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Voltammetric screen-printed carbon sensor modified with multiwalled carbon nanotubes and bismuth film for trace analysis of thallium(I)

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Abstract: The paper investigates the possibility of using commercially available screen-printed sensors with carbon nanomaterials modified working electrodes to anodic stripping voltammetric determination of trace concentrations of Tl(I). Each working electrode was additionally plated *in-situ* with a bismuth film (BiF). The highest analytical signal of Tl(I) at potential of -0.65 V (vs. pseudo-reference silver electrode) was achieved at the screen-printed carbon sensor with multiwalled carbon nanotubes and bismuth film modified working electrode (SPCE/MWCNTs/BiF). The calibration curve was linear in the range of Tl(I) concentrations from $1 \cdot 10^{-8}$ to $1 \cdot 10^{-6}$ mol·dm⁻³ (-0.9 V, 180 s). The developed procedure of Tl(I) determination at this sensor allowed to achieve the low limits of detection and quantification of Tl(I), $2.8 \cdot 10^{-9}$ and $9.3 \cdot 10^{-9}$ mol·dm⁻³ respectively. The method was used to determine thallium in the spiked water samples from the Vistula river.

Keywords: screen-printed sensor, anodic stripping voltammetry, bismuth film, multiwalled carbon nanotubes, thallium(I)

1. Introduction

Thallium is widely distributed in the environment, although it generally occurs at very low concentrations. Tl(I) is not a ubiquitous trace element (Galvan-Arzate and Santamaria, 1998), The average concentration of thallium in the continental crust is 0.49 ppm and in the oceanic crust 0.013 ppm. The main anthropogenic sources of thallium are emissions and solid waste from coal combustion as well as iron and non-ferrous smelting (Peter and Viraraghavan, 2005).

Thallium is highly toxic to the biosphere, its toxicity is even higher than that of Hg, Cd, Pb and Cu (Wallwork-Barber et al., 1985). The main mechanism of toxicity of thallium is related to disturbances of important processes related to potassium, e.g. substitution with potassium in (Na⁺/K⁺)-ATPase, this is due to the fact that Tl⁺ and K⁺ ions are monovalent cations with similar ionic radii (Nasiri-Majd et al., 2015). Human exposure to thallium is mainly associated with the consumption of contaminated food or drinking water. Thallium quickly penetrates into the bloodstream and is distributed throughout the whole body, which leads to its deposition in the bones, nervous system and kidneys (Karbowska, 2016). In the case of acute thallium poisoning, gastrointestinal symptoms usually appear e.g. severe abdominal pain, nausea, vomiting and bloody diarrhoea (Cvjetko et al., 2010). The main symptoms of acute intoxication are also polyneuropathy and alopecia. Chronic effects of thallium poisoning have been seen following occupational exposure. Chronic thallium poisoning begins with tiredness, fatigue, headache and insomnia, nausea and vomiting, muscle and joint pains, paraesthesia, numbness of fingers and toes followed by motor weakness. Hair loss may follow after a period of a few weeks (Kazantzis, 2000).

Bearing in mind the high toxicity of thallium and its effects on human health, it is extremely important to measure its level in environmental samples. For this reason, it is necessary to develop very sensitive analytical methods for the determination of this element. Currently thallium is determined

with many different methods such as spectrophotometry (Pandey et al., 2015), electrothermal atomic absorption spectrometry (ETAAS) (Husakova et al., 2008), solution cathode glow discharge atomic emission spectrometry (SCGD-AES) (Zu et al., 2017), electrothermal vaporization inductively coupled plasma mass spectrometry (ETV-ICP-MS) (Chen et al., 2017) and inductively coupled plasma optical emission spectrometry (ICP-OES) (Biata et al., 2018). However, most of the mentioned methods require a pre-concentration step, including using various types of extraction e.g. liquid-liquid microextraction (LLME) (Escudero et al., 2013) or solid phase extraction (SPE) (Chen et al., 2017). Although these methods have been widely used to determine trace amounts of Tl(I), electroanalytical methods also appear very suitable because they are reliable, sensitive and require less expensive equipment. A lot of literature data concern voltammetric determination of thallium at mercury electrodes (Lu et al., 1999; Munteanu and Munteanu, 2006; Pałdyna et al., 2013). However, in order to reduce work with toxic and simultaneously volatile mercury compounds, different types of sensors for thallium determination were developed, among which the growing popularity became bismuth based electrodes (Domańska and Tyszczyk-Rotko, 2018; Jorge et al., 2007; Kokkinos et al., 2010; Rutyna and Korolczuk, 2014; Węgiel et al., 2016).

