IMPROVEMENTS IN METHODS FOR THE DETERMINATION AND SEPARATION OF TOXIC SELENIUM IONS

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A voltammetric method has been developed for the determination of trace amounts of Se (IV) using a carbon paste electrode modified with mercury and copper. The conditions for the formation of the modified electrode surface and the electrochemical determination of selenium were investigated. It was established that the optimum formation of the copper amalgam was achieved at a potential of 0.34 V, while the optimum accumulation time was 10 s. A 0.05 M formic acid solution was selected as the supporting electrolyte. The mechanism of the Se (IV) electrode process on the Hg/Cu/CPE surface was investigated, and the main electrochemical characteristics were determined. The proposed electrode provided a linear relationship between the analytical signal and selenium concentration in the range of 0.05–30.0 μg/mL, with a correlation coefficient of 0.996. The detection limit for Se (IV) was 0.05 μg/mL. The effect of accompanying ions on the determination of selenium was also studied. The results showed that the proposed method can be used for the determination of Se (IV) in complex multicomponent systems, including samples containing zinc compounds and other accompanying elements.
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Introduction

Growing demand from the industrial sector and stricter regulations on water and wastewater treatment are the main drivers of environmental protection. One of the key factors is the tightening of regulations on water and wastewater treatment at industrial sites. However, the high cost of water and wastewater treatment technologies is holding back innovation in this field. Looking ahead, significant investment in research and development in water treatment technologies will provide opportunities to mitigate these negative factors in the coming years. Given the depletion of water resources, it is imperative to urgently review the existing wastewater management infrastructure and take the necessary steps to increase water treatment capacity at industrial facilities. To avoid environmental damage and the spread of harmful diseases, wastewater must be treated before being discharged into the environment [1-3].

It should be noted that wastewater contains hazardous chemicals from various water treatment facilities. In such a situation, the use of appropriate water quality control processes to remove toxic elements from wastewater and ensure it is safe for consumption is of significant economic importance. One of the toxic elements that has a negative impact on the environment is selenium, which can be found in industrial emissions from fertiliser, fibreglass, steel and copper production plants, as well as in emissions of selenium into the atmosphere and water bodies, and from fossil fuel power stations. Selenium is also present in coal and oil, and is released into the atmosphere when fuel is burned in power stations; it is also found in industrial waste from electronic and computer equipment, such as batteries and accumulators. During soil cultivation, selenium enters the soil via fertilisers and pesticides; furthermore, it is present in small doses in water and soil [4-6].

During ore extraction, this non-metal is released and also enters the environment. Selenium, along with Hg, Cd, Pb, Si, Zn, Ni and Co contained in waste, is one of the most environmentally hazardous elements and requires special attention during environmental assessments. Interest in determining selenium levels in environmental samples is currently driven by its toxic properties at relatively low concentrations, as well as its importance as a trace element in human and animal life. This trace element, which is essential for living organisms, is biologically essential for humans, animals and birds, and enters the human body via food and water through the gastrointestinal tract [7-9].

The selenium content in the human body is 13 mg. Selenium is part of the protein component of tissues in the form of selenium-substituted amino acids, as well as a component of thyroid enzymes, and has a positive effect on visual acuity. Selenium is concentrated in the liver and kidneys, with a significant proportion found in bone tissue. Selenium is classified as a highly toxic element. Its toxic mechanism involves disruption of sulphur metabolism and an inhibitory effect on certain enzymes. Selenium suppresses biological oxidation processes, disrupts phosphorus-calcium metabolism, and is similar in its toxic effects to arsenic. Maximum permissible concentrations of selenium in foodstuffs, mg/kg: meat and fish products – 1.0; bread, vegetables, fruit – 0.5. The minimum lethal dose of selenium for humans is 2–4 mg/kg body weight.

A voltammetric method for the determination of selenium using a mercury-copper-carbon paste electrode has been developed. To modify the electrode for the determination of selenium (IV), a 0.01 M mercury nitrate solution and a carbon paste electrode (CPE) were used. When a voltage of -2.0 V was applied, mercury films formed; the mercury-carbon-paste electrode (M-CPE) was then coated with a 0.01 M copper nitrate solution and a voltage of 0.34 V was applied. As a result, a copper amalgam formed on the surface of the mercury film, which was used to determine trace amounts of selenium (IV). The mechanism of selenium electrode processes on the surface of the mercury-copper-carbon-paste electrode (MMPE) was investigated, and the number of electrons in the half-wave potential was determined [10]. Optimal conditions for selenium determination were selected: accumulation time – 10 seconds; nature of the background electrolyte – 1.0 ml of 0.05 M formic acid; accumulation potential of depolarisers at the working electrode. The half-wave potential for selenium was 0.25 V. When using the modified electrode, the influence of interfering cations on the determination of selenium (IV) ions was reduced, and a concentration range for the calibration curve with a correlation coefficient R = 0.9996 was found.

The authors in [11] proposed a new analytical method for the simultaneous determination of mercury and selenium in fish samples using atomic fluorescence spectrometry (AFS). The method was applied to determine the mercury and selenium content in tinned sardines. Mercury concentrations ranged

from 0.057 to 0.203 μg g^{-1}, and selenium concentrations from 1.76 to 2.21 μg g^{-1}, yielding an average molar ratio (Se:H g) equivalent to 36.

A number of researchers, when determining selenium spectrophotometrically using a chromogenic reagent, developed a method based on the reaction of selenium with potassium iodide in an acidic medium with the release of iodine [12].

In [13], the authors used flow injection photochemical vapour-phase atomic absorption spectrometry (FI-PVG-AAS) to determine selenium ions, optimising the influence of variables on the photochemical formation process. Selenium derivatives were transferred to an externally heated atomisation chamber with a mixture of argon and hydrogen, and selenium was detected at 196.0 nm. Nitrogen was used as the carrier gas for the flow injection system. It was found that nitric acid interferes with the determination; up to 3.0 wt.% HNO_3 can be removed during the reduction of Se(VI) to Se(IV) with hydrochloric acid.

The authors of [14] determined the selenium content in sulphuric acid using hydride generation atomic fluorescence spectrometry (HG-AFS). The results showed that the selenium content in the sulphuric acid samples ranged from 0.332 mg/kg to 2.576 mg/kg. In [15], a highly sensitive analytical method for the determination of selenium was proposed using the platinum-containing tungsten coil hydride generation method on an atomic absorption spectrometer; the detection limit for Se was 21.1 ng/L. The proposed method was also applied to the determination of selenium in certified reference materials.

