, -0.68 V, and -0.82 V. The peak at -0.61 V corresponds to the reduction of As(III) into elemental arsenic and the peak at -0.82 V corresponds to the reduction of arsenic into arsenide (As^{3-}).²⁶ The peak at -0.68 V must be an adsorption peak, because of its shape, and because it disappeared with the addition of Triton X-100, a surface-active reagent. This peak increased first, but then became constant with the continuous addition of As(III).

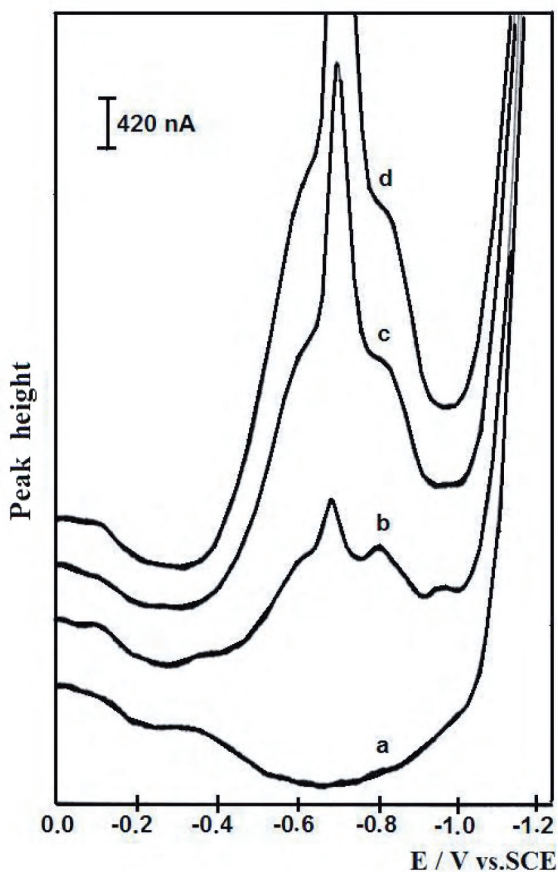
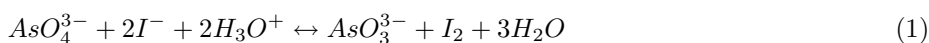


Figure 1. Determination of 1.0×10^{-5} M As(III). a) 10 mL of 0.1 M HCl electrolyte b) a + 0.1 mL of 1.0×10^{-3} M As(III) c) b + 0.1 mL of 1.0×10^{-3} M As(III) d) c + 0.1 mL of 1.0×10^{-3} M As(III).

The peak separation between the first and second peak was 70 mV, and it was 140 mV between the second and third peak; that is, a larger separation was obtained with the peak at -0.82 V (Figure 1). Therefore, the peak at -0.82 V was chosen for the determination of As(III); the quantification limit was 5×10^{-7} M and the LOD was 1.5×10^{-7} M.

Determination of As(V) after reduction into As(III) from 2 As(III) peaks

Since As(V) is not electroactive, it had to be reduced and determined as As(III). For this purpose, KI in an acidic medium was used, as given below.²⁷



As can be seen from Eq. (1), this reduction takes place in acidic medium, and since it is a reversible reaction, the acidity used for the reduction had to be protected in the polarographic cell. This is why 0.1 M HCl was used as the electrolyte for the polarographic determination of 10^{-5} M As(III).

For the reduction of As(V) into As(III), 10 mL of 1.0×10^{-3} M As(V) and 0.5 M HCl and about 0.83 g of KI (0.5 M) was added to a volumetric flask and mixed so that the KI was dissolved completely. In our former study on the digestion losses in natural materials, it was found that the KI and HCl concentrations during the reduction had to be similar.²⁷ This sample solution was then kept warm in a water bath at about 90 °C for 30 min. The solution became pale yellow because of the iodine that formed. During the investigation of the optimum conditions for the reduction, it was found that the color of this solution became dark in a few days, because of the iodine that formed after the oxidation of iodide with air oxygen. Thus, this solution was discarded and prepared again.