Carbon-based nanostructures such as carbon nanofibers, carbon nanotubes, and mesoporous carbons have been extensively used in fabrication of modified electrodes, because of their low price, electrocatalytic activity for a variety of redox reactions, a broad potential window, and relatively inert electrochemistry (Afkhami et al., 2016).

In this work the possibilities of using commercially available screen-printed sensors with carbon nanomaterials modified working electrodes were checked. Each working electrode was additionally plated *in-situ* with a bismuth film (BiF) and used for the determination of trace concentration of Tl(I).

2. Materials and methods

2.1. Apparatus

In this study the electrochemical analyzer μ Autolab from Eco Chemie (Netherlands) was used. All measurements were made using the DropSens screen-printed sensors. Each sensor had a screen-printed unmodified (Ref. 110) or modified with graphene (Ref. 110GPH), carbon nanofibers (Ref. 110CNF) and multiwalled carbon nanotubes (Ref. 110CNT) working carbon electrode, a silver pseudo-reference electrode and a carbon auxiliary electrode. The measurements were carried out in a classic voltammetric cell into which 10 cm³ of solutions were poured. In the deposition step, the solutions were mixed using a magnetic stirrer.

2.2. Reagents

As a basic electrolyte, a buffer solution prepared by mixing a solution of CH₃COONH₄ (BioUltra, Sigma-Aldrich, USA) at a concentration of 1 mol·dm⁻³ and a solution of HCl (TraceSelect, Fluka) at a concentration of 0.47 mol·dm⁻³, was used. The reaction gave a buffer solution with a pH 4.6 and following composition: 0.53 mol·dm⁻³ CH₃COONH₄, 0.47 mol·dm⁻³ CH₃COOH and 0.47 mol·dm⁻³ NH₄Cl. Thallium(I) and bismuth(III) solutions were prepared from standard stock solutions at a concentration of 1 g·dm⁻³ (Merck, Germany). Solution of Na₂EDTA (disodium salt of ethylenediaminetetraacetic acid) was prepared by dissolving weighted portion of reagent (Sigma-Aldrich, USA) in distilled water. To study the interference effect, the standard solutions of Cd(II), Zn(II), Fe(III), Cu(II), Pb(II) (Merck, Germany), Triton X-100, sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB), which were purchased from Fluka, were used. All solutions were prepared using ultra-purified water (> 18 M Ω cm) supplied by the Milli-Q system (Millipore, United Kingdom).

2.3. Optimized procedure for the determination of thallium(I)

During the determination of Tl(I), a 10 cm³ solution with the composition: 0.0265 mol·dm⁻³ CH₃COONH₄, 0.0235 mol·dm⁻³ CH₃COOH and 0.0235 mol·dm⁻³ NH₄Cl buffer solution of pH 4.6, 1·10⁻⁵ mol·dm⁻³ Na₂EDTA and 7.5·10⁻⁶ mol·dm⁻³ Bi(III) was introduced into a classical electrochemical cell. Electrochemical electrode cleaning took place at a potential of 0.5 V for 10 s. Simultaneous deposition of bismuth (Bi³⁺ + 3e → Bi) and thallium (Tl⁺ + e → Tl) took place at a potential of -0.9 V for

180 s. Determination of Tl(I) was carried out with square wave anodic stripping voltammetry (SWASV). The analytical signal was recorded in the potential range from -1.1 to 0.5 V. From the recorded voltammograms, the background was subtracted and the fragments were cut. The frequency was 40 Hz and the amplitude was 75 mV.

3. Results and discussion

3.1. Selection of a screen-printed sensor with a suitable working electrode

In order to select suitable screen-printed sensor for Tl(I) determination measurements were performed in a solution containing $0.0265 \text{ mol}\cdot\text{dm}^{-3}$ $\text{CH}_3\text{COONH}_4$, $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ CH_3COOH and $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ NH_4Cl buffer solution of pH 4.6, $1\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$ Bi(III), $1\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$ Na_2EDTA and $2\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I). The Na_2EDTA added to supporting electrolyte forms permanent complexes with interfering ions (e.g. Cd(II) and Pb(II)) and in consequence they no longer influence the Tl(I) signal. The potential applied to the electrode was changed in the following order: 0.5 V for 10 s and -1.1 V for 60 s. In the first step, the surface of the electrode was electrochemically cleaned, and in the second stage the bismuth film was plated and the thallium was deposited simultaneously (Domańska and Tyszczyk-Rotko, 2018). Measurements under the described conditions were carried out using a screen-printed sensors with a carbon working electrode unmodified or modified with graphene, carbon nanofibers or multiwalled carbon nanotubes. Each electrode was additionally modified with a bismuth film. Voltammograms recorded using different types of electrodes are shown in Fig. 1.

