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СЕРИЯ ГЕОЛОГИИ И ТЕХНИЧЕСКИХ НАУК

♦

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CONTROL OF TRACE QUANTITIES OF METALS IN THE ENVIRONMENTAL SAMPLES, USING MERCURY-FILM INDICATIVE MICROELECTRODES

Abstract. Theoretical bases for the determination of trace quantities in the environmental samples by the method of stripping voltammetry, whose accuracy and reproducibility is associated with the use of the proposed mercury-film tungsten electrode, have been developed. The methods for the formation of a stable mercury film on a substrate of this material have been developed; the factors effecting the sensitivity and resolution of the film electrode have been studied.

Perspectiveness of using the electrochemical methods of analysis, in particular, stripping voltammetry, for exercising a control over toxic and heavy metals in technology-related and mineral raw materials of Kazakhstan has been demonstrated.

Key words: indicative electrode, mercury-film electrode, tungsten, stripping voltammetry, amalgamation.

Nowadays, the transformation of any technology process into a recycling waste-free production is one of the most important tasks. In this connection special attention should be paid to monitoring and regulation of the technology process itself and the determination of the content of toxic substances in industrial waste dumps and industrial wastewaters.

In connection with the accumulation of a large amount of secondary raw materials, containing harmful impurities, including heavy metals, the task of their detection and quantitative determination remains topical up till now. Only the data of analytical control may provide the people with a thread for managing the environmental purity and indicate the moment of necessary intervention for its protection from the accumulation of toxic substances and ecological poisons, whereto heavy metals are directly related. Passing into the human organism through air and water, including the chain: soil → water → animal, they cause various diseases.

The developed indicative microelectrodes on a substrate of the iron family metals and alloys on the basis of such metals, as well as a carbon-containing material, carbon-fiber reinforced plastic, which can be safely related to new means of controlling toxic substances have been earlier proposed as the means for exercising control over toxic and heavy metals [1].

Physical and chemical characteristics of these electrodes and the mechanism of the processes, proceeding at the electrode-solution interface have been studied at the stages of preparation of their surface and subsequent amalgamation. Optimal parameters of their functioning have been determined. A design of a rotating electrode has been developed, which has allowed the metrological characteristics to be improved and analytical signals of toxic elements, such as mercury, zinc, cadmium, lead and copper, to be detected when they are jointly present in the analyzed samples.

While carrying out an electrochemical analysis of the toxic metal compounds a large effect upon the cathode-anode processes is produced by the nature of the indicative electrode matrix, background composition and the presence of other components. The impurities, possessing the same or close values of

potentials, as well as the impurities, forming intermetallic compounds with the analyzed metals, especially hinder the registration of analytical signals. The indicated circumstances determine a necessity to solve a complex problem of development of electrodes, using inexpensive and available materials with the valuable physicochemical and electrochemical properties.

The creation of a mercury-film electrode (MFE) is one of the greatest achievements in the inversion methods of electroanalysis. MFE combines the advantages of a solid and mercury electrodes: it has a wide operating interval of potentials, sufficiently reproducible surface; it does not display, as a rule, any intermetallic interactions of the deposited metals. MFE is obtained by applying mercury to an inert electroconductive substrate [2, 3].

Upon the creation of new indicative electrodes special requirements are set forth for increasing their corrosion resistance, sensitivity, selectivity, expansion of the range of operating potentials, etc. The most promising in this respect are the mercury-film electrodes with an inert substrate, which make it possible to work within a very wide range of potentials, thereby expanding the analytical capabilities.

Graphite and glass carbon are usually used as a substrate. However, the use of such substrates does not ensure the formation of a uniform film due to the presence of microdefects (microscratches, chips, cracks) on the surface. Such material requires an additional treatment, involving certain physical and material costs. This may be excluded by using metal substrates.

It is known that mercury is isolated in the form of a uniform film only on amalgam-forming metals [4,5]. The drawbacks of mercury-film electrodes (MFE) on metal substrates are an instability of the thickness and composition of a mercury film as a result of the deep penetration of mercury into the metal and the formation of amalgams of different concentrations, as well as possible interactions of the analyzed metals, isolated on the electrode, with the substrate metal. These circumstances indicate an important role of a substrate in the electrode functioning.

