

Информационно-коммуникационные и химические технологии  
SRSTI 53.37.91

## VOLTAMMETRIC STUDY OF THE CATHODIC BEHAVIOR OF COPPER, NICKEL AND ZINC IONS IN AMMONIA SOLUTIONS

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The article presents the results of the study of the electrochemical behavior of  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$  by voltammetry on stationary solid electrodes. Polarizable working electrodes (copper, titanium) were used for measurements. The value of the half-wave potentials on the titanium electrode shifts to the negative region with an increase in the concentration of ions in the solution, while on the copper one it remains at the same level. The addition of glycine shifts the half-wave potential of both copper and nickel, zinc to the negative region (by 0,07 V for copper, 0,11 V for nickel, 0,12 V for zinc). In the presence of citric acid, the half-wave potential of copper also shifts to a more negative region, while the reduction potentials of copper, nickel and zinc converge, which contributes to their joint electrodeposition.

**Keywords:** copper electrolyte, copper, nickel, zinc, voltammetry, electrodeposition.

## АММИАК ЕРІТІНДІЛЕРІНДЕГІ МЫС, НИКЕЛЬ ЖӘНЕ МЫРЫ ИОНДАРЫНЫҢ КАТОДТЫҚ ӘРЕКЕТІН ВОЛЬТАМПЕРМЕТРИАЛЫҚ ЗЕРТТЕУ

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Мақалада стационарлық қатты электродтардағы  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$  электрохимиялық әрекетін вольтамперметрия арқылы зерттеу нәтижелері берілген. Өлшеу үшін поляризацияланатын жұмыс электродтары (мыс, титан) пайдаланылды. Титан электродындағы жартылай толқындық потенциалдардың мәні ерітіндідегі иондар концентрациясының жоғарылауымен теріс аймаққа ауысады, ал мыс электродында ол сол деңгейде қалады. Глициннің қосылуы мыстың да, никельдің де, мырыштың да жарты толқындық потенциалын теріс аймаққа жылжытады (мыс үшін 0,07 В, никель - 0,11 В, мырыш - 0,12 В). Лимон қышқылының қатысуымен мыстың жартылай толқындық потенциалы да теріс аймаққа ауысады, ал мыс, никель және мырыштың тотықсыздану потенциалдары жақындасады, бұл олардың бірлескен электротұнбалауына ықпал етеді.

**Түйін сөздер:** мыс электролиті, мыс, никель, мырыш, вольтамперметрия, электротұнбалау.

## ВОЛЬТАМПЕРОМЕТРИЧЕСКОЕ ИССЛЕДОВАНИЕ КАТОДНОГО ПОВЕДЕНИЯ ИОНОВ МЕДИ, НИКЕЛЯ И ЦИНКА В АММИАЧНЫХ РАСТВОРАХ

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В статье представлены результаты исследования электрохимического поведения  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$  методом вольтамперометрии на стационарных твердых электродах. Для измерений использовались поляризуемые рабочие электроды (медный, титановый). Значение потенциалов полуволн на титановом электроде с ростом концентрации ионов в растворе сдвигается в отрицательную область, тогда как на медном остается на одном уровне. Добавление глицина сдвигает потенциал полуволны как меди, так и никеля, цинка в отрицательную область (на 0,07 В у меди, 0,11 В - никеля, 0,12 В - цинка). В присутствии лимонной кислоты потенциал полуволны меди также сдвигается в более отрицательную область, при этом потенциалы восстановления меди, никеля и цинка сближаются, что способствует их совместному электроосаждению.

**Ключевые слова:** медный электролит, медь, никель, цинк, вольтамперометрия, электроосаждение.

**Introduction.** The processing of solutions derived from the electrolytic refining cycle of copper is important both from the point of view of environmental protection, but also in terms of extracting various valuable components (copper, nickel, zinc) from them into marketable products. The existing technologies in this area do not meet modern requirements either in terms of environmental or economic indicators, because they are characterized by bulkiness and low efficiency [1-3]. In this regard, research aimed at developing an electromembrane technology for processing copper electrolyte to obtain a triple alloy (Cu-Ni-Zn) -- nickel silver is relevant.