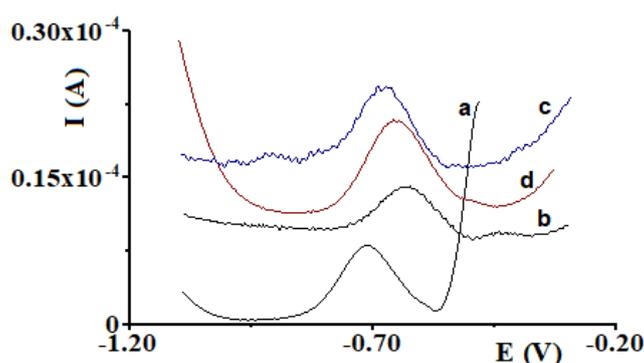


Fig. 1. SWASV curves obtained in a solution containing: $0.0265 \text{ mol}\cdot\text{dm}^{-3}$ $\text{CH}_3\text{COONH}_4$, $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ CH_3COOH and $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ NH_4Cl buffer solution of pH 4.60, $1\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$ Bi(III), $1\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$ EDTA and $2\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I) using screen-printed sensors in which the bismuth film was deposited on a carbon working electrode: (a) unmodified, (b) modified with graphene, (c) modified with carbon nanofibers (CNFs) and (d) modified with multiwalled carbon nanotubes (MWCNTs)

Based on the obtained results, it was found that the highest analytical signal was obtained on a screen-printed carbon electrode modified with multiwalled carbon nanotubes and a bismuth film (SPCE/MWCNTs/BiF). It is connected with high surface area of SPCE/MWCNTs (Eissa et al., 2019). Therefore, for further study the SPCE/MWCNTs/BiF was used.

3.2. Effect of Bi(III) concentration on Tl(I) peak current

In this stage the influence of the Bi(III) concentration on the Tl(I) peak current was studied. The measurements were carried out for Bi(III) concentrations in the range from 0 to $1.5\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$. The solution also contained $0.0265 \text{ mol}\cdot\text{dm}^{-3}$ $\text{CH}_3\text{COONH}_4$, $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ CH_3COOH and $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ NH_4Cl buffer solution of pH 4.6, and $2.5\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I). Bismuth film plating and thallium deposition were carried out for 60 s at a potential of -1.1 V. From Fig. 2, it was proved that bismuth plating of SPCE/MWCNTs enhances the thallium signal. In addition, it can be clearly seen that the highest signal was obtained for a Bi(III) concentration of $7.5\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$. A further increasing of Bi(III) concentration resulted in a decreasing of the Tl(I) peak current. Therefore, for further studies a Bi(III) concentration of $7.5\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$ was selected.

3.3. Influence of potential and time of simultaneous bismuth plating and thallium deposition on Tl(I) peak current

In this part of the experiments, it was examined how the change in potential and time of the film plating and thallium deposition affects the height of Tl(I) signal. The test was performed for Tl(I) concentration of $1 \cdot 10^{-7}$ mol·dm⁻³. The measurements were carried out in a solution containing also 0.0265 mol·dm⁻³ CH₃COONH₄, 0.0235 mol·dm⁻³ CH₃COOH and 0.0235 mol·dm⁻³ NH₄Cl buffer solution of pH 4.6, $1 \cdot 10^{-5}$ mol·dm⁻³ Na₂EDTA and $7.5 \cdot 10^{-6}$ mol·dm⁻³ Bi(III). The potential was changed in the range from -0.6 to -1.1 V. Based on the obtained results (Fig. 3A), it was found that the change in the potential of simultaneous deposition of bismuth film and thallium has a significant influence on the height of the analytical signal of Tl(I). The peak current reached the highest value at the potential of -0.9 V and further change of potential towards more negative values caused a slight decrease in the analytical signal height. Afterwards the time of simultaneous deposition of bismuth film and thallium was investigated for potential of -0.9 V in the range from 0 to 250 s. The results are presented in Fig. 3B. A time of 180 seconds was chosen, because further time elongation caused only a slight increase in the Tl(I) peak current.

