

UDC 541.138: 54.061/.062: 543.253: 54-39: 541.459

M. YE. BLAZHEYEVSKIY, O. O. MOZGOVA

National University of Pharmacy, Kharkiv

VOLTAMMETRIC DETERMINATION OF MAGNESIUM MONOPEROXY-PHTHALATE IN PURE SUBSTANCE AND DISINFECTANT “DISMOZON PUR”

The electrochemical behavior of magnesium monoperoxyphthalate (MMPP) has been studied using alternating current voltammetry with square wave modulation in potential range +1.0...-1.2 V at carbosital electrode as indicating (working) and auxiliary electrode (reference electrode Ag, AgCl/KCl(sat)). The peak was obtained at $E_p = +0.15V$ on the background of $0.1 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ and $0.02 \text{ mol L}^{-1} \text{ KHSO}_4$ ($\text{pH} \approx 3$), whose height was rising proportionally to MMPP concentrations increasing. The linear dependence was observed in the MMPP concentration range $(2.42-10.72) \times 10^{-5} \text{ mol L}^{-1}$, calibration curve equation was $I_p = (6.4 \pm 0.2) \times 10^3 \times c$ ($r = 0.998$); $\text{LOD} = 7.26 \times 10^{-6} \text{ mol L}^{-1}$, $\text{LOQ} = 2.42 \times 10^{-5} \text{ mol L}^{-1}$. For determination of MMPP in disinfectant “Dismozon pur” was used the addition method, so RSD were 1.82, 1.61 and 1.48 % ($\delta = -0.40 \dots -0.04 \%$); ϵ were 2.3, 2.0 and 1.8 % for solutions with the MMPP concentration of 4.02×10^{-5} , 5.35×10^{-5} and $6.70 \times 10^{-5} \text{ mol L}^{-1}$ respectively.

Key words: magnesium monoperoxyphthalate, voltammetry, carbosital electrode, disinfectant

STATEMENT OF THE PROBLEM

Magnesium monoperoxyphthalate hexahydrate (MMPP) is organic peroxide belonging to the family of oxygenreleasing agents. It is effective disinfectant and bleaching agent. It is widely used in medicine, agriculture, food and other industries as a disinfectant, antiseptic and sterilizing agent [6, 7, 15, 18].

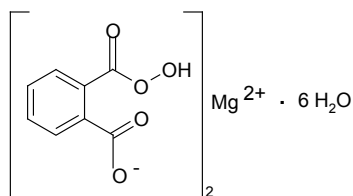


Fig. 1. Formula Magnesium monoperoxyphthalate hexahydrate

In the active substance MMPP the active oxygen component monoperoxyphthalic acid is present in a stable, storable form. The microbiologically active principle of all oxygen donors is the active – nascent – oxygen, which oxidizes all vital cell components of the microorganisms. It has bactericidal, yeasticidal, tuberculocidal, sporicidal, virucidal activity against enveloped viruses (incl. HBV, HIV, HCV), activity.

MMPP is also used as the active ingredient in certain disinfectants such as “Dismozon pur” (BODE Chemie GmbH, Hamburg). Besides the MMPP (80 %) (mass fraction of active oxygen 3.9 %) it comprises excipients: sodium dodecylbenzenesulfonate (SDBS), sodium cumenesulphonate, isotridecanol-ethoxylate and esterificated methylhydroxyethyl cellulose. SDBS acts as a surfactant, thus provides contact with pathogens oxidant; reacts with membrane lipids and proteins causing denaturation of the cell membranes. As a surface disinfectant MMPP exhibits a broad spectrum biocidal effect. Its wide surface compatibility enables its use on sensitive materials, such as plastic and rubber equipment used in hospitals.

“Dismozon pur” is suitable for the disinfectant cleaning of washable surfaces in a wide variety of medical areas and also for food-processing operations and industry. As a result of its microbiological performance and its specific active substance (MMPP) it is recommended for routine use in sensitive areas and areas in the proximity of patients, such as e.g. operating theatres, intensive care units and obstetric units. It is used for comprehensive high-performance disinfection of washable surfaces and equipment in areas that are relevant to hygiene and sensitive to odours, especially for the disinfection of highly sensitive materials. “Dis-

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mozon pur” is also available as sterile version for use in pharmaceutical class A and B cleanrooms.