As shown in Figure 1, As(III) had 3 peaks in 0.1 M HCl, at -0.61 V, -0.68 V, and -0.82 V. Since KI was used for the reduction of As(V), there would be KI in the electrolyte and it may have changed the peak potentials. To see its effect, 0.2 mL of reduced 1.0×10^{-3} M As(V) (2.0×10^{-5} M in the cell) was added to 10 mL of 0.1 M HCl and a polarogram was taken; 2 peaks were observed for As(III), the first at -0.68 V and the second at -0.82 V. As can be seen from Figure 2, after reduction of As(V) into As(III) with KI, the adsorption peak disappeared, and a peak for the iodine that formed during the reaction appeared at about -1.0 V. As expected, this peak decreased with nitrogen purging through the solution.

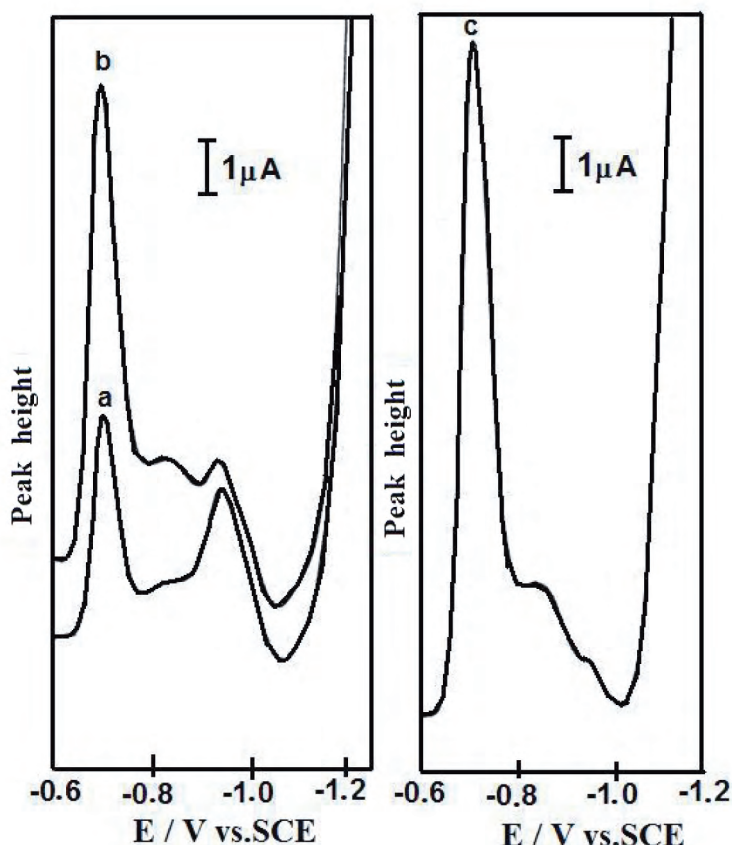


Figure 2. Determination of As(V) in a sample after reduction to As(III) using the peak at -0.68 V. a) 10 mL of 0.1 M HCl + 0.2 mL of 1.0×10^{-3} M reduced As(V) b) a + 0.2 mL of 1.0×10^{-3} M As(III) c) b + 0.2 mL of 1.0×10^{-3} M As (III) Reduction of As(V): 10 mL of (1×10^{-3} M As(V) + 0.5 M HCl) + 0.83 g of KI (0.5 M).

The determination was conducted with both of these peaks in order to see which peak was preferable. For this purpose, the determination of 2×10^{-5} M As(V) after reduction was done using the peak of As(III) at -0.68 V; for arsenic, $(1.99 \pm 0.23) \times 10^{-5}$ M was obtained (Figure 2). The same sample was then used for As(V) determination from the second peak at -0.82 V (Figure 3). Since this peak was smaller than the peak at -0.68 V, the current range had to be changed from $1.0 \mu\text{A}/\text{cm}$ to $0.4 \mu\text{A}/\text{cm}$, and 2×10^{-5} M As(V) could be determined from the -0.82 V peak as $(1.98 \pm 0.26) \times 10^{-5}$ M with high accuracy.