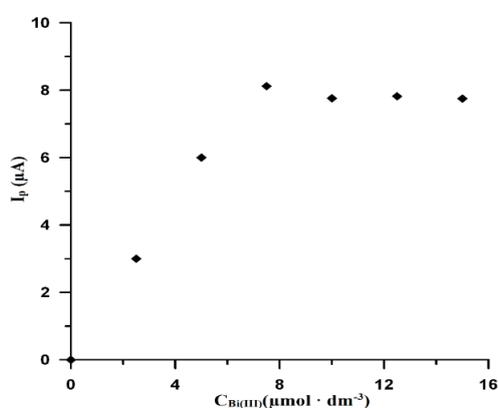


Fig. 2. Influence of Bi(III) concentration on Tl(I) ($2.5 \cdot 10^{-7}$ mol·dm⁻³) peak current. Bismuth film was plated and thallium deposited simultaneously for 60 s at potential of -1.1 V

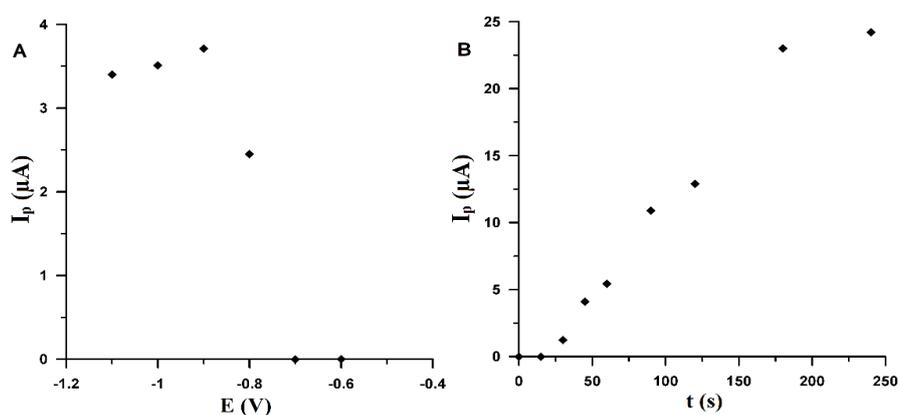


Fig. 3. Influence of a potential (A) and time (B) of simultaneous bismuth plating and thallium deposition on the Tl(I) peak current at a concentration of $1 \cdot 10^{-7}$ mol·dm⁻³

3.4. Calibration curve, repeatability and reproducibility

In order to make measurements for the calibration curve, 0.0265 mol·dm⁻³ CH₃COONH₄, 0.0235 mol·dm⁻³ CH₃COOH and 0.0235 mol·dm⁻³ NH₄Cl buffer solution of pH 4.6, $1 \cdot 10^{-5}$ mol·dm⁻³ Na₂EDTA, $7.5 \cdot 10^{-6}$ mol·dm⁻³ Bi(III) and Tl(I) in the concentration range from $1 \cdot 10^{-9}$ to $5 \cdot 10^{-6}$ mol·dm⁻³, were placed in the measuring cell. Measurements were made for a 180 s deposition time and a potential of -0.9 V. It was found that the calibration curve of Tl(I) is linear in the concentration range from $1 \cdot 10^{-8}$ to $1 \cdot 10^{-6}$ mol·dm⁻³. The linear range of the calibration curve is shown in Fig. 4 and can be described by

the equation $y = (114.7 \pm 0.5)x + (0.5 \pm 0.03)$, where y is the Tl(I) peak current in (μA), and x its concentration in ($\mu\text{mol}\cdot\text{dm}^{-3}$). The correlation coefficient (r) is 0.9993. The limit of detection (LOD) and quantification (LOQ) were calculated as 3 and 10-times of the standard deviation (SD, $n=3$) for the blank divided by the calibration curve slope ($\text{LOD} = 3\text{SD}/a$; $\text{LOQ} = 10\text{SD}/a$). The calculated LOD and LOQ of Tl(I) are $2.8\cdot 10^{-9}$ and $9.3\cdot 10^{-9}$ $\text{mol}\cdot\text{dm}^{-3}$, respectively.