Currently, in the development of methods for determining selenium using physicochemical methods, the limits of the minimum detectable concentrations have practically been reached, with detection limits at the nanogram per litre (ng/L) level; various studies are also being conducted on the separation and concentration of ultramicro quantities of selenium ions using various modified electrodes. Thus, for the isolation of selenium from purified sulphur using a combined extraction-atomic absorption method, two variants have been proposed: extraction of selenium diethyldithiocarbamate with ethyl acetate from a sulphuric acid solution at pH=3, and extraction of selenium dithionate with chloroform from a sulphuric acid solution with a concentration of 5.25–6.0 M [16]. The accuracy of selenium determination in purified sulphur was confirmed by the sample variation method. The limit of determination for selenium is 1×10^{-5} % by mass; the Sr values in the selenium content range are 2×10^{-5}–1×10^{-4} % by mass, varying from 0.10 to 0.12.

Materials and method

Given the potential toxicity of selenium at elevated concentrations, regular monitoring of its content in environmental and biological samples is necessary. In natural waters, selenium is mainly present in the form of oxyanions, SeO_3^{2-} and SeO_4^{2-}, whose different chemical properties and mobility determine their behaviour in aquatic systems. Therefore, the development of reliable methods for the determination and speciation of selenium remains an important analytical task. In particular, methods based on the determination of selenium oxyanions at low concentration levels in water samples have been reported [17]. Various methods have been developed for the determination of selenium species in food and biological matrices. For example, different selenium species in selenium-enriched polysaccharides were determined by HPLC-ICP-MS after enzymatic hydrolysis with proteinase K [18]. Selenium in rice was determined by atomic fluorescence spectrometry [19], while the determination of selenium in food and environmental samples was also carried out using hydride-generation high-resolution continuum-source quartz-furnace atomic absorption spectrometry [20,21]. In green tea samples, fluorescence spectrometry was used for selenium determination [22], whereas elemental selenium in yeast was determined by anion-exchange HPLC-ICP-MS [23].

Methods based on chromatographic separation coupled with atomic spectrometric detection have also been applied to the simultaneous determination and speciation of selenium and other elements. Thus, the simultaneous speciation of arsenic and selenium in seafood and onion samples was performed by high-performance liquid chromatography coupled with inductively coupled plasma mass spectrometry [24]. Selenium in garlic and onion was determined by electrothermal atomic absorption spectrometry after a preliminary separation and enrichment procedure [25]. A vortex-assisted liquid–liquid microextraction method was also developed for the determination of trace selenium in natural water, agricultural soil and food samples [26]. ICP-MS has been applied to the determination of arsenic, selenium, tin, antimony, lead and bismuth in nickel–chromium alloys [27].

The above methods provide high sensitivity and satisfactory analytical characteristics; however, they generally require expensive instrumentation, multistep sample preparation or complex separation procedures. Consequently, the development of relatively simple and sensitive electrochemical methods for selenium determination remains relevant. Spectrophotometric approaches have also been used in selenium-related analytical studies. For example, selenium dioxide has been employed in the spectrophotometric determination of tetracycline through the formation of a coloured reaction product [28]. In addition, potassium iodide and starch have been proposed as reagents for the rapid spectrophotometric determination of trace amounts of selenium [29]. Other instrumental approaches have been applied to the determination of selenium in biological and environmental samples. These include neutron activation analysis for selenium determination in hair [30], inductively coupled plasma mass spectrometry for the analysis of biological and food samples [31,32], energy-dispersive X-ray fluorescence spectrometry for the determination of selenium and other elements in plastic materials [33], and electrospray ionization mass spectrometry for the identification of selenium-containing species [34]. High-performance liquid chromatography coupled with inductively coupled plasma optical emission spectrometry has also been used for the determination of selenium species in biological samples [35].

More complex combinations of chromatographic, mass-spectrometric and nuclear magnetic resonance techniques have been applied to the characterization of selenium-containing compounds and selenium fractions in biological matrices [36–38]. Despite the high sensitivity of many existing methods, their practical application may be limited by the need for expensive equipment, complicated sample preparation and specialized analytical procedures. This creates a need for alternative methods that combine adequate sensitivity and selectivity with a relatively simple procedure and accessible instrumentation. In this regard, the development of a voltammetric method for the determination of Se(IV) using a mercury–copper-modified carbon-paste electrode is of practical interest.

Results and Discussion

Carbon-paste electrodes (CPEs) are widely used in electrochemical analysis because their composition and surface properties can be readily modified by incorporating electroactive substances into a carbon-based paste. The electrode is generally prepared from conductive carbon material and a binder, while the electrochemical process takes place at the electrode–solution interface. The analytical response of a CPE depends on the composition and dispersion of the electroactive component, the nature of the binder and the conditions of the electrochemical measurement. Depending on the nature of the electroactive component and the products formed during electrolysis, several types of electrochemical transformations may occur at carbon-paste electrodes. These include oxidation or reduction of a solid component with the formation of soluble products, solid-phase transformations accompanied by the formation of a new solid phase, and electrochemical reactions involving

adsorbed species. In practice, the analytical signal is usually related to the current generated during the electrochemical transformation of the analyte, and its magnitude depends on the concentration and distribution of the electroactive component in the electrode material.

The choice of the working electrode is particularly important in stripping voltammetry because the electrode material and its surface properties directly affect analyte accumulation, the subsequent stripping process, sensitivity, selectivity and the reproducibility of the analytical signal. Therefore, the selection and modification of the electrode surface are essential stages in the development of voltammetric methods for the determination of trace elements. In the present study, a carbon-paste electrode modified with mercury and copper was investigated for the determination of Se(IV). The modification of the electrode surface was aimed at improving the accumulation of selenium and increasing the sensitivity of the stripping voltammetric response. The electrochemical behaviour of Se(IV) at the modified electrode was studied, and the main conditions affecting the analytical signal were optimized.

Methods for the determination of selenium(IV) using various solid electrodes: 1.0 ml of a Se(IV) solution with a concentration of 1 μg/ml was added to the sample (20.00 ml), then 5.0 ml of 0.2 M sodium sulphite was added and the concentration was adjusted to the mark with deionised water, after which the solid electrodes were immersed and stripping voltammetry readings were recorded. The data obtained are presented in Table 1 and Figure 1.

From Figure 1 it can be seen that the most accurate analytical signals of selenium are obtained on carbon-paste electrodes, which have a large operating potential range from -1.0 to +1.5 V.

