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VOLTAMMETRIC DETERMINATION OF NI(II) USING 1-[(5-(3-NITROBENZYL)-1,3-THIAZOL-2-YL)DIAZENYL]NAPHTHALENE-2-OL

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A new reagent, 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazenyl]naphthalen-2-ol, which is a derivative of 1-[(5-benzyl-1,3-thiazol-2-yl)diazenyl]naphthalene-2-ol, was proposed for the voltammetric determination of nickel(II). The voltammetric characteristics of aqueous-ethanolic solutions (50% v/v) of NBnTAN and this reagent in the presence of nickel(II) ions were investigated. Optimal conditions for the determination of nickel(II) using 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazenyl]naphthalen-2-ol on the background of sodium chloride at pH 7.0 ($C_{\min}=1,3 \times 10^{-7}$ M) were determined. The selectivity of this technique with respect to related metal ions was investigated. Approbation was performed during the analysis of an aluminum-based alloy (A 1891).

Keywords: 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazenyl]naphthalen-2-ol, thiazolylase dyes, voltammetry, nickel (II).

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1. Introduction

Nickel is widely used in various industries, including the production of Ni–Cd batteries, galvanization, pigmentation of paints, ceramics, medicine (surgical and dental prostheses), magnetic tapes, jewelry industry, as a catalyst, stainless steel kitchen utensils, and more. Compared to other transition metals, nickel is moderately toxic, but still at low concentrations has a toxic effect on the human body and can cause diseases of the nasopharynx and lungs, allergic contact dermatitis, the formation of malignant tumors [1–3]. Therefore, it is necessary to control the content of this metal in the environment using methods of rapid and reliable determination of Nickel in a wide concentration range. Most of the developed methods for the determination of Nickel are non-selective and often require the use of masking agents.

The main task of modern analytical chemistry is the new compounds-complexes introduction into the analysis as reagents, which are synthesized by targeted modulation, which will allow to develop sensitive and highly selective methods for the elements determination. One such compound is thiazolylase dyes, which are widely used in spectrophotometric analysis due to their high sensitivity and selectivity [4]. 1-(2-Thiazoliazol)-2-naphthol is used for atomic absorption determination of Ni(II) and Co(II) in water samples after extraction at the cloud point [5]. Also, this reagent forms

chelates with ions of transition metals, including Ni(II) ions, on the basis of which a method of flow-injection determination was developed [6]. In particular, our group developed methods for the determination of ions Co(II), Ni(II), Cu(II), Cd(II), Hg(II) with 1-(5-benzylthiazol-2-yl)azonaphthalen-2-ol (BnTAN), as well as Pd(II) ions with its methoxy and nitro derivatives [7–15]. These studies led to the study of the interaction of Ni(II) ions with 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazanyl]naphthalen-2-ol (NBnTAN), which was obtained by the introduction of nitro groups in the compound BnTAN, in order to develop a voltammetric method for the determination of Nickel.

2. Materials and methods of the experiment

Voltammetric measurements were performed on a computerized installation MTEch OVA-410 [16] with a triangular shape of the polarization voltage in the three-electrode thermostat cell (indicator electrode - dropping mercury electrode (DME), the reference electrode – saturated calomel electrode (SCE), auxiliary electrode – platinum electrode). Characteristics of the capillary in a solution of 0.16 mol/L sodium perchlorate without voltage: $m=0.706$ mg/s, $\tau=7.5$ s. Analytical studies were performed at room temperature (~ 20 °C) at a polarization voltage of 1.0 V/s. Dissolved oxygen was removed from the investigated solutions by bubbling the purified argon for 10 min.

The acidity of the medium was measured on an ionometer AI-123 using a combined glass electrode. The desired pH value was created using solutions of hydrochloric acid, sodium hydroxide, acetate and ammonia buffer solutions.

The stock solution of Ni(II) ions and all ions used in the study of selectivity were prepared by dissolving the exact mass of pure metal (99.999 %) in hydrochloric or nitric acid, or mixtures thereof. Working solutions of the metals were prepared by diluting an aliquot of the corresponding starting solutions in distilled water and using HCl or HNO₃ for providing an acidic medium in these solutions.

A stock solution of 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazanyl]naphthalen-2-ol was prepared by dissolving an exact portion of the pre-purified reagent in pure ethanol (96 %). NBnTAN working solutions were prepared by diluting an exact aliquot of the stock solution in ethyl alcohol.