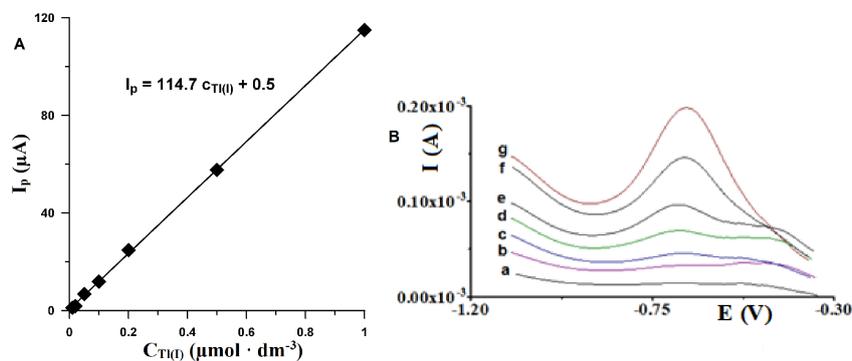


Fig. 4. Linear range of the Tl(I) calibration curve obtained at the SPCE/MWCNTs/BiF (A), SWASV curves obtained during the determination of increasing concentrations of Tl(I) (B): (a) $1\cdot 10^{-8}$; (b) $2\cdot 10^{-8}$; (c) $5\cdot 10^{-8}$; (d) $1\cdot 10^{-7}$; (e) $2\cdot 10^{-7}$; (f) $5\cdot 10^{-7}$; (g) $1\cdot 10^{-6}$ $\text{mol}\cdot\text{dm}^{-3}$

Comparison of the obtained LOD with those obtained by other methods such as voltammetry (Domańska and Tyszczyk-Rotko, 2018; Jorge et al., 2007; Kokkinos et al., 2010; Lu et al., 1999; Munteanu and Munteanu, 2006; Pałdyna et al., 2013; Rutyna and Korolczuk, 2014; Węgiel et al., 2016), spectrophotometry (Pandey et al., 2015), ETAAS (Husakova et al., 2008) and SCGD-AES (Zu et al., 2017) is presented in Table 1. When compared with other voltammetric methods that enable determination of Tl(I), the obtained LOD in some cases is higher. However, in the most cases lower LODs were obtained using voltammetry as compared to other methods. It should be added, that the proposed voltammetric procedure uses screen-printed sensors that are small size and can be used in portable devices in field analysis.

Moreover, the repeatability of the signal was determined by successive measurements ($n = 3$) of each studied Tl(I) concentration from the calibration graph at the same electrode. The relative standard deviation of the peak current in the range from 4.5% to 5.2% was obtained. Additionally, the electrode-to-electrode reproducibility was estimated with three replicate determinations of Tl(I) at concentration of $1\cdot 10^{-7}$ $\text{mol}\cdot\text{dm}^{-3}$ at three independently prepared SPCE/MWCNTs/BiF. The relative standard deviations of the peak current of 5.5% was obtained.

Table 1. Comparison of different thallium determination methods

Method (electrode)	Limit of detection ($\text{mol}\cdot\text{dm}^{-3}$)	Reference
Spectrophotometry	$1.4\cdot 10^{-8}$	(Pandey et al., 2015)
ETAAS	$4.2\cdot 10^{-9}$	(Husakova et al., 2008)
SCGD-AES	$5.8\cdot 10^{-8}$	(Zu et al., 2017)
SWASV (SPCE/BiF)	$8.5\cdot 10^{-10}$	(Domańska and Tyszczyk-Rotko, 2018)
SWASV (Rotating-disc BiFE)	$1.1\cdot 10^{-8}$	(Jorge et al., 2007)
SWASV (Microfabricated BiFEs)	$2.9\cdot 10^{-9}$	(Kokkinos et al., 2010)
SWASV (Nafion/mercury film electrode)	$1.0\cdot 10^{-11}$	(Lu et al., 1999)
ASV (Thin-film mercury-carbon electrode)	$1.0\cdot 10^{-10}$	(Munteanu and Munteanu, 2006)
DPASV (HMDE)	$7.3\cdot 10^{-10}$	(Pałdyna et al., 2013)
SWASV (BiFE)	2.1×10^{-11}	(Rutyna and Korolczuk, 2014)
DPASV (BiABE)	5.0×10^{-12}	(Węgiel et al., 2016)
SWASV (SPCE/MWCNTs/BiF)	2.8×10^{-9}	This work

DPASV - differential pulse anodic stripping voltammetry; HMDE - hanging mercury drop electrode; BiABE - bismuth bulk annular band electrode