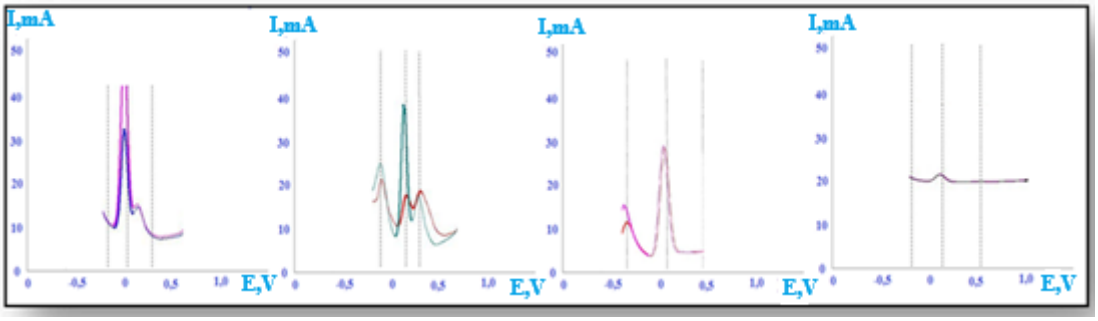


Fig.1. Current-voltage curves (peaks) of selenium obtained with electrochemical electrodes of different nature: 1) Platinum 2) Silver; 3) Graphite; 4) Gold.

Table 1. Comparative results of selenium determination depending on the nature of the electrodes

Contained Se(IV), (analysis conditions)	h, mm	Electrode type	Found Se(IV), mcg	N	S	S_r
Se (IV) 10.5 mcg/ml (background 0.05M formic acid) d.t.= 2.5 mcA En = 0.744V tn= 10s	18	Gold	10.22±0.36	5	0.311	0.030
	22	Silver	10.40±0.18	5	0.158	0.015
	27	graphite	10.49±0.08	5	0.067	0.006
	10	Platinum	10.42±0.16	5	0.143	0.014

As shown in Table 1, the graphite electrode provided a well-defined analytical signal and demonstrated the best electrochemical characteristics among the electrodes

investigated. The shape of the working surface, which approximates a hemispherical geometry, contributed to a high current density and a stable analytical response. The relative standard deviation did not exceed 0.006, indicating good reproducibility of the measurements. These characteristics were considered important for the subsequent development of the stripping voltammetric method for Se(IV) determination.

It is known that the introduction of small amounts of auxiliary metal ions into the analyzed solution can improve the electrochemical response, reduce the detection limit and enhance the reproducibility of metal determination [39,40]. In the present study, mercury and copper were selected as modifying components. During the electrochemical deposition step, these components form a modified surface on the carbon-paste electrode, resulting in the Hg/Cu/CPE system. The in situ formation of the mercury-containing surface provides a relatively uniform distribution of the deposited phase and can improve the accumulation of the analyte during the stripping voltammetric determination.

The interaction between selenium and mercury is also of interest because mercury–selenium compounds can form under appropriate conditions. The formation of HgSe has been investigated in relation to the interaction of mercury with amorphous selenium, and the properties of the resulting selenium-containing phase depend on the reaction conditions and the presence of oxygen [41,42]. However, this information should be considered only as a basis for understanding possible Hg–Se interactions and should not be directly interpreted as evidence of HgSe nanoparticle formation under the conditions used in the present electrochemical experiment.

The carbon-paste electrode was prepared using spectrally pure graphite powder (grade B-3) and purified wax as the binder. The graphite powder was preliminarily ground in a porcelain mortar and passed through a sieve to obtain a particle size of approximately 160 mesh. The required amounts of graphite and wax were weighed on an analytical balance. The wax was melted in a porcelain crucible in a sand bath at 75–85 °C, after which the graphite powder was gradually added with continuous stirring. The graphite-to-wax ratio was 1:1 by mass. The resulting mixture was then heated to approximately 150 °C and, with continuous stirring, gradually cooled to approximately 90 °C. At this temperature, the carbon paste was introduced into polyethylene tubes 60 mm in length and 0.3 mm in internal diameter using a syringe. A copper or silver wire with a diameter of 0.05 mm and a length of 90 mm was used as the electrical contact. The prepared carbon-paste electrodes were allowed to stand for 7–10 days to ensure complete hardening and stabilization of the paste. This procedure improved the mechanical stability of the paste and reduced the risk of surface damage during mechanical renewal of the electrode.

A 0.01 M solution of mercury(II) nitrate and CPE were used to modify the electrode for the determination of selenium(IV). When a voltage of -2.0 V was applied, mercury films formed; the R-CPE was then immersed in a 0.01 M copper(II) nitrate solution and a voltage of 0.34 V was applied. As a result, a copper amalgam formed on the surface of the mercury film, which was used to determine trace amounts of selenium (IV).

$$C + Hg^{+2} _ CHg + Cu^{2+} — CHg(Cu) \quad (1)$$

$$C + Hg^{+2} _ CHg + Cd^{2+} — CHg(Cd) \quad (2)$$

The procedure for the determination of selenium using the Hg/Cu-modified carbon-paste electrode was as follows. An aliquot of 1.0 mL of the Se(IV) standard solution with a concentration of 10.0 μg/mL was transferred into an electrochemical cell with a total volume of 20.0 mL. Then, 5.0 mL of 0.2 M sodium sulfite solution was added, and the solution was diluted to the mark with deionized water. The modified solid electrodes were subsequently immersed in the analytical solution, and the corresponding voltammetric signals were recorded. The resulting voltammograms are shown in Fig. 2.

$$SeO_3^{2-} + Cu(Hg) _ Se^{2+} + Cu^{2+} \quad (3)$$

$$Se^{2+} + Cu^{0} _ Cu^{2+} + Se^{0} \quad (4)$$

The electrochemical reduction of the accumulated selenium-containing species to elemental selenium (Se^0) is accompanied by the appearance of a well-defined peak on the voltammogram. The peak current is directly related to the concentration of Se(IV) in the analyzed solution and can therefore be used as the analytical signal. The experiments demonstrated that the use of the Hg/Cu-modified carbon-paste electrode (Hg/Cu/CPE) improved the sensitivity and efficiency of Se(IV) determination. The voltammograms obtained using the modified electrode showed well-defined analytical signals for copper and selenium (Fig. 2). It is known that, in stripping voltammetry, the height and shape of the analytical peaks are strongly influenced by the potential applied during the preconcentration stage [43]. Therefore, the effect of the copper amalgamation potential on the analytical signal was investigated. As shown in Fig. 3, increasing the copper amalgamation potential resulted in an increase in the analytical signal up to 0.34 V. At this potential, the analytical signal reached a plateau, indicating complete copper amalgamation. Further increase in the potential did not result in a significant increase in the signal. Therefore, an amalgamation potential of 0.34 V was selected as the optimum value for the subsequent experiments. The selection of the appropriate amalgamation potential is important for obtaining a uniform and reproducible modified electrode surface. An insufficient potential may result in incomplete copper deposition, whereas excessively high potentials may alter the composition and electrochemical properties of the deposited phase. Therefore, the optimized potential of 0.34 V was used in the subsequent determination of Se(IV).