“Dismozon pur” is supplied as a granulate in 20 g sachets. The sachet permits simple and reliable dosing. It is used as aqueous solutions in the working concentrations of 0.5 to 4 %. The use-solution must be renewed every working day so that the active oxygen level necessary for the product’s full microbiological effectiveness is guaranteed.

ANALYSIS OF RECENT RESEARCH AND PUBLICATIONS

The mass fraction determination of active oxygen in the preparation is carried out by cerimetric and iodometric volumetric analysis [3].

Extensive literature survey reveals that, due to the well-known selectivity and sufficiently high sensitivity, the most common method of the peroxide compounds analysis are a voltammetry methods with variety indicator electrodes [1, 4, 8-14, 16, 17, 19-22]. Along with dropping mercury electrode (DME), platinum and gold electrodes the electrodes based on carbon materials such as glassy carbon, pyrographite and carbosital are widely used.

ALLOCATION OF THE UNSETTLED BEFORE PARTS OF THE COMMON PROBLEM

Carbon electrodes offer new feasibility of the peroxides analytical determinations which virtually impossible carry with mercury electrodes, although it does not have constantly updating surface properties and not so easy to use, because they require a lot of effort for their preparation and service. Carbo-sital is the material from pyrographite, but unlike him, due to its specific structure has isotropic properties, requires no special orientation in the electrode device, has a high electrical conductivity and it is practically non-porous, so that there are small residual current. It has a low resistivity, practically impermeable to gases and resistant to oxidation by atmospheric oxygen. It is less fragile, easily to mechanical polishing [2] and relatively cheap than glassy carbon. A low rates of electrode processes are characteristic of the carbosital electrode (probably due to poor oxygen adsorption capacity). It should be noted that carbosital electrode can successfully replace mercury (negative potential region) and platinum (positive potential area) electrodes in the analysis of peroxide compounds complex mixtures by voltammetry.

FORMULATION OF OBJECTIVES

The aim of the present work is to determine the feasibility of quantitative determination of MMPP in pure substance and disinfectant “Dismozon

pur” by cathodic voltammetry using carbosital electrode as indicating electrode.

THE MAIN MATERIAL OF RESEARCH MATERIALS AND METHODS

The solution of MMPP («Impuls», Poland) was freshly prepared and standardized iodometrically. The stock solution was prepared by dissolving 0.7732 g of the powder (mass fraction of MMPP 55%) in a 100 mL volumetric flask by double distilled water to give the concentration of 1.34×10^{-2} mol L⁻¹. 10 mL of 1.34×10^{-2} mol L⁻¹ solution of MMPP was diluted in a 100 mL volumetric flask with double distilled water to obtain a 1.34×10^{-3} mol L⁻¹ of MMPP solution.

The background solution consists of mixture of potassium hydrogensulfate (KHSO₄) and sodium sulfate (Na₂SO₄) solutions. 68.1 g of KHSO₄ in a 500 mL volumetric flask by double distilled water to give the concentration of 1 mol L⁻¹. 142.0 g of Na₂SO₄ in a 1000 mL volumetric flask by double distilled water to give the concentration of 1 mol L⁻¹.

The sample preparation which was subjected to the analytical procedures for the analysis of MMPP was disinfectant “Dismozon pur” (BODE Chemie GmbH, Hamburg). This solution was prepared by dissolving 1.0 g of preparation (accurate weight) in 100 mL volumetric flask by double distilled water to give a concentration of preparation 1.0 % (concentration of MMPP in mass was 2.7×10^{-2} mol L⁻¹). 10 mL of this solution was diluted in 100 mL volumetric flask with double distilled water to obtain a solution of 2.7×10^{-3} mol L⁻¹ MMPP (control by iodometric method).

The pH was measured using an ionmeter type I-160M (Belarus) with a glass electrode of ESL-43-07 type paired with Ag, AgCl/KCl (sat) electrode.