As can be seen, the accuracy was very good for both peaks. The limit of quantification was 3×10^{-7} M, and the LOD for As(V) was 1×10^{-7} M (signal-to-noise ratio of 3). Since the response to standard additions was better for the peak at -0.82 V than the peak at -0.68 V, the peak at -0.82 V was primarily used in our further studies.

Interference studies

The most common cations, such as Fe(III), Cu(II), Pb(II), and Zn(II), and anions such as sulfate, chloride, and nitrate, were investigated for their interference effects.

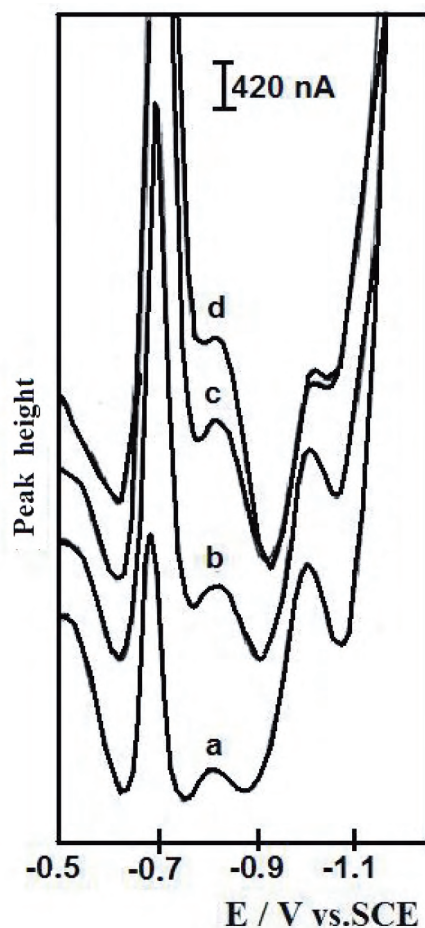


Figure 3. Determination of As(V) in a sample after reduction into As(III) using the peak at -0.82 V. a) 10 mL of 0.1 M HCl + 0.2 mL of 1.0×10^{-3} M reduced As(V) b) a + 0.1 mL of 1.0×10^{-3} M As(III) c) b + 0.1 mL of 1.0×10^{-3} M As(III) d) c + 0.1 mL of 1.0×10^{-3} M As(III) Reduction of As(V): 10 mL of (1×10^{-3} M As(V) + 0.5 M HCl) + 0.83 g of KI (0.5 M).

Cations were studied from 2 respects of their interference. The first was overlapping peaks with As(III) and the second was their reduction potential, in order to see their possibility of reacting with As(III).

Peak potentials

The peak potentials of these cations were determined by taking polarograms in 10 mL of 0.1 M HCl.

The peaks of Fe(III), Cu(II), Pb(II), and Zn(II) were -0.13 V, -0.14 V, -0.38 V, and -0.98 V, respectively. As(III), on the other hand, had 3 peaks, at -0.61 V, -0.68 V, and -0.82 V, in this medium. As can be seen, none of these peaks was expected to have peak overlap with As(III). The Zn peak was the nearest, but it had 160 mV of difference from the last peak of As(III). Sulfate, chloride, and nitrate ions are electroinactive and thus had no peak overlap.

Standard reduction potentials

The standard reduction potentials are important because of the possible redox reaction with As(III). The standard reduction potentials were +0.70 V, +0.34 V, -0.12 V, -0.76 V, and +0.58 V for Fe(III), Cu(II), Pb(II), Zn(II), and As(V), respectively. According to these data, only a reaction between Fe(III) and As(III) is possible.

If there is a sample containing 3 of these ions, As(III), As(V), and Fe(III), at the beginning, As(III) will be oxidized by Fe(III). In this case, there may be 2 possible conditions.