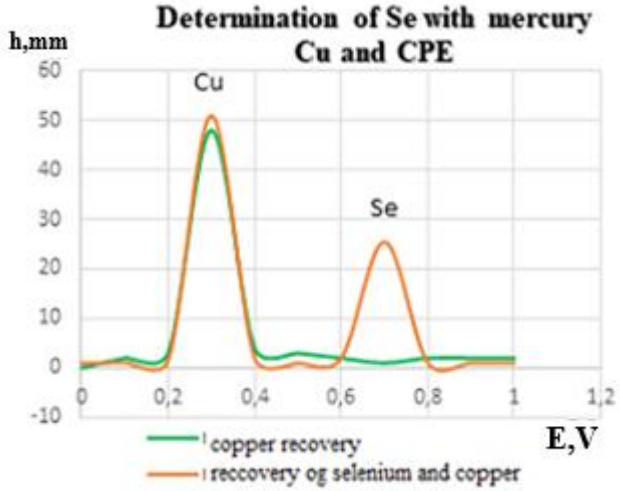


Fig.2. Voltammograms for the determination of selenium using a Hg/Cu-modified carbon-paste electrode**.**

When using the mercury-copper-carbon electrode amalgamation method to study the amalgamation potential, 1.0 ml of a Cu(II) solution with a concentration of 10.0 μg/ml and 5.0 ml of 0.2 M sodium sulphite are added to a 25.0 ml test tube, and dilute to the mark with deionised water. Solid electrodes are immersed in the solution, creating different voltages, and the accumulation potential of the analytical signal is measured; the data obtained are shown in Fig. 3. The accumulation time of the electroactive substance on the electrode in stripping voltammetry is also one of the key parameters affecting the height of the anodic peaks.

Figure 3 shows that the analytical signal reaches a plateau at an amalgamation potential of 0.34 V, indicating that copper amalgamation is essentially complete at this potential. Therefore, an amalgamation potential of 0.34 V was selected for subsequent experiments. Several approaches can be used to improve the sensitivity of stripping voltammetric determinations, including reducing the current range, adjusting the recorder scale and increasing the accumulation time of the analyte on the electrode surface [44]. The effect of the first two parameters is primarily determined by the design and sensitivity of the instrument, whereas the influence of the accumulation time on the analytical signal depends on the nature of the analyte and its concentration in the analyzed solution

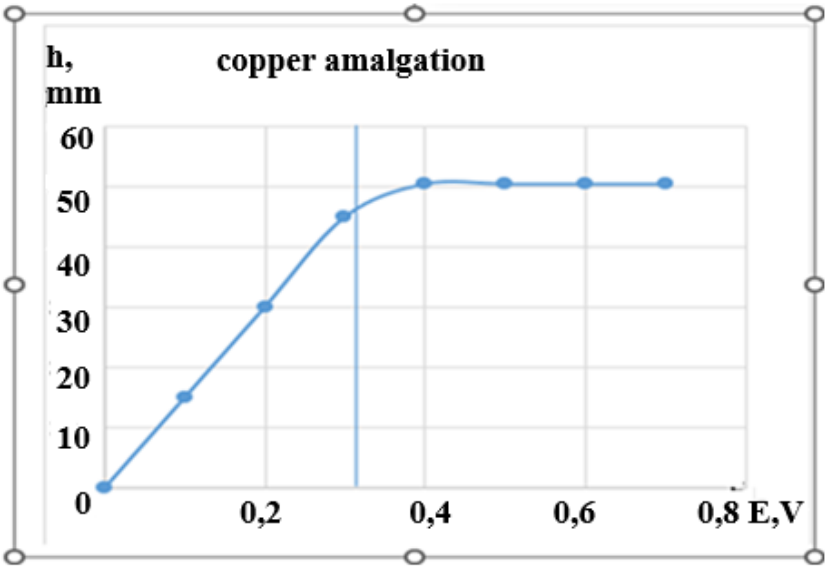


Fig.3. Effect of the copper amalgamation potential (E_a) on the analytical signal.

The accumulation time of copper on the surface of the Hg/Cu-modified carbon-paste electrode was therefore investigated. For this purpose, 1.0 mL of a Cu(II) solution with a concentration of 10.0 μg/mL, 1.0 mL of 0.05 M formic acid and 5.0 mL of 0.2 M sodium sulfite solution were introduced into an electrochemical cell with a total volume of 25.0 mL. The solution was diluted to the mark with deionized water. The modified solid-state electrode was then immersed in the solution, and the analytical signal was recorded at different accumulation times. The dependence of the copper peak height on the accumulation time is presented in Table 2 and Fig. 4.

Table 2. Dependences of copper peak heights on the accumulation time on modified carbon-paste electrodes

Peak height, modification time	Eim= 0,34 V; CCu = 1,0 mcg/ml						
h,mm	10	15	25	25	30	30	30
t, s	2	4	6	8	10	20	30

As shown in Table 2 and Fig. 4, the copper peak height increased with increasing accumulation time up to 10 s and subsequently remained practically constant. Therefore, an accumulation time of 10 s was selected as the optimum value for the determination of copper. Further prolongation of the accumulation time did not result in a significant increase in the analytical signal. The composition of the supporting electrolyte is an important factor affecting the formation and intensity of the analytical signal. Therefore, the electrochemical

behaviour of selenium was investigated in various supporting electrolytes to select the optimal operating conditions. The following media were examined: hydroxide, chloride and thiocyanate solutions of alkali metals and ammonium; sulfuric, phosphoric, acetic and formic acids; Britton–Robinson and acetate buffer solutions; and mixtures of ammonium hydroxide and acetic acid over a range of concentrations and pH values. The procedure for investigating the effect of the supporting electrolyte on the determination of selenium was as follows. An aliquot of 1.0 mL of a Se(IV) solution with a concentration of 10.0 μg/mL and 2.0 mL of the corresponding supporting electrolyte were introduced into an electrochemical cell with a total volume of 25.0 mL. Then, 5.0 mL of 0.2 M sodium sulfite solution was added, and the solution was diluted to the mark with deionized water. The modified solid-state electrode was immersed in the solution, and the analytical signal was recorded. The results are presented in Table 3 and Fig. 5.

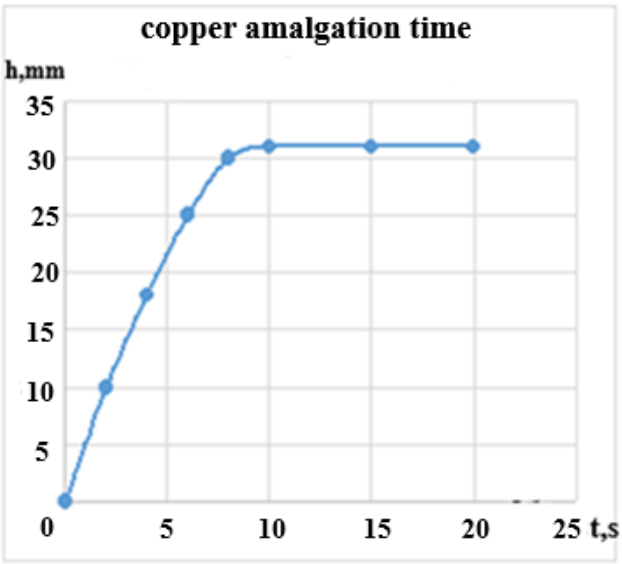


Fig.4. Dependence of the height of the peaks of a) copper on the time of their accumulation on modified carbon-paste electrodes

Figure 5 shows that, when determining Se(IV) by stripping voltammetry, the best analytical signal is obtained in the presence of a background electrolyte consisting of 0.05 M formic acid. All subsequent studies were carried out using this optimal background electrolyte.