Electrochemical measurements were carried out in the analyzer AVS-1.1 (Volta, St. Petersburg) with a three-electrode scheme by the alternating current mode with a square wave modulation in potential range +1.0...-1.2 V, W = 1000 rpm, the amplitude 40 mV, $\nu = 65$ Hz. The values of potential peaks directly at the maximum were measured by electrochemical sensor “Module EM-04” with the accuracy of ± 5 mV. The carbosital electrode was used as a working and an auxiliary electrode, and Ag, AgCl/KCl (sat) electrode type EVL-1M4 as a reference electrode.

RESULTS AND DISCUSSION

The effect of nature and pH of background solution

The effect of pH on the reduction process was investigated by recording voltammograms of MMPP at a 2.7×10^{-4} mol L⁻¹ concentration

at several pH values ranging from 1.5 to 6.0 (Fig. 2). A mixture of $0.5 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4 + 0.05 \text{ mol L}^{-1} \text{ KHSO}_4$ was used as background solution and the pH of the solution from 1.5 to 6.0 was changed gradually adding $\text{NaOH } 0.2 \text{ mol L}^{-1}$. The pH was measured using ionmeter type I-160M (Belarus) with a glass electrode type ESL-43-07 paired with Ag,AgCl/KCl (sat) electrode.

As can be seen, height of MMPP reduction peak decreases and potential of reduction peak is shifted toward more electronegative values with increasing of background electrolyte pH from 1.5 to 6.

The maximum peak (I_p) occurs at a pH approximately 3 and at a pH around 6 analytical signal almost disappears (Fig. 3). The effect of pH on peak potential (E_p) shows the following: when pH value increases in the interval from 2 to 4, E_p remains almost constant, but E_p decreases sharply to negative value with pH increasing over 4 (Fig. 4). So, the optimal peak for the analysis ($E_p = +0.15 \text{ V}$) was obtained at $\text{pH} \approx 3$ on the background of $0.1 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ and $0.02 \text{ mol L}^{-1} \text{ KHSO}_4$.

The calibration curves method is used to quantify the concentration of MMPP. The study was conducted in solutions with a concentration of MMPP from 2.42×10^{-5} to $10.72 \times 10^{-5} \text{ mol L}^{-1}$.

The procedure of quantitative determination of MMPP in pure substance

Working solutions were prepared by diluting different volumes of stock solution 1.0, 2.0, 2.5, 3.0 and 4.0 mL of the stock solution and 5 mL of

$1 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4 + 1 \text{ mL}$ of $1 \text{ mol L}^{-1} \text{ KHSO}_4$ each in 50 mL volumetric flask by double distilled water. 25 mL of the working solution of pure substance was transferred to the cell. The voltammograms were recorded by scanning the potential toward the negative direction in the potential range from +1.0 V to -1.2V. All data were obtained at room temperature.

The peak was obtained at $E_p = +0.15 \text{ V}$ on the background of $0.1 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ and $0.02 \text{ mol L}^{-1} \text{ KHSO}_4$ ($\text{pH} \approx 3$), whose height was rising proportionally to MMPP concentrations increasing. The graph was plotted in the following coordinates: the height of peaks I_p in μA at $E_p = +0.15 \text{ V}$ on the ordinate axis and corresponding concentration of MMPP c in mol L^{-1} on the abscissa axis (Fig. 5). The calibration curve equation was $I_p = (6.4 \pm 0.2) \times 10^3 \times c$ ($r = 0.998$); $\text{LOD} = 7.26 \times 10^{-6} \text{ mol L}^{-1}$, $\text{LOQ} = 2.42 \times 10^{-5} \text{ mol L}^{-1}$. The obtained results are summarized in Table 1.

The reproducibility was evaluated from 5 repeated electrochemical signal measurements of model solutions with 5.36×10^{-5} , 6.70×10^{-5} and $8.04 \times 10^{-5} \text{ mol L}^{-1}$ concentrations of MMPP. The precision of the developed method in terms of the relative standard deviation (RSD) were 2.91, 2.88 and 2.21 %, respectively ($n = 5$, $P = 0.95$). The obtained results were agreement with those obtained by using the reference method of iodometric titration, with an accuracy $\delta = -0.45 \dots +0.78 \%$. The obtained results are summarized in Table 2.