For the development of electromembrane technologies [4-10], information is needed on the behavior of ammonia complexes of metals (copper, nickel, zinc) in membrane systems. In the literature, such data are presented extremely concisely. Therefore, this work is devoted to the study of voltammetric parameters of electrolysis [11-14] on the process of joint cathodic deposition of copper, nickel and zinc. If there are a number of interesting works on the production of copper-nickel-based double alloys [15-17], then there are practically no data on the production of triple alloys by electrolysis.

**Materials and methods.** To clarify the mechanism of electrode reactions in the  $\text{Cu}^{2+}$ - $\text{Ni}^{2+}$ - $\text{Zn}^{2+}$  system, voltammetry on solid electrodes was used. The removal of polarograms was performed on a potentiostat-galvanostat (model M273 (USA)).

Polarizable working electrodes (copper, titanium), auxiliary (graphite) and non-polarizable reference electrode (silver chloride) were used for measurements. The area of the copper and titanium working electrode is 0.070  $\text{cm}^2$ . Registration of polarograms began with an equilibrium (-0.4 V-0.46 V). Polarizable voltage up to 2 V. The scanning speed is 10 mV/sec.

The studies were conducted against a background of 1M  $\text{NH}_4\text{OH}$  at the following salt concentrations (in mol/L):  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  ( $10^{-1}$ ,  $10^{-2}$ ,  $10^{-3}$ ,  $10^{-4}$ );  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$  ( $10^{-1}$ ,  $10^{-2}$ ,  $10^{-3}$ ,  $10^{-4}$ );  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  ( $10^{-1}$ ,  $10^{-2}$ ,  $10^{-3}$ ,  $10^{-4}$ ) with citric acid additives in g-eq/l: 0,01; 0,025; 0,05; 0,1 and glycine in g-eq/l: 0,01; 0,025; 0,05; 0,1.

**Results and discussion.** The standard potential of the Cu/ $\text{Cu}^{2+}$  system is a significantly positive value compared to the standard potential of the Ni/ $\text{Ni}^{2+}$  and Zn/ $\text{Zn}^{2+}$  systems:

$$\hat{A}_{\text{Cu}/\text{Cu}^{2+}} = 0.34\hat{A};$$

$$\hat{A}_{\text{Ni}/\text{Ni}^{2+}} = -0.25\hat{A};$$

$$\hat{A}_{\text{Zn}/\text{Zn}^{2+}} = -0.76\hat{A};$$

As a result, in non-complexing media at copper reduction potentials, the discharge of nickel and zinc at the cathode does not occur. This is the basis, for example, for the process of electrolytic refining of copper, where nickel and zinc, without being released at the cathode, accumulate in sulfuric acid solutions. If complexing reagents are introduced into the system - ammonia, citric acid, glycine, sufficiently stable complexes with  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$  ions are formed in solution. Data on the stability of these compounds are given in Table 1.

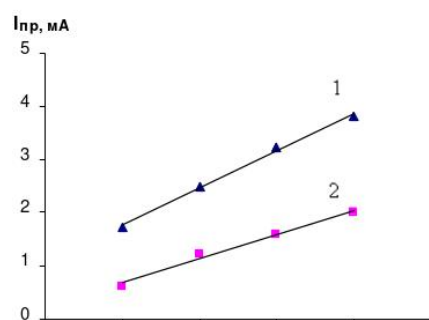
The formation of ammonia complexes leads to a shift in the reduction potential of copper to a more negative region, and for copper this shift was significant. The value of the reduction potential of copper still remained positive than the potential for the release of nickel and zinc. The recovery potential of nickel and zinc shifts to the positive region.