1) If As(III) is present in a larger concentration than Fe(III), both As(III) and As(V) will be present in this sample and the Fe(III) will be used up. For their determination, As(III) is determined first from the polarogram by standard additions, and then As(V) is reduced with KI and determined as As(III).

2) However, if Fe(III) is present in a larger concentration than As(III), in this case, no As(III) will be present, but As(V) and Fe(III) will be present. The Fe(III) in this case will create a problem. During the determination of As(V), it has to be reduced with KI and determined as As(III). If Fe(III) is present, however, it will oxidize the As(III) formed from the reduction into As(V), and thus its determination will not be correct. However, in this case, the addition of KI for the reduction of As(V) will also reduce the Fe(III) present, and the interference effect of Fe(III) will be eliminated. Thus, addition of KI in excess is needed and will not create any problems.

Determination of 1.0×10^{-5} M As(III) and 1.0×10^{-5} M As(V) in a synthetic sample

As(III) was first determined, and then As(V) was reduced and its content was calculated from the difference. A 50-mL synthetic solution containing 1.0×10^{-5} M As(III), 1.0×10^{-5} M As(V), and 0.1 M HCl was prepared.

For the determination of As(III) in a synthetic sample, 10 mL of this solution was taken and added to the polarographic cell; nitrogen gas was passed and a polarogram was taken. Aliquots of 0.1 mL of standard additions of 1.0×10^{-3} M As(III) were made and 1.0×10^{-5} M As(III) was determined as 0.98×10^{-5} M using the peak at -0.82 V (Figure 4).

For As(V) determination, KI (0.17 g) was added to 10 mL of this synthetic sample so that its concentration was about 0.1 M, and it was warmed up in a water bath to 90 °C for 30 min so that the reduction of As(V) was accomplished. After it cooled, it was added to a polarographic cell, nitrogen was passed, and a polarogram was taken. A peak at -0.82 V was observed. Standard additions were made using 0.2 mL of 1.0×10^{-3} M As(III) (the As(III) in the cell was 2.0×10^{-5} M) and a similar increase in peak height was observed (Figure 5). The total As(III) was found to be 1.88×10^{-5} M. When the As(III) found (0.98×10^{-5} M) (Figure 4) was subtracted from the total As(III) (1.88×10^{-5} M), As(V) was found at 0.9×10^{-5} M instead of 1.0×10^{-5} M. Some other examples for this kind of synthetic sample are given in Table 2.

Determination of total arsenic quantity in tap water of Ankara

Arsenic may be accumulated in the body from drinking water and from some foods, although they contain very small amounts of it. Thus, its trace determination in water, foods, and soil is very important. According to

Table 2. Determination of As(III) and As(V) quantities in synthetic samples containing 1.0×10^{-5} M As(III) and 1.0×10^{-5} M As(V).

Ions (in polarographic cell)	Determined (M)	\bar{X}	s	$\bar{X} \pm ts/\sqrt{N}$
1.0×10^{-5} M As(III)	0.98×10^{-5}	1.0×10^{-5}	5.86×10^{-7}	$(1.0 \pm 0.09) \times 10^{-5}$
	1.07×10^{-5}			
	0.96×10^{-5}			
2.0×10^{-5} M total arsenic	1.88×10^{-5}	1.98×10^{-5}	1.56×10^{-6}	$(1.98 \pm 0.26) \times 10^{-5}$
	2.16×10^{-5}			
	1.90×10^{-5}			
1.0×10^{-5} M As(V)	0.90×10^{-5}	0.98×10^{-5}	1.0×10^{-6}	$(0.98 \pm 0.16) \times 10^{-5}$
	1.09×10^{-5}			
	0.94×10^{-5}			