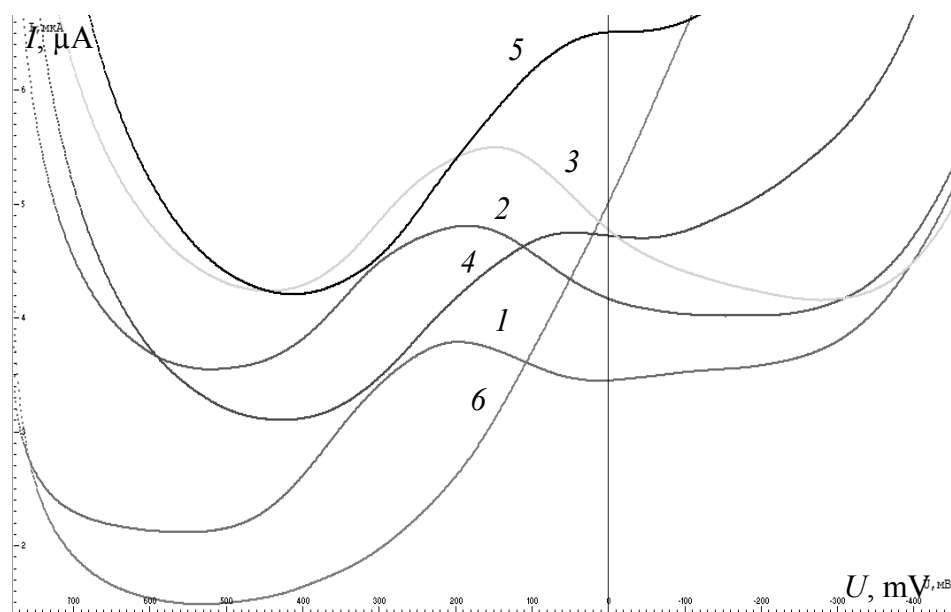


Fig. 2. Voltammogram of MMPP reduction at a concentration of $2.7 \times 10^{-4} \text{ mol L}^{-1}$ at different pH values of the background solution: 1 – 1.5, 2 – 2.0, 3 – 2.9, 4 – 3.5, 5 – 4.0, 6 – 6.0

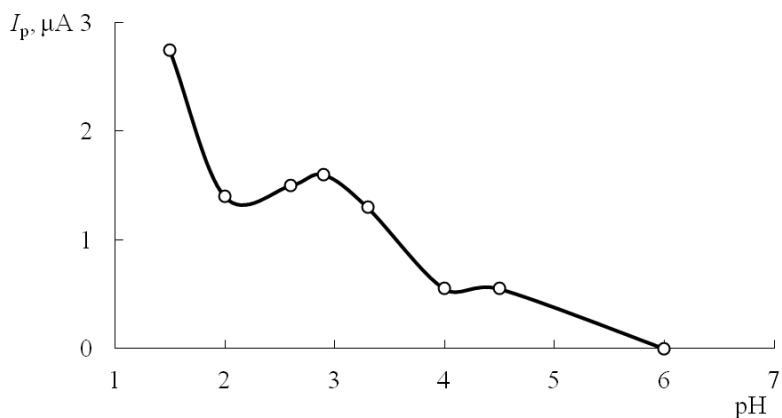


Fig. 3. Influence of pH on the peak intensity of the reduction process of MMPP at the carboxitall electrode (reference electrode Ag,AgCl/KCl(sat))