Table 1. Stability of copper, nickel and zinc complexes

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Metal ion	lg K <sub>1</sub>	lg K <sub>1,2</sub>	lg K <sub>1,2,3</sub>	lg K <sub>1,2,3,4</sub>	lg K <sub>1,2,3,4,5</sub>	lg K <sub>1,2,3,4,5,6</sub>
Ammonia complexes						
Cu <sup>2+</sup>	3,99	7,33	10,06	12,03	11,43	8,9
Ni <sup>2+</sup>	2,69	4,79	6,40	7,47	8,10	8,01
Zn <sup>2+</sup>	2,18	4,43	6,74	8,70	-	-
Citrate complexes						
[(CH <sub>2</sub> ) <sub>2</sub> C(OH)(COO) <sub>3</sub> ] <sup>3-</sup>						
Cu <sup>2+</sup>	5,90	-	-	-	-	-
Ni <sup>2+</sup>	5,40	-	-	-	-	-
[(CH <sub>2</sub> ) <sub>2</sub> C(OH)(COOH)(COO) <sub>2</sub> ] <sup>2-</sup>						
Cu <sup>2+</sup>	3,42	-	-	-	-	-
Ni <sup>2+</sup>	3,30	-	-	-	-	-
[(CH <sub>2</sub> ) <sub>2</sub> C(OH)(COOH) <sub>2</sub> COO] <sup>-</sup>						
Cu <sup>2+</sup>	2,26	-	-	-	-	-
Ni <sup>2+</sup>	1,75	-	-	-	-	-

These data were obtained for both copper and titanium stationary electrodes. The polarization curves are shown in Figures 1-4. The magnitude of the limiting current increased with an increase in the concentration of metal ions. It should be noted that the limiting currents on the titanium electrode were 5-10 times higher than the corresponding values obtained on the copper electrode.




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0,0001 0,001 0,01 0,1  $C_{Cu^{2+}, Ni^{2+}}$

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Figure 1 - Dependence of the limiting reduction current of  $Cu^{2+}$ (1) and  $Ni^{2+}$ (2) on a copper electrode on the concentration of ions in solution. Ion concentrations, mol/l: 0,0001; 0,001; 0,01; 0,1

The reduction of copper, nickel and zinc on a titanium electrode occurs in a more negative potential range compared to copper (Table 2), although the nature of the electrode does not significantly affect the shape of the polarograms. The value of the half-wave potential on a titanium electrode shifts to a less negative region with an increase in the concentration of ions in the solution, whereas on a copper electrode it remains at the same level. So, for example, when the copper concentration changes from  $10^{-4}$  to  $10^{-1}$  M on a titanium electrode, the half-wave potential changes from -0,68 V to -0,54 V, on a copper electrode it remains at -0,54 V to -0,52 V.

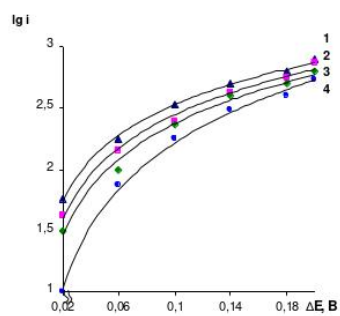


Figure 2 - Dependence of lg I on the  $\Delta E$ -reduction process of  $\text{Cu}^{2+}$  concentration of  $10^{-1}$  mol/l on a titanium electrode with the addition of citric acid, g-eq/l: 1-0,01; 2-0,025; 3-0,05; 4-0,1

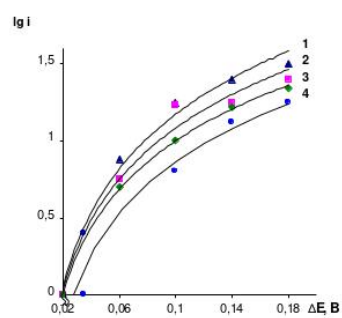


Figure 3. Dependence of  $\lg I$  on the  $\Delta E$ -reduction process of  $Ni^{2+}$  concentration of  $10^{-1}$  mol/l on a copper electrode with the addition of citric acid, g-eq/l: 1-0,01; 2-0,025; 3-0,05; 4-0,1