3.5. Interferences

The influence of foreign metal species present in natural water samples on thallium peak was checked based on the addition of following metal ions: Cd(II), Zn(II), Fe(III), Cu(II) and Pb(II). Additionally, the effect of surfactants (CTAB, SDS and Triton X-100) on Tl(I) peaks was tested. The measurements were carried out in a solution containing $0.0265 \text{ mol}\cdot\text{dm}^{-3}$ $\text{CH}_3\text{COONH}_4$, $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ CH_3COOH and $0.0235 \text{ mol}\cdot\text{dm}^{-3}$ NH_4Cl buffer solution of pH 4.6, $1\cdot 10^{-5} \text{ mol}\cdot\text{dm}^{-3}$ Na_2EDTA , $7.5\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$ Bi(III) and $1\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I). No influence on thallium peak (error in excess of $\pm 10\%$) was observed after addition of $4\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ Cu(II), $8\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ of Zn(II), $2\cdot 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$ of Fe(III), $1\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ of Cd(II), $8\cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-3}$ of Pb(II), $2 \text{ mg}\cdot\text{dm}^{-3}$ Triton X-100, $0.5 \text{ mg}\cdot\text{dm}^{-3}$ SDS and $0.1 \text{ mg}\cdot\text{dm}^{-3}$ of CTAB.

3.6. Application

The developed voltammetric method for the determination of Tl(I) at the SPCE/MWCNTs/BiF was used in the analysis of spiked water samples collected from the Vistula river. The determinations were carried out for samples pre-acidified with nitric acid(V) to pH 2, and then mineralized for 3 hours in a UV mineralizer. The measurements were taken in a measuring cell to which 1 cm^3 of spiked with standard solution of Tl(I) water sample and the remaining reagents were added in amounts resulting from the optimization of the procedure. Fig. 5 shows the voltammograms obtained during the determination of Tl(I) in the spiked water samples from the Vistula river.

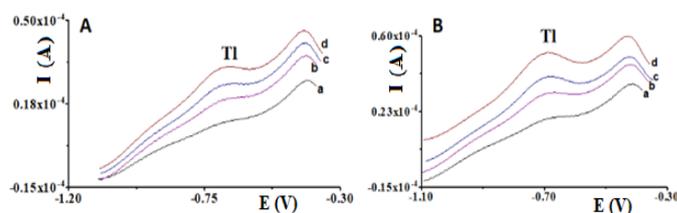


Fig. 5. SWASV curves obtained for the determination of Tl(I) in mineralized water from the Vistula river (A): a) 1 cm^3 sample + $1\cdot 10^{-8}$, b) as (a) + $1\cdot 10^{-8}$, c) as (a) + $2\cdot 10^{-8}$, d) as (a) + $3\cdot 10^{-8} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I), (B): a) 1 cm^3 sample + $2\cdot 10^{-8}$, b) as (a) + $2\cdot 10^{-8}$, c) as (a) + $4\cdot 10^{-8}$, d) as (a) + $6\cdot 10^{-8} \text{ mol}\cdot\text{dm}^{-3}$ Tl(I)

Table 2 summarizes the results obtained during Tl(I) determination in the spiked water samples from the Vistula river. On the basis of the value of recoveries, it can be concluded that the developed procedure can be used to determine Tl(I) in river waters with satisfactory results.

Table 2. The recovery values obtained during the determination of Tl(I) in the spiked water samples from the Vistula river

Tl(I) concentration ($1\cdot 10^{-8} \text{ mol}\cdot\text{dm}^{-3}$)		
Added	Determined \pm SD (n = 3)	Recovery (%)
1.0	1.06 ± 0.08	106.0
2.0	1.89 ± 0.04	94.5

4. Conclusions

In the presented article, a commercially available screen-printed sensor with carbon/multiwalled carbon nanotubes working electrode modified during analysis with bismuth film was applied for the first time for the determination of Tl(I). The developed square wave anodic stripping voltammetric procedure with the use of SPCE/MWCNTs/BiF allows for simple and quick determination of trace amounts of Tl(I). The bismuth plating of SPCE/MWCNTs enhances the thallium signal. A low detection limit of $2.8\cdot 10^{-9} \text{ mol}\cdot\text{dm}^{-3}$ was achieved. Negligible influence of metal ions as well as surfactants was observed. Moreover, the proposed SPCE/MWCNTs/BiF sensor has been applied for the determination of Tl(I) in the spiked river water samples with satisfactory results. Taking into account the sensitivity, selectivity and repeatability towards the determined Tl(I) it can be stated that the proposed sensor is a useful tool for thallium analysis in water samples, with low instrumental cost and a simple sample treatment.

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