The data in the table show that the highest values of the selenium analytical signal are observed when 0.05 M formic acid is used. Table 4 shows the relationship between the height of the selenium peaks and the concentration of background electrolytes.

The data in the table indicate that the optimal concentration of the background electrolyte for the determination of selenium is 1.0 ml of 0.05 M formic acid.

Method for the determination of selenium: 1.0 ml of Se(IV) of various concen-ra-tions, 1.0 ml of 0.05 M formic acid and 5.0 ml of 0.2 M sodium sulphite were added to the test cell (25.00 ml), then the volume was made up to the mark with deionised water. After this, solid electrodes were immersed and the voltammetry values were measured with dissolution in separate solutions. The data obtained are presented in Fig. 6.

Figure 6 shows that distinct peaks are formed at a selenium concentration of 25 μg/ml. Within this concentration range, the calibration curve exhibits a linear relationship, with a correlation coefficient approaching one (R = 0.9996).

The accuracy of the developed stripping voltammetry method for the determination of selenium was investigated using the 'input-found value' method at various selenium concentrations. As an example, Table 5 presents the results of the accuracy assessment of the developed stripping voltammetry methods, obtained from multiple (at least 5) parallel determinations of selenium ions.

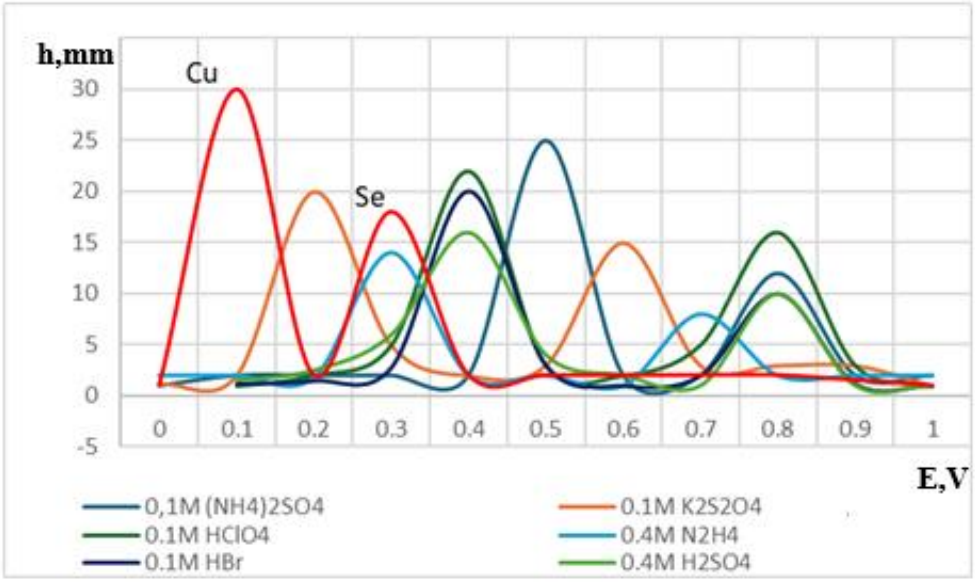


Fig.5. The influence of various background electrolytes on the determination potential during IV determination of Se(IV).

Table 3. Results of studying the influence of the nature of the background electrolyte on stripping voltammetric determination of selenium (d.t. 1,0 mkA; τ=10 s; C_{Se} =10,0 mcg/dm^3; n=5)

№	Nature and concentration of background electrolyte	Found Se, mkg (P=0,95; $\bar{x} \pm \Delta X$)	S	Sr
1	0.4 M H_2SO_4	10.02±0,16	0.129	0.013
2	0.4 M N_2H_4	9.87±0,26	0.213	0.022
3	0.1M $K_2S_2O_5$	9.68±0,54	0.434	0.045
4	0.1 M HBr	9.79±0,49	0.397	0.041
5	0.1 $(NH4)_2SO4$	10.02±0.14	0.114	0.011
6	0.05M formic acid	9.98±0,14	0.115	0.012
7	0.1 M HCIO4	9.84±0.40	0.320	0.033

Table 4. Results of studying the dependence of selenium (IV) on the background electro-lyte concentration (C_{Se}=10,0 mcg/dm^3; d.t. = 1,0 mA, τ=10s, P=0,95)

Background concentration, mcg/l	Найдено Me, Found Me, mcg ($\bar{x} \pm \Delta X$)	n	S	S_r
0.25	9.99±0.13	5	0.106	0.011
0.5	9.95±0.13	5	0.107	0.011
1.0	9.98±0.15	5	0.119	0.012
2.0	9.94±0.12	5	0.097	0.010

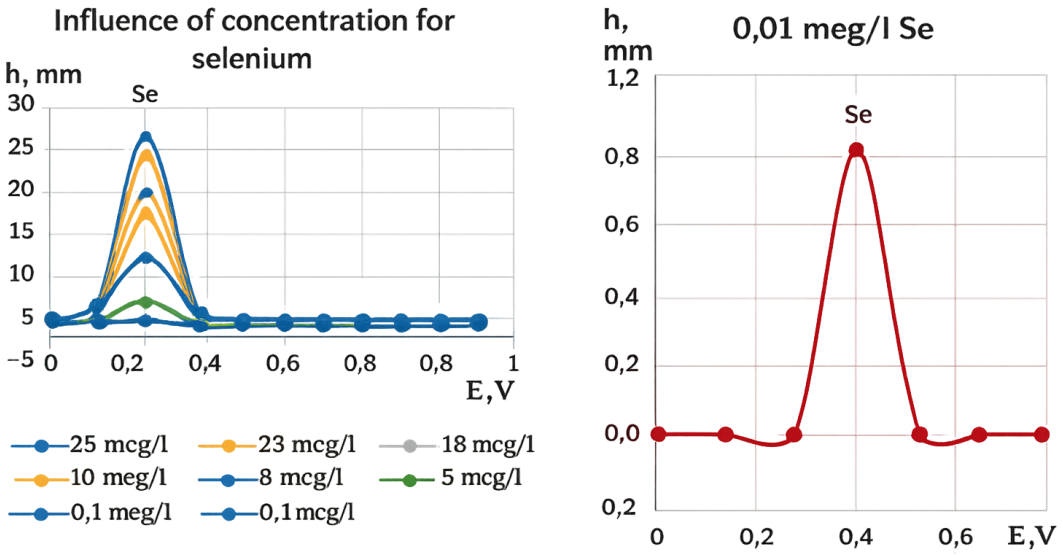


Fig.6. Effect of concentration on the potential of stripping voltammetric determination in the determination of Se(IV)