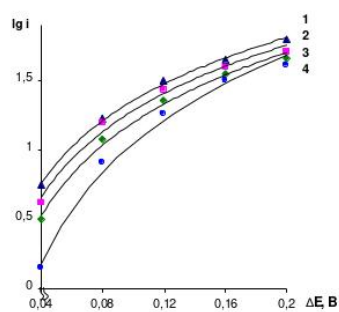


Figure 4 - Dependence of  $\lg I$  on the  $\Delta E$ -reduction process of  $Zn^{2+}$  concentration of  $10^{-1}$  mol/l on a copper electrode with the addition of citric acid, g-eq/l: 1-0,01; 2-0,025; 3-0,05; 4-0,1

Table 2 - The effect of the concentration of ammonia complexes on  $-E_{1/2}$  and  $I_{pr}$

Concentration, mol/l $Cu^{2+}$	$Ni^{2+}$	$Zn^{2+}$	$I_{pr}$ , mA	$-E_{1/2}$ , B	Cathode material
$10^{-1}$			3,9	0,52	
$10^{-2}$			3,15	0,54	
$10^{-3}$			2,4	0,53	Cu
$10^{-4}$			1,7	0,54	
	$10^{-1}$		3,3	0,58	
	$10^{-2}$		2,85	0,60	
	$10^{-3}$		2,3	0,59	Cu

Concentration, mol/l Cu <sup>2+</sup>	Ni <sup>2+</sup>	Zn <sup>2+</sup>	I <sub>pr</sub> , mA	-E <sub>1/2</sub> , B	Cathode material
10 <sup>-1</sup> 10 <sup>-2</sup> 10 <sup>-3</sup> 10 <sup>-4</sup>	10 <sup>-4</sup>	10 <sup>-1</sup>	1,8	0,56	Cu
		10 <sup>-1</sup>	2	0,62	
		10 <sup>-2</sup>	1,5	0,58	
		10 <sup>-3</sup>	1,2	0,58	
		10 <sup>-4</sup>	0,7	0,565	
	10 <sup>-1</sup>	10 <sup>-1</sup>	60	0,54	Ti
		10 <sup>-2</sup>	47	0,60	
		10 <sup>-3</sup>	35	0,63	
		10 <sup>-4</sup>	22	0,66	
		10 <sup>-1</sup>	51	0,62	
	10 <sup>-2</sup>	10 <sup>-1</sup>	40	0,64	Ti
		10 <sup>-2</sup>	40	0,64	
		10 <sup>-3</sup>	29	0,63	
		10 <sup>-4</sup>	18	0,65	
		10 <sup>-1</sup>	44	0,68	
	10 <sup>-3</sup>	10 <sup>-1</sup>	34	0,67	Ti
		10 <sup>-2</sup>	34	0,67	
		10 <sup>-3</sup>	25	0,64	
		10 <sup>-4</sup>	15	0,635	
		10 <sup>-1</sup>	15	0,635	

Citric acid shifts the copper half-wave potential to a more negative area. If, in the absence of citric acid, the difference in the half-wave potentials of copper and nickel is (on a titanium electrode) -0,08 V, copper and zinc -- 0,14 V, then in the presence of citric acid in the solution, the difference in the half-wave potentials of copper and nickel is 0,015 V, copper and zinc -0,005 V. In this case, the reduction potentials of copper, nickel and zinc converge. This promotes the joint electrodeposition of Cu, Ni and Zn. Table 3 shows the data on the effect of citrate ions on the reduction potentials and the magnitude of the limiting current.