Precious metals are usually used as metal substrates for mercury, which are inert to mercury and adhere well thereto [6, 7].

Method of procedure. Measurements by the method of stripping voltammetry were carried out with the help of PI-50-1.1 potentiostat and CBA-1 system, using molybdenum and tungsten as an electrochemical sensor, MB-50 alloy with the visible surface area of 0.2cm^2. Electrochemical regeneration of the electrode surface was carried out after each measurement, holding an electrode at the positive potential of 0.7 V and mechanical regeneration was carried out by grinding the working surface with a special paste (or Al_20_3 powder), then by filter paper. A silver chloride (Ag^+/AgCl) electrode (s.c.e.), whose potential in relation to a normal hydrogen electrode of 0.1 M KC1 is equal to +0.222 V at 20°C, was used as a reference; a platinum wire was used as an auxiliary electrode. Voltammograms were registered on GTDA-1 two-dimensional recording potentiometer in the mode of anode stripping voltammetry with a potential sweep rate of 0.1 V/s.

Experimental. A mercury-film electrode (MFE) combines the advantages of a solid and mercury electrodes: it has a wide operating interval of potentials, sufficiently reproducible surface; it does not display, as a rule, any intermetallic interactions of the deposited metals. MFE is obtained by applying mercury to an inert electro-conductive substrate [1-4].

It is known that mercury is isolated in the form of a uniform film only on amalgam-forming metals [4, 5]. The drawbacks of mercury-film electrodes (MFE) on metal substrates are an instability of the thickness and composition of a mercury film as a result of the deep penetration of mercury into the metal and the formation of amalgams of different concentrations, as well as possible interactions of the analyzed metals, isolated on the electrode, with the substrate metal. These circumstances indicate an important role of a substrate in the electrode functioning, which should ensure good adhesion with mercury and be inert thereto. With this purpose mainly precious metals are used [2-5]. Mercury-film electrodes on an iridium substrate allow one to determine the presence of Cd and Pb within the range of contents of 0.1–5 mg/l [3]. A mercury-film electrode of platinum with electrochemical regeneration of a mercury film has been studied in detail *in situ* in the presence of cadmium ions (C < 2-10" mol/l), providing good adhesion of mercury to a platinum substrate [6].

It has been shown that the electrochemical properties of the obtained MFE remain constant with the thickness of a mercury film of ~10 *{d*= 0.14 mm) and ~25 μm *(d*- 0.45 mm). It is assumed, that with an increase of the diameter *d* of Pt-film an edge effect becomes more pronounced. This requires an increase

of the film thickness. When using an immobile MFE at the background of 0.1 M $HC10_4$ strong signals of cadmium and indium (III) reduction have been recorded in the presence of complexonates, causing their ligand-induced adsorption.

Precious metals are mainly used as metal substrates for mercury, which have good adhesion to mercury and are inert in relation thereto [3-6, 9].

The material, proposed by us as a substrate, tungsten, possesses such physical properties as strength, hardness and elasticity, and has an obvious advantage over the precious metals by its cost (much more inexpensive as compared with them), being, therefore, an available constructional material. Besides, it is practically insoluble in mercury ($L= 6.8\cdot10^{-20}$ at.%) [10], which affords us ground to assume that MFE on its basis should correspond by its properties more to the mercury electrodes and mercury.

The mercury potential depends, to a large extent, on the nature of anions, which is connected with the tendency of ions of mercury (I) and (II) to form salts of low solubility and complex ions (table).