Table 5. Results of stripping voltammetric determination of metals in their individual solutions

Introduced Me mcg		Found Me, mcg ($P=0.95$; $n=5$; $\bar{x} \pm \Delta X$)	S	Sr
Se	1.0	0.98±0.10	0.081	0.083
	3.5	3.45±0.16	0.127	0.037
	5.0	4.93±0.17	0.136	0.028
	6.0	5.94±0.18	0.143	0.024
	8.3	8.43±0.20	0.158	0.019
	10.6	10.69±0.20	0.159	0.015
	14.8	14.93±0.20	0.163	0.011
	17.3	17.22±0.21	0.167	0.010
	19.4	19.31±0.22	0.174	0.009
	22.3	22.41±0.23	0.186	0.008
	25.4	25.45±0.24	0.193	0.008
	28.3	28.19±0.25	0.198	0.007
	31.5	31.58±0.25	0.203	0.006
	33.0	33.15±0.25	0.205	0.006
	35.0	35.21±0.26	0.209	0.006
	37.2	37.44±0.27	0.215	0.006
	38.5	38.41±0.29	0.231	0.006
	39.0	39.21±0.30	0.243	0.006
	39.5	39.43±0.30	0.245	0.006
	40.0	40.09±0.31	0.251	0.006

Table 5 shows that the selenium concentrations determined by the proposed stripping voltammetric method were in good agreement with the amounts introduced into the analytical solutions. The relative standard deviation (Sr) ranged from 0.006 to 0.083, indicating good reproducibility of the measurements. The lower limit of detection for selenium was 0.05 μg/mL, demonstrating the high sensitivity of the developed method. To investigate the electrochemical behaviour of selenium at the Hg/Cu-modified carbon-paste electrode, the half-wave potential and the number of electrons involved in the electrode

process were determined. For this purpose, selenium solutions of different concentrations were analyzed, and the corresponding analytical signals were recorded. The logarithmic values of the wave heights were then calculated. The results are presented in Table 6 and Fig. 6. Under the optimized conditions, the dependence of the half-wave potential on the concentration of selenium ions was investigated using the Lingane method [45,46]. According to the Lingane approach, the relationship between the half-wave potential and the logarithm of the concentration of the electroactive species can be used to determine the number of electrons involved in the electrode process from the slope of the resulting linear dependence.

The determination was performed as follows. An aliquot of the selenium solution of the required concentration, 1.0 mL of 0.05 M formic acid as the supporting electrolyte and 5.0 mL of 0.2 M sodium sulfite solution were introduced into a 25.0 mL electrochemical cell. The solution was diluted to the mark with deionized water, after which the modified electrode was immersed and the analytical signal was recorded. The experimental data obtained for the determination of selenium are presented in Table 6 and Fig. 6. The standard potential of the selenium electrode process was determined by extrapolating the dependence of the half-wave potential on the logarithm of the selenium concentration to zero concentration. The results are summarized in Table 6.

Table 6. Calculation of the electroreduction of selenium(IV) on a copper-mercury UPE

Metal	Background electrolyte	CMe, Ppm	E	I_{max}	$\lg \frac{I}{I_d - I}$	$E_{1/2}$
Selenium	formic acid	1.0	0.1	18	-0.351	0.25
		2.0	0.2	22	-0.139	
		3.0	0.3	26	0.124	
		4.0	0.4	30	0.311	
		5.0	0.5	34	0.504	
		6.0	0.6	38	0.715	
		7.0	0.7	42	0.923	

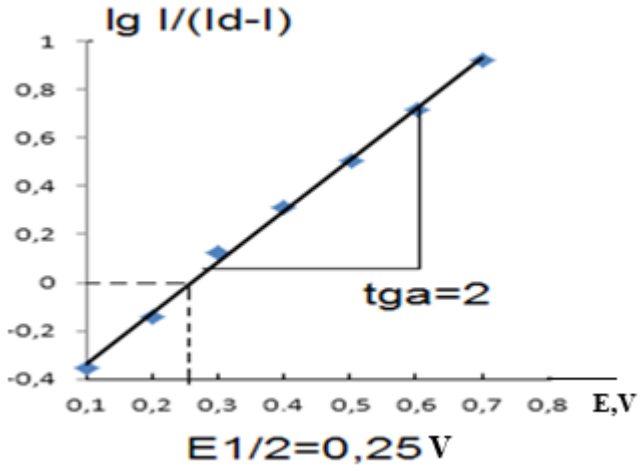


Fig.7 Graph for calculating the electroreduction of selenium

Figure 7 shows that, when selenium is being reduced, the number of electrons released is 2, which proves that selenium is reduced from +4 to 0, whilst copper donates 4 electrons. The half-wave potential of selenium found is 0.25 V, and with the optimal background

electrolyte, the half-wave potential shifts towards the positive side, leading to a 10-fold increase in selectivity.

By selecting the optimal conditions for obtaining the best analytical signal for selenium, it is impossible to fully guarantee the accuracy and reproducibility of the experimental data obtained without determining these conditions in individual solutions. As an example, Table 7 presents the results of an accuracy assessment of the developed dissolution voltammetry methods, obtained from multiple (at least 5) parallel determinations of selenium.

Table 7 shows that, when determining various selenium concentrations, the measured concentrations correspond to the calculated values, and the relative standard deviation does not exceed 0.114, which indicates good reproducibility and accuracy of the developed dissolution voltammetry methods.

The elements under investigation were quantified using an additive method, as the calibration curves are straightforward within the optimised metal concentration ranges (Fig. 8)

Table 7. Results of stripping voltammetric determination of metals in their individual solutions

C (Se) mcg	1	5	9	13	17	21	25	29	33	37	40
h (Se) mm	4	8	12	16	18	20	25	26	30	30	30

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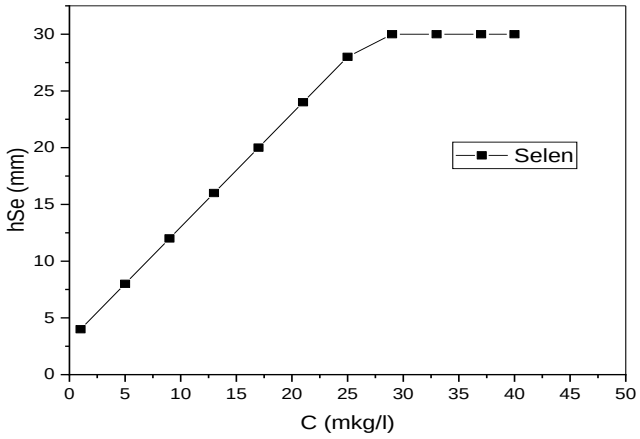


Fig.8. Selenium calibration graph

In order to obtain more complete information about the metrological characteristics of the developed stripping voltammetric methods for determining selenium, some metrological characteristics and analytical parameters were calculated, the values of which are presented in Table 8.