Table 3 -The effect of citric acid additions on -E<sub>1/2</sub> and I<sub>pr</sub>

Concentration, mol/l						
Cu <sup>2+</sup>	Ni <sup>2+</sup>	Zn <sup>2+</sup>	C <sub>citric acid</sub>	I <sub>pr</sub> , mA	-E <sub>1/2</sub> , B	Cathode material
10 <sup>-1</sup>	10 <sup>-1</sup>	10 <sup>-1</sup>	0	3,9	0,52	Cu
			0,01	3,5	0,54	
			0,025	3,2	0,53	
			0,05	3,4	0,54	
			0,1	3,9		
			0	3,3	0,58	Cu
			0,01	2,6	0,60	
			0,025	2,1	0,59	
			0,05	1,6	0,56	
			0,1	1,5		
			0	2	0,62	Cu
			0,01	1,7	0,58	
			0,025	1,5	0,58	
			0,05	1,6	0,565	
			0,1	1,3		
10 <sup>-1</sup>	10 <sup>-1</sup>	10 <sup>-1</sup>	0	60	0,54	Ti
			0,01	58	0,60	
			0,025	56	0,63	
			0,05	50	0,66	
			0,1	40		



# 0.1. VOLTAMMETRIC STUDY OF THE CATHODIC BEHAVIOR OF COPPER, NICKEL AND ZINC IONS IN AMMONIA SOLUTIONS

Concentration, mol/l Cu <sup>2+</sup> Ni <sup>2+</sup>	Zn <sup>2+</sup>	C <sub>citric acid</sub>	I <sub>pr</sub> , mA	-E <sub>1/2</sub> , B	Cathode material
10 <sup>-1</sup>		0	51	0,62	Ti
		0,01	48	0,64	
		0,025	41	0,63	
		0,05	41	0,65	
		0,1	36,6		
	10 <sup>-1</sup>	0	44	0,68	Ti
		0,01	41	0,67	
		0,025	38	0,64	
		0,05	37	0,635	
		0,1	32		

The value of the limiting current decreased with an increase in the concentration of the citric acid additive, especially when using a titanium electrode. Glycine additives shift the half-wave potential of both copper, nickel and zinc to a less negative region (by 0,078 V for copper, 0,11 V for nickel, 0,12 V for zinc). But a significant gap between them remains. The presence of glycine also led to a significant change in the value of the limiting current at the electrodes (Table 4).

Changes in the voltammetric behavior of metals in the presence of citric and aminoacetic (glycine) acids are associated with the noted processes of complexation, the transition of metals from one complex form to another.

Table 4 - The effect of glycine additives on -E<sub>1/2</sub> and I<sub>pr</sub>

Concentration, mol/l Cu <sup>2+</sup> Ni <sup>2+</sup>	Zn <sup>2+</sup>	Glycine, г-экв/л	I <sub>пр</sub> , mA	-E <sub>1/2</sub> , B	Cathode material
10 <sup>-1</sup>		0	3,9	0,52	Cu
		0,01	3,4	0,54	
		0,025	3,1	0,53	
		0,05	3,3	0,54	
		0,1	3,1		
	10 <sup>-1</sup>	0	3,3	0,58	Cu
		0,01	2,9	0,60	
		0,025	2,5	0,59	
		0,05	2,6	0,56	
		0,1	2,65		
	10 <sup>-1</sup>	0	2	0,62	Cu
		0,01	1,7	0,54	
		0,025	1,6	0,52	
		0,05	1,8	0,525	
		0,1	1,5	0,50	

**Conclusions.** Thus, if complexing reagents - ammonia, citric acid, glycine - are introduced into the system, sufficiently stable complexes with Cu<sup>2+</sup>, Ni<sup>2+</sup>, Zn<sup>2+</sup> ions are formed in the solution. Glycine makes the system more stable, which is associated with the formation of homogeneous complexes. Citric acid reduces the limiting current, but the equilibrium character remains unchanged. At the same time, the reduction potentials of copper, nickel and zinc converge, which contributes to their joint electrodeposition. The results of the research can be used to solve problems in the field of processing solutions derived from the cycle of electrolytic refining of copper.

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