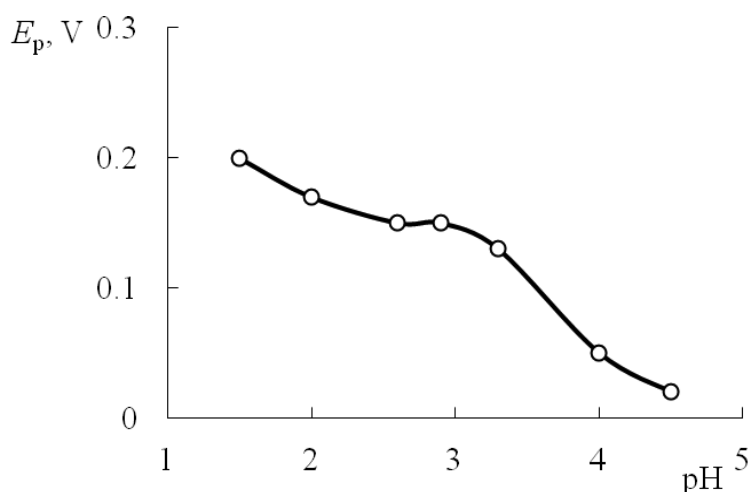


Fig. 4. Influence of pH on the peak potential of the reduction process of MMPP at the carboxitall electrode (reference electrode Ag,AgCl/KCl(sat))

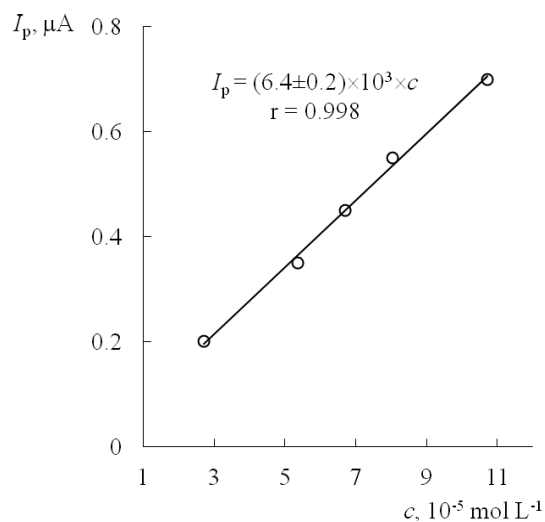


Fig. 5. The calibration graph of the MMPP reduction current peak vs. concentration on the background of $0.1 \text{ mol L}^{-1} \text{ Na}_2\text{SO}_4$ and $0.02 \text{ mol L}^{-1} \text{ KHSO}_4$ ($\text{pH} \approx 3$) at carboxitall electrode (reference electrode Ag,AgCl/KCl(sat)); $E_p = 0.15 \text{ V}$

Table 1

ANALYTICAL CHARACTERISTICS OF THE CALIBRATION GRAPH OF MMPP VOLTAMMETRIC DETERMINATION PROCEDURE IN PURE SUBSTANCE

Parameters	Data
Concentration ranges (mol L ⁻¹)	(2.42-10.72)×10 ⁻⁵
Regression equation	$I_p = (6.4 \pm 0.2) \times 10^3 \times c$
<i>a</i>	6.4×10 ³
<i>b</i>	0.023
Δa	0.7×10 ³
Δb	0.05
<i>S_a</i>	0.2×10 ³
<i>S_b</i>	0,02
Correlation coefficient (<i>r</i>)	0.998
LOD (mol L ⁻¹)	7.26·10 ⁻⁶
LOQ (mol L ⁻¹)	2.42·10 ⁻⁵

Table 2

EVALUATION OF ACCURACY AND PRECISION OF MMPP VOLTAMMETRIC DETERMINATION PROCEDURE IN MODEL SOLUTION OF PURE SUBSTANCE (*N* = 5; *P* = 0.95%)

Taken, mol L ⁻¹	Found, mol L ⁻¹	Recovery, %±SD	RSD, %	ε , %	δ^* , %
5.36×10 ⁻⁵	(5.40±0.19)×10 ⁻⁵	100.78±3.65	2.91	3.62	+0.78
6.70×10 ⁻⁵	(6.67±0.24)×10 ⁻⁵	99.55±3.57	2.88	3.58	-0.44
8.04×10 ⁻⁵	(8.04±0.22)×10 ⁻⁵	99.98±2.32	2.22	2.75	-0.25

* Relative to the average reference method of iodometric titration

The procedure of quantitative determination of MMPP in "Dismozon pur"

It was found that surface active substances (SAS), which is the part of the test solution of sample preparation has a catalytic effect (current increases). The current increases, probably due to relief desorption of reduction products from the electrode surface, and the acceleration of electron transfer in the course of electrochemical reactions is caused by the ability of SAS to adsorb on hydrophobias surface of electrode and to form surface film that changes the overpressure [5, 23]. So it was decided to use the addition method for analysis of the preparation.