Table 8. Results of some metrological characteristics obtained during the development of stripping voltammetric methods for the determination of tellurium and selenium

Me	Linearity range of concentrations on calibration graphs, mcg/dm^3	r*	$C_н$, mcg/dm^3	C_{min}, mcg/dm^3
Se	0.050 – 30.0	0.996	0.05	30.0

* r – degree of correlation

The data in Table 8 show that the proposed stripping voltammetry methods for the determination of selenium are suitable for the analysis of zinc (dust, slag, etc.) and wastewater, as their lower detection limits,

calculated using the 3s criterion, are at or below the MPC (maximum permissible concentration).

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Since the zinc industry is characterised by varying composition and a wide range of concentrations of the elements it contains, from n 10^{2} (Cd^{2+}, Co^{2+}) to n 10^{-4} mg/dm ^{3} (Ag^{+}, Ge^{+2} , Sb^{+3} , Te^{4+}, Se^{4+}, etc.) and below, a variety of element substitution forms, and compositional variability, it is necessary to have data on the quantitative ratios and mutual influence of the elements present in order to establish the feasibility of determining the metals under study in real samples, as the accuracy and reproducibility of the data obtained depend largely on these factors.

It was therefore of interest to investigate the feasibility of determining selenium in model binary, ternary and more complex salt mixtures consisting of those cations that are frequently and widely present alongside the metals of interest.

Metals such as potassium, sodium, calcium and magnesium do not have a significant effect on the determination of selenium by stripping voltammetry and are therefore not considered. Cadmium, copper, zinc, manganese and iron are of greatest interest for studying the influence of accompanying elements; it was therefore necessary to determine their maximum permissible concentrations, which would not exert a significant interfering effect on the determination of selenium [47].

Method for the determination of selenium in the presence of interfering ions: 1.0 ml of a Se(IV) solution at a concentration of 10.0 μg/ml and 1.0 ml of 0.05 M formic acid, 0.1 μg (Fe(II), Fe(III), Zn(II), 0.01 μg Co(II), Cd(II), Ge(II), Sb(III) and Sn(II)), 5.0 mL of 0.2 M sodium sulphite, and the solution was made up to the mark with deionised water. The solid electrodes were then immersed whilst applying various voltages to them, and the voltammetric analysis values were recorded, taking into account interfering ions. The data obtained are presented in Fig. 9.

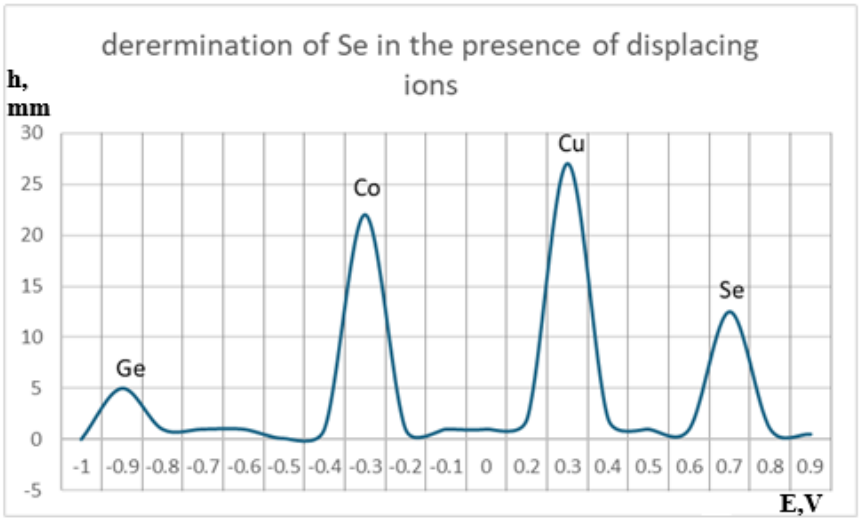


Fig.9. Effect of concentration on the potential of stripping voltammetric determination in the determination of Se(IV) with interfering ions.

Figure 9 shows that the determination of selenium by voltammetry with dissolution is possible in the presence of several metals in a single solution.

The figure shows that the selected modified mercury-copper solution (Fig. 9), with an optimal background electrolyte value, can be used in the presence of interfering ions or with a zinc matrix containing impurities of various metals. The data obtained for the determination of selenium are presented in Table 9.

Table 9. Results of the influence of extraneous accompanying interfering cations on the accuracy and reproducibility of stripping voltammetric determination of selenium (C_{Se} = 10.0 mcg; X – associated metal, P = 95, n=5)

[Se]:[X]	Found Se, mcg ($\bar{x} \pm \Delta X$)	S	S_r
Se:Cu 1:100	9.46±0.84	0.679	0.072
Se:Cd 1:100	9.40±1.35	1.085	0.115
Se: n 1:80 1:100	 9.99±0.47 9.96±0.45	 0,379 0,364	 0.038 0.037
Se:Te 1:10 1:25 1:50	 9.56±1.47 9.86±0.51 9.78±0.06	 1.180 0.414 0.052	 0.123 0.042 0.005
Se:Mn 1:100	9.94±0.14	0,.114	0.011
Se:Fe 1:100	9.82±0.16	0.130	0.013
Se:Ge 1:1	9.9±0.20	0.158	0.016
Se:Tl 1:1	9.86 ± 0.19	0.152	0.015
Se:Ni 1: 1	9.92 ± 0.22	0.179	0.018
Se:Sb 1: 1	9.92 ± 0.18	0.148	0.015

Table 9 presents the results obtained when establishing the maximum permissible concentrations of various foreign metls that interfere with the determination of selenium in binary mixtures at various ratios; the accuracy of these concentrations was verified using the ‘addition–subtraction’ method.

Table 9 shows that selenium can be determined with an acceptable margin of error in the presence of 100-fold amounts of cadmium, copper, iron and manganese, whilst zinc at such concentrations leads to overestimated values; therefore, the optimal ratio is [Se]:[Zn]=1:80. At 100 times the concentration of tellurium, the selenium peak is completely suppressed, whilst at 50 times the concentration, the results are underesti-mated; therefore, the optimal ratio is [Se]:[Te] = 1:25, at which accurate and reproducible selenium determination results are still observed. Determination of selenium in model mixtures. To establish the feasibility of determining selenium in real samples, model mixtures of various combinations were prepared to simulate waters of different origins.