Method of voltammetric determination of Ni(II) by the peak of the complex compound using 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazanyl]naphthalen-2-ol: in a flask of 25.0 mL put an aliquot of the investigated solution containing 0.6 to 17.0 µg of Ni(II), add 2.5 mL of $4.0 \cdot 10^{-4}$ M solution of 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazanyl]naphthalen-2-ol, 2 mL of 2.0 M sodium chloride solution, 12.5 mL of ethyl alcohol and distilled water to ~ 20.0 mL. A pH value of 7.0 is set on the pH meter or ionometer using ammonia buffer. Then bring to the mark with distillate and re-adjust the pH. The resulting solution is transferred to a polarographic cell and bubbled with purified argon for 10 min. After that, a voltammogram in the potential range -0.20 – -1.20 V is obtained and the height of the cathode peak is measured. The concentration of Ni(II) is determined by the calibration curve method.

Method of aluminum-based alloy (Al891) sample preparation: samples of alloy (0.1362 g) are placed in a heat-resistant chemical vessel and add 2.0 mL of NaOH solution with a concentration of 10.0 M. After the reaction, the glass is placed on a sand bath and

heated to a complete stop of the alloy dissolution, then cool and dilute the alkaline solution with 20–25 mL of water. Then carefully add 20.0 mL of a mixture of concentrated nitric and hydrochloric acids (1:3) to the cold solution. The precipitate is filtered off, washed with HNO₃ solution (1:1) (4 times 5.0 mL). The obtained filtrate is evaporated in a sand bath to wet salts, 20 mL of HCl solution (1:4) is added to a beaker, transferred to a 50.0 mL volumetric flask and made up to the mark with distilled water. The working solutions were prepared by diluting the stock solution.

3. Results and discussion

The reagent derived from thiazole-1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl) diazenyl] naphthalen-2-ol was used. The structural formula of NBnTAN is shown in Fig. 1. This reagent was synthesized by the staff of the Organic Chemistry Department of Ivan Franko National University of Lviv.

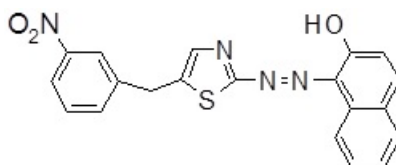


Fig. 1. Structural formula of 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl) diazenyl]naphthalen-2-ol
NBnTAN is a reddish-brown powder, poorly soluble in water but highly soluble in ethanol

Before investigating the possibility of voltammetric determination of Ni(II) using NBnTAN, it was necessary to study the electrochemical properties of this reagent. First, voltammograms of reduction of aqueous-ethanolic (50 % v/v) NBnTAN solutions at different concentrations were obtained (Fig. 2). As can be seen from Fig. 2 this reagent is quite polarographically active. Which indicates the possibility of its use in voltammetric studies. The polarographic current is rectilinearly dependent on the NBnTAN concentration shown in Fig. 3.

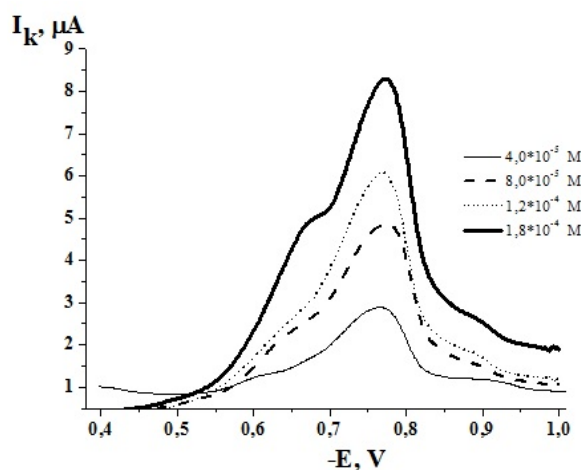


Fig. 2. Voltammograms of aqueous-ethanolic solutions (50 % v/v) NBnTAN at different concentrations of reagent, C(NaCl)=0.16 M, V=1.0 V/s