Working solutions were prepared by diluting different volumes of stock solution of the sample preparation 1.5, 2.0 and 2.5 mL with 5 mL of 1 mol L⁻¹ Na₂SO₄ + 1mL of 1 mol L⁻¹ KHSO₄ each in 50 mL volumetric flask by double distilled water. A typical procedure involves preparing several solutions containing the same amount of unknown, but different amounts of standard. For example, three 50 mL volumetric flasks are each filled with 1.5 mL of the unknown and then the standard is added in differing amounts, such as 0.1 and 2 mL. The flasks are then diluted to the mark and mixed well. 25 mL of each prepared solutions were transferred to the cell. The voltammograms were recorded by scanning the potential toward the negative direction in the potential range from

+1.0 V to -1.2 V. All data were obtained at room temperature.

At first voltammogram of test solution was recorded and then a solution of known aliquots of standard solution C_{st} was added and again voltammogram was recorded. The concentration of the test solution C_x is given by the equation:

$$C_x = C_{st} \cdot \frac{I_x}{I_{x+st} - I_x},$$

where I_x – current peak of test solution; I_{x+st} – current peak of test solution with the addition of a standard substance.

The high sensitivity of this method is accompanied by a very good reproducibility. The reproducibility was evaluated from 5 repeated electrochemical signal measurements of model solutions with the MMPP concentrations 4.02×10⁻⁵, 5.35×10⁻⁵ and 6.70×10⁻⁵ mol L⁻¹. The precision of the method developed in terms of the relative standard deviation (RSD) were 1.82, 1.61 and 1.48 % ($\delta = -0.40...-0.04\%$) respectively. The results obtained are summarized in Table 3.

CONCLUSIONS AND PROSPECTS FOR FURTHER RESEARCH

Thus, a new voltammetric method of aqueous solutions of magnesium monoperoxyphthalate determination in pure substance and disinfectant "Dismozon pur" using carbosital electrode as

Table 3

**THE RESULTS OF VOLTAMMETRIC DETERMINATION OF MAGNESIUM
MONOPEROXYPHthalate IN "DISMOZON PUR" (N = 5; P = 0.95%)**

Taken, mol L ⁻¹	Found, mol L ⁻¹	Recovery, %±SD	RSD, %	ε, %	δ*, %
4.02×10 ⁻⁵	(4.00±0.09)× 10 ⁻⁵	99.60±2.26	1.82	2.3	-0.40
5.35×10 ⁻⁵	(5.35±0.11)× 10 ⁻⁵	99.96±1.99	1.61	2.0	-0.04
6.70×10 ⁻⁵	(6.68±0.12)× 10 ⁻⁵	99.73±1.84	1.48	1.8	-0.26

* Relative to the average reference method of iodometric titration [3]

indicating electrode has been developed and the possibility of its quantitative determination has been shown.

The linear dependence is observed in the concentration ranges of pure substance varies from 2.42×10⁻⁵ to 10.72×10⁻⁵ mol L⁻¹. The calibration curve equation is $I_p = (6.4 \pm 0.2) \times 10^3 \times c$ ($r = 0.998$); LOD = 7.26×10⁻⁶ mol L⁻¹, LOQ = 2.42×10⁻⁵ mol L⁻¹; RSD = 2.21...2.91 %; ε = 2.75 ...3.62 %; δ = -0.44...+0.78 % for the electrochemical signal of 5.36×10⁻⁵, 6.70×10⁻⁵ and 8.04×10⁻⁵ mol L⁻¹ MMPP. For determination of MMPP in disinfectant "Dismozon pur" was used the addition method, so RSD were 1.82, 1.61 and 1.48 % (δ = -0.40...-0.04%); ε were 2.3, 2.0 and 1.8 % for solutions with the MMPP concentration of 4.02×10⁻⁵, 5.35×10⁻⁵ and 6.70×10⁻⁵ mol L⁻¹ respectively.