Standard mercury potentials in the solutions under study [9]

Electrode reaction	E_0,V (n.h.e.)	E_0, V (s.c.e.)
$Hg + 2OH^- = HgO + H_2O$	0.929	0.689
$2Hg + SO_4^{2-} = Hg_2SO_4 + 2e$	0.615	0.378
$2Hg + 2Cl^- = Hg_2Cl_2 + 2e$	0.268	0.031
$2Hg + 4Cl^- = HgCl_4^{2-} + 2e$	0.48	0.243
$2Hg + 2SCN^- = Hg_2(SCN)_2 + 2e$	0.22	-0.017
$2Hg + 2OH^- = Hg_2O + H_2O + 2e$	0.123	-0.114

In the non-complex-forming medium the standard mercury potential is shifted to the positive values up to ~ +0.4 ÷ +0.7 V. If any substance, forming an insoluble compound or complex with mercury ions, is present in the solution, the potential shifts to the less positive and even negative values the stronger the less soluble the precipitate is, the more stable the complex is, and the higher the concentration of the substance, forming the precipitate or complex, is. It is known that the precipitates with mercury are formed in the presence of ions of Cl^-, Br^-, I^-, N_3^-, OH^-, SH^-, S^{2-} etc., the complexes are formed with SCN^-, CN^-, SO_3^{2-}, $S_2O_3^{2-}$, EDTA, etc. [3].

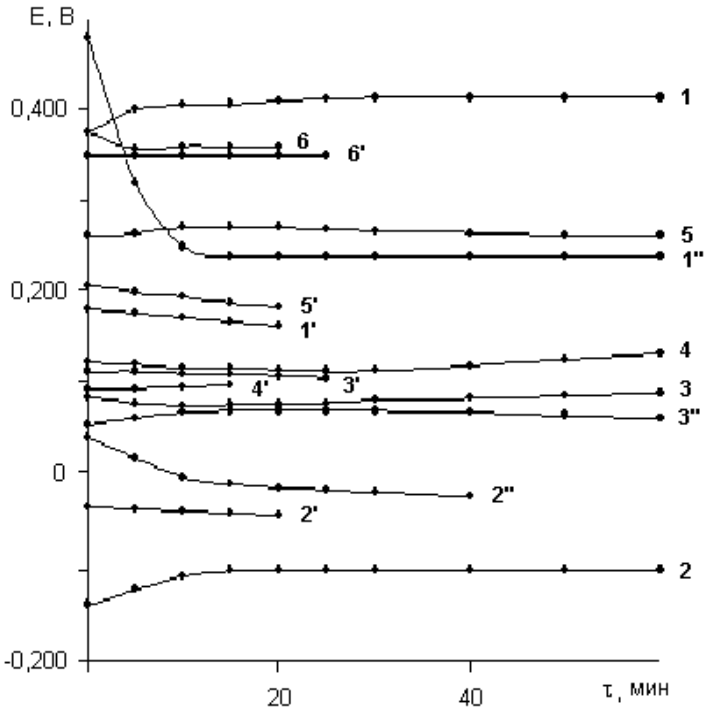

Figure 1 –
Potential-time dependences of RPWE (1-6),
RE (1'-6 '), RPSUE (1 "-3")
in 0.1N solutions of electrolytes
1-1" – H_2SO_4;
2-2" – KSCN;
3-3" – KCl;
4, 4' – HCl;
5, 5' – H_3PO_4;
6, 6 ' – $H_2SO_4 + 1.68\cdot10^{-2}$ N $HgSO_4$

In this connection, for the identification of the peculiarities of the mercury-film electrodes on the basis of tungsten (RPWE), the stationary potentials (E_c) of the mercury-film samples based on W have been compared with the mercury pool and RPSUE in 0.1 N solutions of various electrolytes (figure 1).

As it follows from the obtained results, E_c of the mercury-film samples on the basis of tungsten is close to E_c of the above mercury electrodes. A certain difference in the values of the steady-state potentials seems to be explained by an interaction between mercury and the substrate metal. We guess that in the surface layer of a metal substrate, upon mercury deposition, the formation of tungsten bronzes (Hg_xWO_3) is possible, whereupon a mercury coating is formed.

Upon adding of Hg^{2+} ions to 0.1 NH_2SO_4 solution the recorded potential E_c for all electrodes has the same values. This is due to the fact, that since Hg on all electrodes is present as metal, in the elemental state, its activity is equal to 1 and mercury ions are potential-defining in the indifferent H_2SO_4, solution, containing $1.68 \cdot 10^{-2}$ NHg^{2+}.