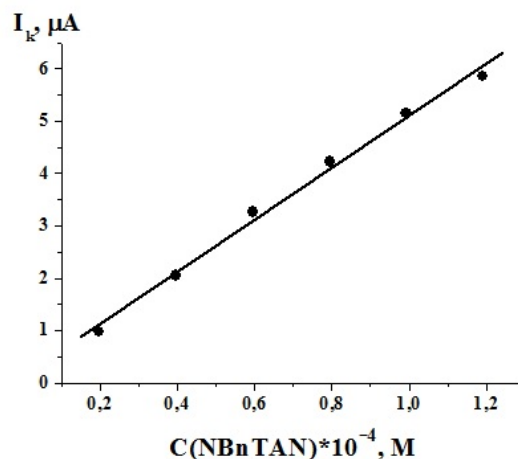


Fig. 3. The dependence of the polarographic current on the concentration of aqueous-ethanolic solutions (50 % v/v) NBnTAN, C (NaCl)=0.16 M, V=1.0 V/s

Voltammetric studies of NBnTAN ($C=4.0 \times 10^{-5}$ M) at different acidity of the medium were also performed and it was found that with increasing acidity of the medium the peak shifts to the cathode region.

The polarographic behavior of the NBnTAN reagent in the presence of Ni(II) ions was studied. The obtained polarograms of complex formation at different pH are shown in Fig. 4.

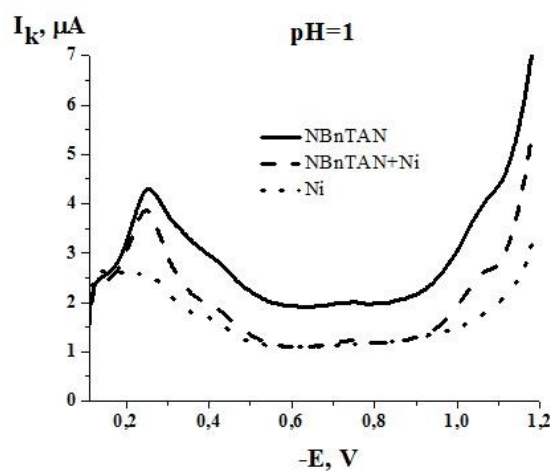


Fig. 4. Voltammograms of aqueous-ethanolic solutions (50% v/v) NBnTAN in the presence of Ni(II) ions at different pH values: $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M, $C(\text{Ni(II)})=1.0 \times 10^{-4}$ M, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s

The formation of the complex is observed in the pH range of 5.0–11.0. A stable complex is formed at pH=7.0, because the first wave of the reagent reduction completely disappears and the reduction potential shifts to a more cathode region. At a potential of -0.640 V, we observe a significant reduction in the peak of the complex ($\Delta I = 1.011$ μA). At other pH values, as can be seen from Fig. 4, there is an increase in the peak of the complex compared to the peak of recovery of the reagent.

The obtained dependences of the magnitude of the recovery peak potential on the acidity of the medium and the height of the polarographic current of the complex on the acidity of the medium are shown in Fig. 5.

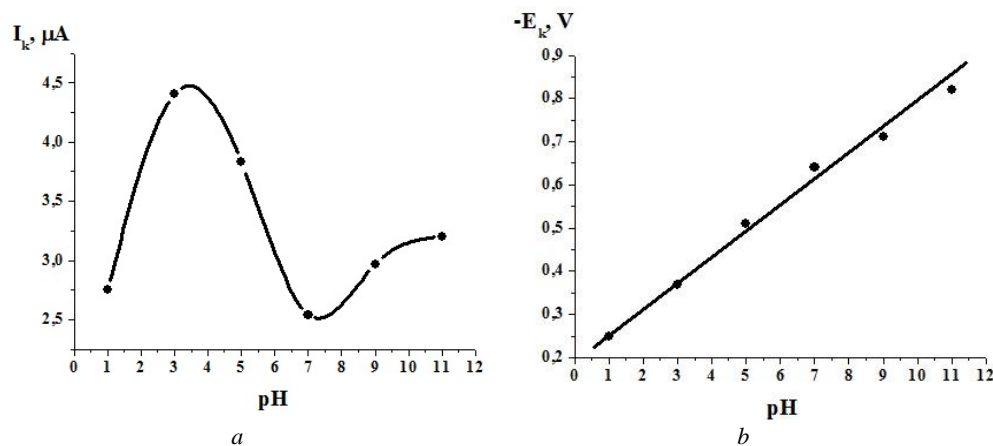


Fig. 5. Dependence of polarographic current (a) and reduction potential (b) of aqueous-ethanol solutions (50 % v/v) NBnTAN in the presence of Ni(II) ions on the acidity of the medium, $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M, $C(\text{Ni(II)})=1.0 \times 10^{-4}$ M, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s

The forms of Ni(II) existence in solutions were calculated using Visual MINTEQ 3.1. Within the concentrations of Ni(II) ions 1.0×10^{-6} M – 1.0×10^{-4} M, dimeric and polymeric aquahydroxochloride complexes of Nickel do not exist, so the total concentration of the metal does not affect the molar fractions of individual forms of existence.