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УДК 541.138: 54.061/.062: 543.253: 54-39: 541.459

Н. Е. Блажеевский, Е. А. Мозговая

ВОЛЬТАМПЕРОМЕТРИЧЕСКОЕ ОПРЕДЕЛЕНИЕ МОНОПЕРОКСИ-ФТАЛАТА МАГНИЯ В СУБСТАНЦИИ И ДЕЗИНФЕКЦИОННОМ СРЕДСТВЕ «ДИСМОЗОН ПУР»

Методом катодной вольтаперометрии с использованием как индикаторного углесталлового электрода изучено электрохимическое поведение монопероксифталата магния (ММПФ) в интервале потенциалов $E = +1,0... -1,2$ В (отн. нас. Ag,AgCl/KCl). Пик (I_p) был получен при $E_p = +0,15$ В на фоне 0,1 моль/л Na_2SO_4 и 0,02 моль/л $KHSO_4$ (рН \approx 3), высота которого увеличивается с ростом концентрации ММПФ. Линейная зависимость наблюдалась в диапазоне концентраций $(2,42-10,72) \cdot 10^{-5}$ моль/л, уравнение градуировочного графика: $I_p = (6,4 \pm 0,2) \cdot 10^3 c$ ($r = 0,998$); LOD = $7,26 \cdot 10^{-6}$ моль/л, LOQ = $2,42 \cdot 10^{-5}$ моль/л. Для определения ММПФ в растворах дезинфицирующего средства использовали метод добавок. При вольтамперометрических измерениях модельных растворов дезинфицирующего средства «Дисмозон пур» с концентрацией ММПФ $4,02 \cdot 10^{-5}$, $5,35 \cdot 10^{-5}$ и $6,70 \cdot 10^{-5}$ моль/л RSD составило 1,82, 1,61 и 1,48 % соответственно; $\delta = -0,40...-0,04$ % (относительно среднего референс-метода йодометрического титрования).

Ключевые слова: монопероксифталат магния, вольтамперометрия; углесталловый электрод, дезинфицирующее средство.

УДК 541.138: 54.061/.062: 543.253: 54-39: 541.459

М. Є. Блажеєвський, О. О. Мозгова

ВОЛЬТАМПЕРОМЕТРИЧНЕ ВИЗНАЧЕННЯ МАГНІЙ МОНОПЕРОКСИ-ФТАЛАТУ У СУБСТАНЦІЇ ТА ДЕЗИНФЕКЦІЙНОМУ ЗАСОБІ «ДИСМОЗОН ПУР»

Методом катодної вольтаперометрії з використанням як індикаторного вуглесталлового електрода вивчено електрохімічну поведінку магнію монопероксифталату (ММПФ) в інтервалі потенціалів $E = +1,0... -1,2$ В (відн. нас. Ag,AgCl/KCl). Пік (I_p) був отриманий при $E_p = +0,15$ В на фоні 0,1 моль/л Na_2SO_4 та 0,02 моль/л $KHSO_4$ (рН \approx 3), висота якого зростає зі збільшенням концентрації ММПФ. Лінійна залежність спостерігалася у діапазоні концентрацій $(2,42-10,72) \cdot 10^{-5}$ моль/л, рівняння градуировального графіка: $I_p = (6,4 \pm 0,2) \cdot 10^3 c$ ($r = 0,998$); LOD = $7,26 \cdot 10^{-6}$ моль/л, LOQ = $2,42 \cdot 10^{-5}$ моль/л. Для визначення ММПФ у розчинах дезінфекційного засобу використовували метод добавок. При вольтамперометричних вимірюваннях модельних розчинів засобу «Дисмозон пур» с концентрацією ММПФ $4,02 \cdot 10^{-5}$, $5,35 \cdot 10^{-5}$ та $6,70 \cdot 10^{-5}$ моль/л RSD дорівнювало 1,82, 1,61 та 1,48 % відповідно; $\delta = -0,40...-0,04$ % (відносно середнього референс-метода йодометричного титрування).

Ключові слова: монопероксифталат магнію, вольтамперометрія; вуглесталловий електрод, дезінфекційний засіб.

Адреса для листування:

61118, м. Харків, вул. Блюхера, 4,

Кафедра фізичної та колоїдної хімії НФаУ

E-mail: helio_helen@rambler.ru

Тел. (0572) 67-98-38

Надійшла до редакції:

01.05.2015 р.