n = 3, 90% CI

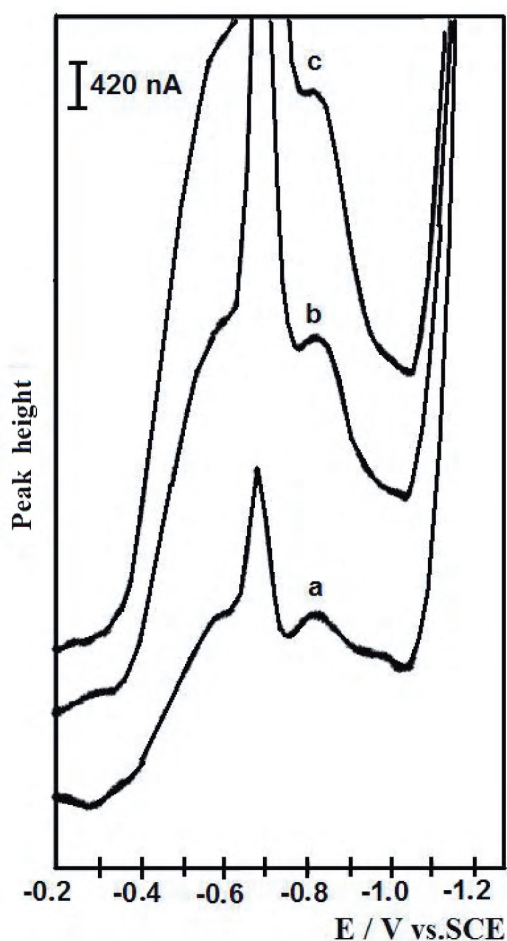


Figure 4. Determination of As(III) in a synthetic sample containing 1.0×10^{-5} M As(III) and 1.0×10^{-5} M As(V).
 a) 10 mL of synthetic sample b) a + 0.1 mL of 1.0×10^{-3} M As(III) c) b + 0.1 mL of 1.0×10^{-3} M As(III) Synthetic sample: 50 mL of (1.0×10^{-5} M As(III) + 1.0×10^{-5} M As(V) + 0.1 M HCl).

WHO, the allowed level in drinking water is $10 \mu\text{g/L}$.¹ In this study, the newly established method was applied to the tap water of the city of Ankara.

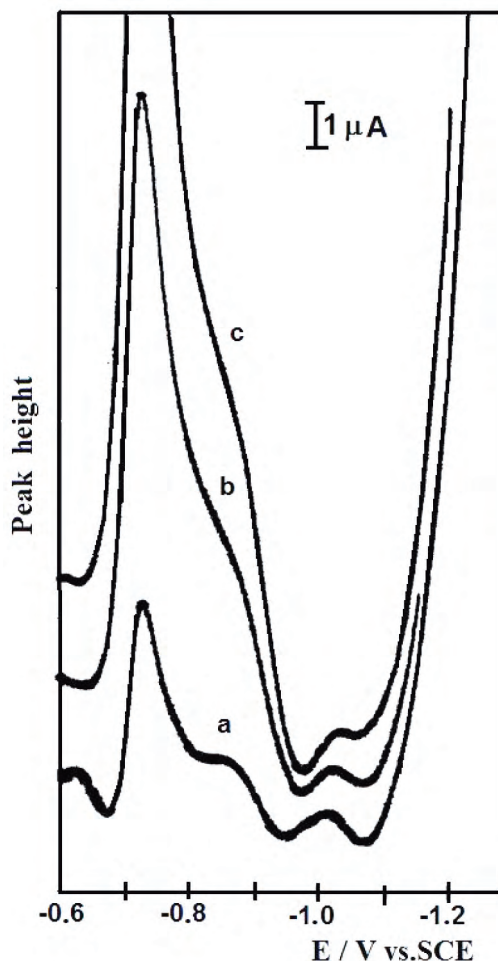


Figure 5. As(V) determination after reduction of a synthetic sample containing 1.0×10^{-5} M As(III) and 1.0×10^{-5} M As(V). a) 10 mL of reduced synthetic sample b) a + 0.2 mL of 1.0×10^{-3} M As(III) c) b + 0.2 mL of 1.0×10^{-3} M As(III) Reduced synthetic sample: 10 mL of (1.0×10^{-5} M As(III) + 1.0×10^{-5} M As(V) + 0.1 M HCl) + 0.17 g of KI (0.1 M).