The increase of current (Fig. 4) at pH=3.0 can be explained by the different forms of the reagent coexistence in solution. The decrease of the peak at pH=7.0 corresponds to a decrease in the proportion of Ni^{+2} . A further increase of current to pH=11.0 is due to an increase in the proportion of $\text{Ni}(\text{OH})_2$.

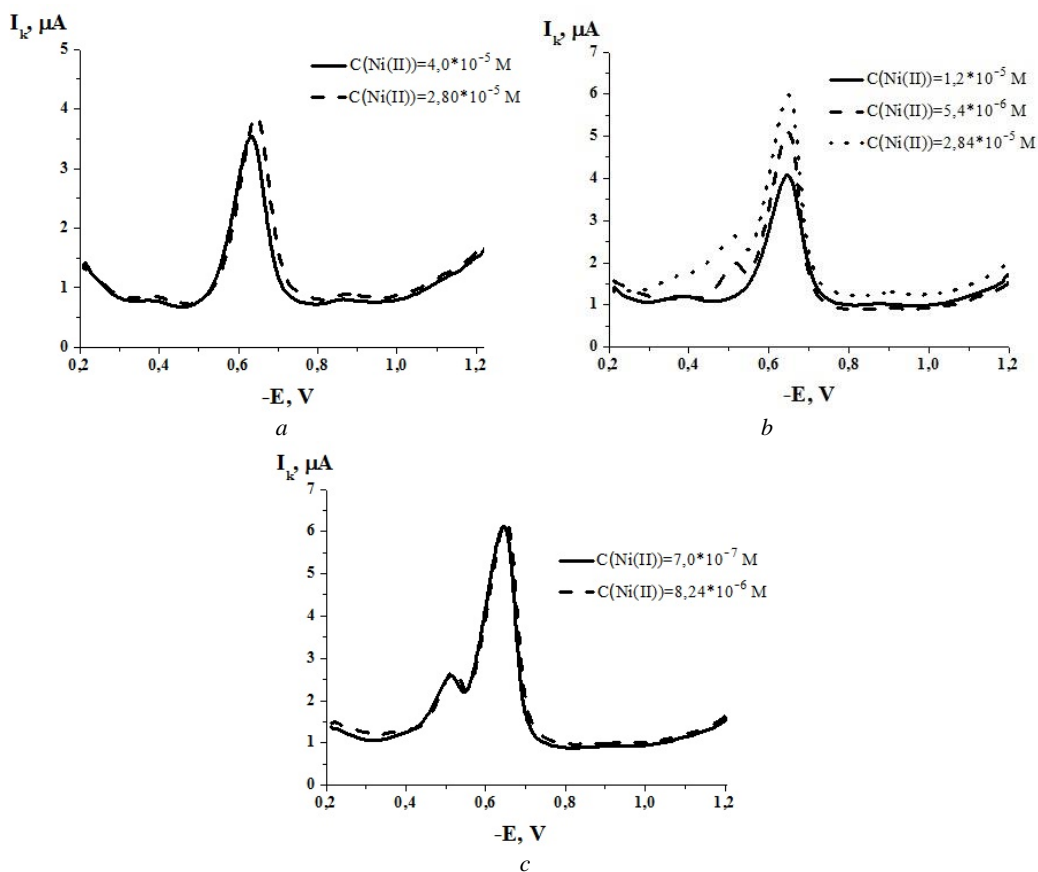


Fig. 6. Voltammograms of aqueous-ethanol solutions (50% v/v) NBnTAN in the presence of Ni(II) ions at a concentration of excess reagent: $C(\text{NBnTAN})=4.0 \times 10^{-5} \text{ M}$, $C(\text{NaCl})=0.16 \text{ M}$, $V=1.0 \text{ V/s}$, $\text{pH}=7.0$

Studies of the range of determined concentrations were performed at an azo dye concentration of $4.0 \times 10^{-5} \text{ M}$. Voltammograms of aqueous-ethanolic solutions (50 % v/v) of the complex with an excess of reagent were obtained (Fig. 6).