The study of the steady-state potentials of the mercury-film samples in different media shows not only the defining role of mercury on their surface, but also an effect of the substrate metal upon the electrochemical characteristics of the electrode. It has been established, that the behavior of the electrode with a tungsten substrate somehow differs from that of the pure mercury electrodes [2, 11, 12].

Alongside with the potentiometric measurements, which have made it possible to study the electrodes without the imposition of external polarization, the cathode-anode processes proceeding on the electrode have been studied and the range of the operating potentials has been established for the evaluation of the effect of the substrate metal upon the functional characteristics of the electrode by way of recording cyclic voltammetric curves.

Voltammograms of pure mercury, represented by the mercury pool (RE) and mercury-film samples of tungsten and platinum in the sulfuric medium, have been taken for comparison (figure 2).

The comparison of the polarization curves shows that in case of RPWE a current horizontal area is narrower than that of a mercury electrode, since the field of potentials of the curve kinetic site is wider.

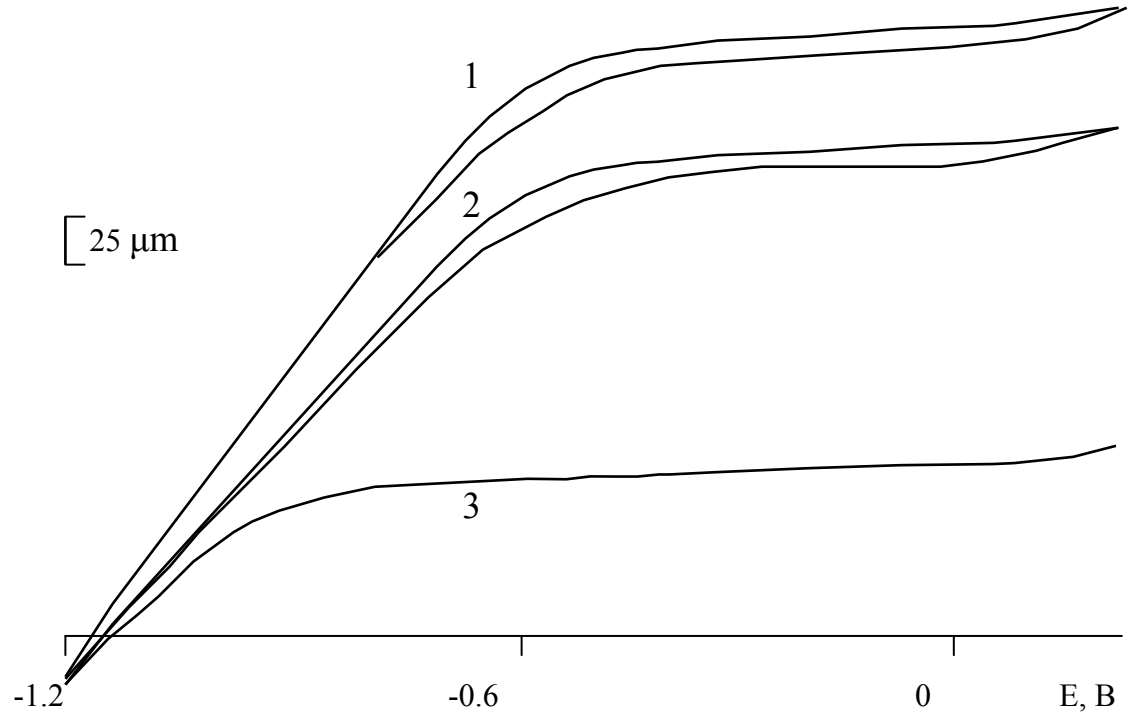

Figure 2 – Comparison of the working region
of the potentials of RPWE (1), RPPRtE (2) and RE (3) in 1M $H2SO_4$

This seems to be explained by an interaction between mercury and tungsten, and results in decreasing of hydrogen overpotential (down to the value of –0.8 V), in comparison with the mercury electrode (≥1B) [1].

Probably, the shift of the reduction potential of H^+-ions to the positive side occurs due to the change of the nature of the surface substrate layer. The horizontal curve area and ranges of operating potentials (figure 2) show an identity of polarization curves for RPWE and RPRtE(curves1, 2) and similarity of the characteristics of these electrodes to RE (curve 3).