There is a possibility that As(III) in water is oxidized with oxygen. In this case, As(V) has to be reduced and the total arsenic has to be determined.

For the determination of total arsenic in water, the As(V) present has to be reduced first. For this purpose, 0.17 g of KI (0.1 M) was added to 10 mL of 0.1 M HCl and warmed up in a water bath about for 30 min at about 90°C . This 10-mL sample was added to a polarographic cell and a polarogram was taken, but no peak was observed. Standard additions were then made using 1.0×10^{-5} M As(III) solution (Figure 6). As can be seen in Figure 6 (line c), a peak of 5 mm in height at -0.82 V appeared when the As(III) concentration

reached 3.0×10^{-7} M. Since our LOD was 1.5×10^{-7} M As(III) and since no peak was observed for the tap water, its concentration must be lower than this concentration, which is lower than the allowed limit.

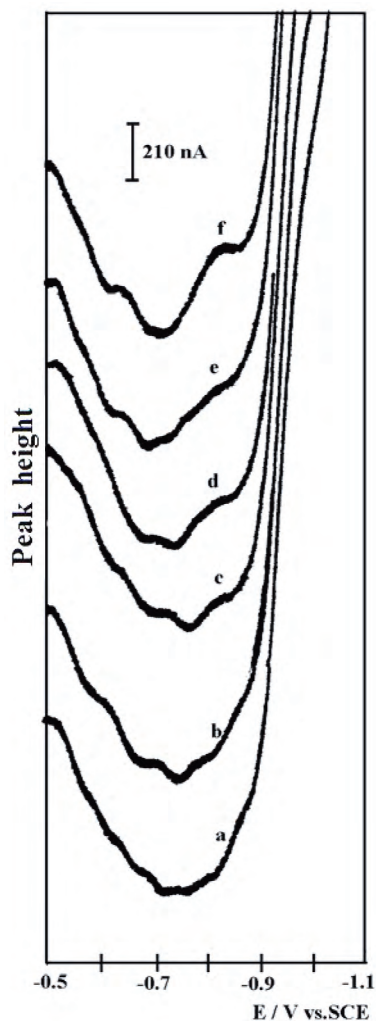


Figure 6. Determination of total As(V) in tap water of Ankara (0.1 M HCl + 0.1 M KI). a) 10 mL of Ankara tap water (0.1 M HCl + 0.1 M KI) b) a + 0.1 mL of 1.0×10^{-5} M As(III) c) b + 0.2 mL of 1.0×10^{-5} M As(III) d) c + 0.1 mL of 1.0×10^{-4} M As(III) e) d + 0.1 mL of 1.0×10^{-4} M As(III) f) e + 0.1 mL of 1.0×10^{-4} M As(III).

Conclusion

In most speciation methods, procedures such as extraction, separation, and enrichment are needed, which are time consuming and have a danger of pollution. In many of them, only total quantities are determined. On the other hand, electrochemical methods are more selective and there is usually no need of preconcentration or separation procedures. Each species can be determined separately. However, it is not possible to determine As(III) and As(V) species simultaneously from one polarogram, since As(V) is not electroactive. Thus, As(V)

had to be reduced first into As(III) using KI and determined from the As(III) peak, which is very sensitive. In this DPP method, among the many electrolytes tested, 0.1 M HCl was found to be the most suitable electrolyte for the determination of As(III). The LOD for As(III) and As(V) was found to be 1.5×10^{-7} M and 1.0×10^{-7} M, respectively.

No interference was observed for most of the common cations and anions. Only Fe(III), which is generally present in natural materials, can create problems in As(V) determination. This is because the As(III) formed from the reduction of As(V) will be oxidized by the Fe(III) present and the As(V) determination will not be correct. However, in this case, KI can reduce the Fe(III) present and the interference effect of Fe(III) will be eliminated. Thus, addition of KI in excess is needed, which will not create any problems. The newly established method was successfully applied to a synthetic sample and to the tap water of the city of Ankara.

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