To determine the optimal ratio of the reacting components with the highest yield of the complex compound corresponding to its composition, the molar-ratio method was used. It showed that increasing metal content in the system at a constant concentration of azo dye maximum current is reached for the following ratio $C(\text{Ni(II)}):C(\text{NBnTAN})=1: 2$ (Fig. 7).

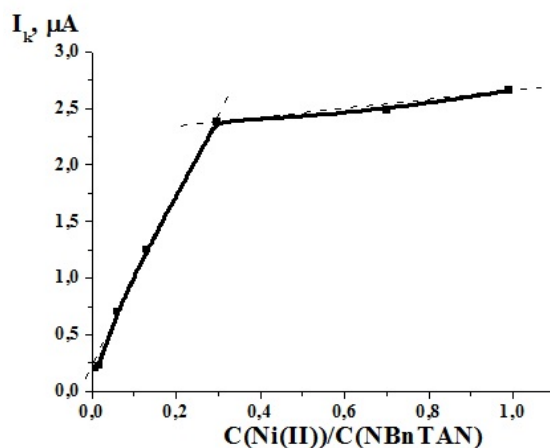


Fig. 7. Dependence of the cathode current for aqueous-ethanolic solutions (50 % v/v) NBnTAN from the ratio of the Ni(II) concentration to NBnTAN, $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M, $\text{pH}=7.0$, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s

Based on the linear dependence the current of the complex on the concentration of Ni(II) in the solution (Fig. 8), the metrological characteristics of the voltammetric determination of Nickel using NBnTAN at $\text{pH}=7.0$ were determined (Table 1). The linearity of the analytical signal is one order of magnitude.

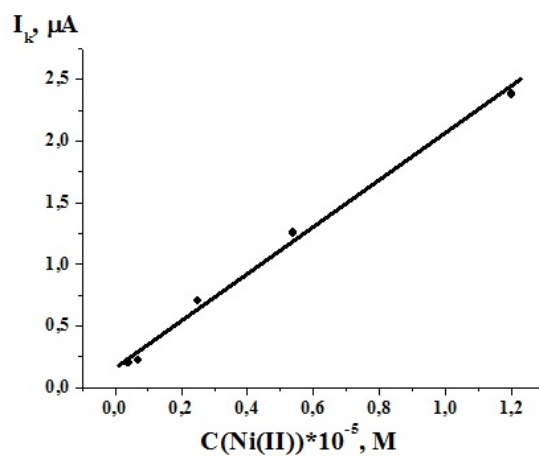


Fig. 8. Calibration graph of voltammetric determination of Ni(II) at the peak of a complex compound using aqueous-ethanolic solutions (50 % v/v) NBnTAN, $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M, $\text{pH}=7.0$, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s

Table 1

Metrological characteristics of voltammetric determination of Ni (II) at the peak of the complex compound using aqueous-ethanolic solutions (50 % v/v) NBnTAN, $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M, $E_k^p=-0.640$ V, pH=7.0, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s

Calibration equation, C(Ni), M	$I=0.16+0.19 \times 10^{-6} C_{\text{Ni(II)}}$
Limit of detection, M	1.3×10^{-7}
Limit of quantification, M	4.0×10^{-7}
Linearity range, M	$(0.04-1.20) \times 10^{-5}$
Correlation coefficient, R	0.9982

An important task in the development of analytical method was to study the effects of foreign ions that can interfere with the determination of Ni(II). The ions of the metals for which the selectivity of the voltammetric determination was studied were selected from among the metals that may be associated with nickel in industrial and natural facilities. The selectivity studies of the developed method were performed by gradually increasing the concentration ratio of Ni (II) to the foreign metal until the deviation in the peak height value in the presence of an interfering element did not exceed ± 5 % relative to the peak height value in its absence.

The results of the selectivity study of the Ni(II) ions voltammetric determination using NBnTAN are given in Table 2. It was found that the presence of the most metals, which often accompany nickel don't interfere in the determination Ni(II), except: Co(II), Mn(II), Cu(II), Zn(II).