Method for determining selenium in model mixtures: 1.0 ml of a Se(IV) solution with a concentration of 10.0 μg/ml and 1.0 ml of 0.05 M formic acid were added to the analysis cell (25.00 ml); multi-element solutions were then added and the volume was made up to the mark with deionised water, the composition of which is given in the table. Solid electrodes were then immersed, with various voltages applied to them, and voltammetric measurements were recorded during dissolution in model mixtures, i.e. binary mixtures [46,47]. The data obtained are shown in Fig. 10.

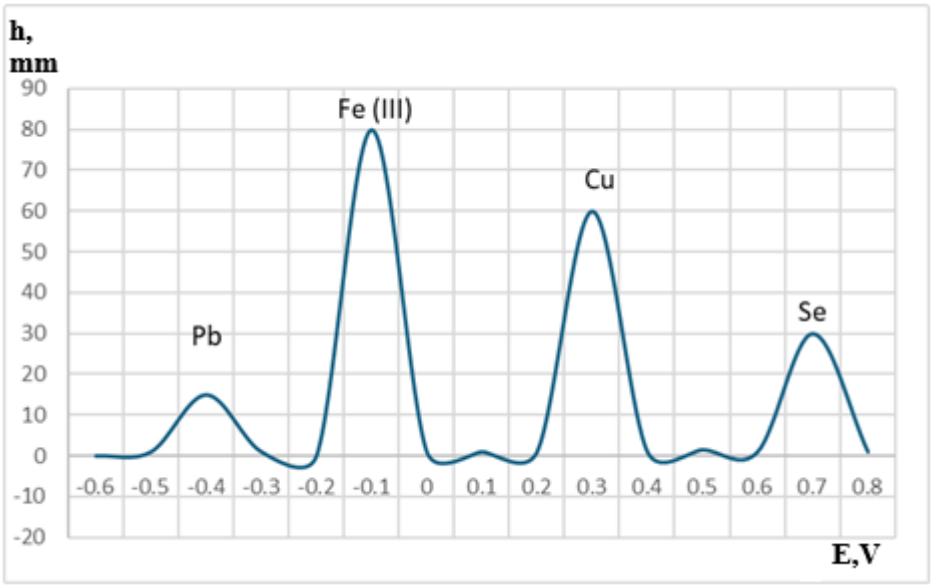


Fig.10. IV Determination of selenium in model mixtures.

From Figure 10 it can be seen that the chosen method and conditions for the stripping voltammetric determination of selenium in the analyzed mixtures were the same as when analyzing their individual solutions. Some of the data we obtained on the determination of the studied metals in binary, ternary and more complex model mixtures are presented in Table.

Table 10. Stripping voltammetric results determination of selenium in model mixtures n=5

Composition of the analyzed mixture, mcg	Found Se, mcg (P=0.95; $\bar{x} \pm \Delta X$)	S	Sr
Se(1.0)+ Cu(6.0)+ Pb(0.8)+ Fe(20)	0.98 ± 0.02	0.019	0.019
Se(2.0)+Pb(0.4)+Cd(0.4)+Fe(30)	2.01 ± 0.07	0.053	0.026
Se(5.0)+Cu(5.0)+Fe(25.0)	4.91 ± 0.69	0.552	0.112
Se(7.0)+Cd(0.75)+Co(8.0)+Cu(2.0)	7.02 ± 0.34	0.270	0.038
Se(7.5)+Fe(0.5)+(7.0)+Te(0.1)	7.40 ± 0.20	0.158	0.021
Se(8.0)+Cd(2.0)+Cu(5.0)+Fe(8.0)	7.86 ± 1.07	0.862	0.110
Se(10.0)+Cu(1.5)+Pb(60.0)+Fe(60.0)	9.90 ± 0.20	0.158	0.016

As can be seen from the data in Table 10, obtained by dissolution voltammetry, the determination of selenium in model binary, ternary and more complex mixtures is entirely feasible, and the relative standard deviation does not exceed 0.144 in any case, which indicates the comparatively good accuracy and reproducibility of the methods developed.

Conclusions

Based on the research carried out, it can be concluded that the CPE developed, in terms of operating mode, metrological, operational and other characteristics, are on a par with analogous solid electrodes. Using CPE as a basis, MCPE can be obtained during the course of experiments, which makes it possible to determine both elements for which mercury acts as an interfering agent and other metals for which mercury acts as a positive ‘substrate’; all

of this enhances the sensitivity of dissolution voltammetry methods for the determination of various metals. It has been established that the nature, concentration and pH of the background electrolyte have a significant influence on the selectivity and sensitivity of the determination of the metals under investigation. Experiments have shown that another factor influencing the analytical signal of the target metal is the accumulation potential of depolarisers at the working electrode; altering this potential makes it possible to increase the sensitivity of the metal determination and eliminate the interfering effect of accompanying sample components. The metrological characteristics of the results for the determination of the metals under investigation in model mixtures simulating real-world samples suggest that the developed dissolution voltammetry methods can be applied to analyse real-world samples simulating waters of various origins for selenium ion content.

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TOKSİK SELEN İONLARININ TƏYİNİ VƏ AYRILMASI METODLARININ TƏKMİLLƏŞDİRİLMƏSİ

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Civə və mis ilə modifikasiya olunmuş kömür pastası elektrodu (Hg/Cu/KPE) istifadə etməklə Se(IV)-ün mikromiqdarlarının təyini üçün voltamperometrik metod işlənilmişdir. Modifikasiya olunmuş elektrod səthinin formalaşması və seleniumun elektrokimyəvi təyini şəraitləri tədqiq edilmişdir. Müəyyən edilmişdir ki, mis amalqamasının optimal formalaşması 0.34 V potensialda, optimal toplanma müddəti isə 10 saniyə təşkil edir. Fon elektroliti kimi 0.05 M qarışqa turşusu məhlulu seçilmişdir. Hg/Cu/KPE səthində Se(IV) elektrod prosesinin mexanizmi öyrənilmiş və əsas elektrokimyəvi xarakteristikaları müəyyən edilmişdir. Təklif olunan elektrod analitik siqnal ilə seleniumun qatılığı arasında 0.05–30.0 mq/ml diapazonunda xətti asılılıq təmin edir (korrelyasiya əmsalı 0.996). Se(IV) üçün təyinat həddi 0.05 mq/ml təşkil etmişdir. Birlikdə müşayiət edən ionların seleniumun təyininə təsiri də tədqiq olunmuşdur. Nəticələr göstərmişdir ki, təklif olunan metod sink birləşmələri və digər müşayiətedici elementlər olan mürəkkəb çoxkomponentli sistemlərdə Se(IV)-ün təyini üçün istifadə edilə bilər.

Açar sözlər: *təyinetmə metodları, elektrodlar, təmizləmə, təhlükəli kimyəvi birləşmələr, selen.*