Thus, the established similarity of the electrochemical properties of the mercury-film electrodes on a metal substrate (RPWE and RPRtE) affords us ground to conclude that the functional characteristics of the electrodes are mainly determined by the nature of the substrate metal.

A possibility of evaluating the quality of coating has been shown by the method of cyclic voltammetry, and the correctness of such an estimate has been established when using new materials as indicative electrodes in the method of stripping voltammetry. The conditions ensuring selectivity of their determination have been revealed using the example of Pb and Tl.

The regularities of the electrochemical behavior of Pb, Hg, Tl, Cu and Au have been studied in indifferent and complex-forming electrolytes on the new microelectrodes. The new electrodes have been approbated for the first time in the analysis of waters and gold-containing products. Persepectiveness of their using as indicative electrodes for electroanalysis has been demonstrated.

A conclusion has been drawn that one of the criteria for choosing electrode materials is their solubility in mercury and corrosion resistance in the working electrolytes, which, in its turn, is determined by the composition and structure of their crystalline lattice, physical and chemical properties of the oxide films, formed on the sample surface and solid reaction products.

Conclusions. Thus, the established similarity of the electrochemical properties of the mercury-film electrodes on a metal substrate (RPWE and RPRtE) affords us ground to make a conclusion of a possibility to use a mercury-film tungsten indicative microelectrode for exercising control over the trace quantities of metals in the environmental samples**.**

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КОНТРОЛЬ СЛЕДОВЫХ КОЛИЧЕСТВ МЕТАЛЛОВ В ОБЪЕКТАХ ОКРУЖАЮЩЕЙ СРЕДЫ С ИСПОЛЬЗОВАНИЕМ РТУТНО-ПЛЕНОЧНЫХ ИНДИКАТОРНЫХ МИКРОЭЛЕКТРОДОВ

Аннотация. Разработаны теоретические основы определения следовых количеств металлов в объектах окружающей среды методом инверсионной вольтамперометрии, точность и воспроизводимость которых связана с использованием предлагаемого ртутно-пленочного вольфрамового электрода. Разработаны методы формирования устойчивой ртутной пленки на подложке из этого материала; исследованы факторы, влияющие на чувствительность и разрешающую способность пленочного электрода.

Показана перспективность использования электрохимических методов анализа, в частности, метода инверсионной вольтамперометрии, для осуществления контроля токсичных и тяжелых металлов в техногенном и минеральном сырье Казахстана.

Ключевые слова: индикаторный электрод, ртутно-пленочный электрод, вольфрам, инверсионная вольтамперометрия, амальгамирование.

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ҚОРШАҒАН ОРТА ОБЪЕКТІЛЕРІНДЕ СЫНАП-ПЛЕНКАЛЫ ИНДИКАТОРЛАР МИКРОЭЛЕКТРОДТАРДЫ ПАЙДАЛАНА ОТЫРЫП МЕТАЛДАРДЫҢ ӨТЕ АЗМӨЛШЕРІН БАҚЫЛАУ

Аннотация. Инверсті вольтамперометрия тәсілін қолданып қоршаған орта объектілерінде металдардың өте аз мөлшерінің теориялық негіздерін анықтау, жаңадан өндірілуі және дәлдігі ұсынылатын сынап-пленкалы вольфрамды электродты пайдаланумен байланысты. Осы материалдан тұрақты сынап пленкасын қалыптастыру әдістері әзірленді; пленкалы электродтың әсер етуші факторларға сезімталдығы мен рұқсат беретін қабілеті зерттелді.

Электрохимиялық талдау әдістерін пайдалану болашағы көрсетілген, атап айтқанда, вольтамперометрия инверсті әдісін жүзеге асыру үшін ҚР техногенді және минералды шикізаттарда уытты және ауыр металдардың мөлшерін бақылау.

Түйін сөздер: индикаторлық электрод, сынап-пленкалы электрод, вольфрам, инверсті вольтамперометрия, амальгамирлену.

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