Table 2

Tolerance limits of foreign metal ions for voltammetric determination of Ni(II) using NBnTAN
 $(C_{\text{Ni(II)}}=5.0 \times 10^{-6}$ M, $C(\text{NBnTAN})=4.0 \times 10^{-5}$ M,
 $E_k^p=-0.751$ V, pH=7.0, $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s)

Ion	$C_{\text{Me}}/C_{\text{Ni}}$	Ion	$C_{\text{Me}}/C_{\text{Ni}}$	Ion	$C_{\text{Me}}/C_{\text{Ni}}$
Cu(II)	5	Mn(II)	2	Mg(II)	>200*
Al(III)	>200*	Fe(III)	30	Ca(II)	>200*
Zn(II)	1	Co(II)	1	Ba(II)	>200*
Cd(II)	50	Pb(II)	50	Sr(II)	>200*

* Large excesses were not investigated.

The method efficiency of Ni(II) voltammetric determination using NBnTAN was tested during the analysis of an aluminum-based alloy (A1891, $C(\text{Ni})=2,7 \times 10^{-4}$ M). The composition of the alloy is presented in Table 3.

Table 3

The composition of the alloy aluminum type AK15MN (A1891)

Index CO	Mass fraction of elements, %											
	Si	Cu	Ni	Fe	Mg	Mn	Sb	Zn	Pb	P	Sn	Al
1891	12.8	2.72	0.59	1.64	0.30	0.37	0.028	0.078	0.12	0.011	0.0042	remainder

To control the correctness of the developed method of Nickel (II) voltammetric determination with NBnTAN as a reference method used the atomic absorption spectroscopy method (AAS) (Table 4).

Table 4

The results of voltammetric determination of the nickel content
in the standard sample A1891 ($C(\text{NBnTAN})=4.0 \cdot 10^{-5}$ M, $E_c^p=-0.751$ V, pH=7.0,
 $C(\text{NaCl})=0.16$ M, $V=1.0$ V/s, $n=3$, $P=0,95$)

Method	$\bar{X} \pm \frac{St_\alpha}{\sqrt{n}}$, %	Sr,
Developed method	0.63±0.06	3.8
AAS	0.61±0.04	2.6
Declared content	0.59	

The results of approbation of the developed method on complex real objects are in good agreement with the results obtained by the reference atomic absorption analysis (Table 4), which confirms the correctness of this method.

4. Conclusion

The formation of the NBnTAN complex with Ni(II) ions was studied. It was found that the chelate Ni(II)–NBnTAN is formed in the pH range of 5.0–11.0. A stable complex is formed at pH=7.0. The optimal conditions for the interaction of aqueous-ethanolic solutions (50 % v/v) of NBnTAN with nickel(II) ions are established: the ratio of the components of complex compound C(Ni(II)): C(NBnTAN)=1:2; acidity of the medium (pH=7.0); the maximum of the polarographic current is observed at the potential $E_c^p=-0.751$ V. A sensitive ($C_{\min}=1.3 \times 10^{-7}$ M) and easy-to-perform technique of voltammetric determination of Nickel using the peak of the complex compound was developed by means of 1-[(5-(3-nitrobenzyl)-1,3-thiazol-2-yl)diazanyl]naphthalen-2-ol on the background of 2.0 M NaCl at pH 7.0, scan rate was $V=1$ V/s. The determination of nickel(II) is not hindered by significant excess of many concomitant metals except: Co(II), Cu(II), Zn(II) and Mn(II). The interfering effect of these metals can be explained by the fact that they also form complexes with NBnTAN. However, since nickel and cobalt are always present simultaneously in natural deposits, this technique is not suitable for the determination of Ni(II) in such objects, but can be successfully used to determine the latter in Ni–Cd batteries and alloys. based on Fe(III), Al(III), Pb(II), Cd(II).

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**ВОЛЬТАМПЕРОМЕТРИЧНЕ ВИЗНАЧЕННЯ Ni(II) З ВИКОРИСТАННЯМ
1-[(5-(3-НІТРОБЕНЗИЛ)-1,3-ТІАЗОЛ-2-ІЛ)ДІАЗЕНІЛ]НАФТАЛЕН-2-ОЛУ****О. Федішин, Л. Олексів, С. Тимошук, О. Тимошук***Львівський національний університет імені Івана Франка,
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Для вольтамперометричного визначення Ni(II) запропоновано новий реагент – 1-[(5-(3-нітробензил)-1,3-тіазол-2-іл)діазеніл]нафтален-2-ол, який є похідним 1-[(5-бензил-1,3-тіазол-2-іл)діазеніл]нафтален-2-олу. Знайдено, що хелат Ni(II)-NBnTAN утворюється в діапазоні кислотності середовища 5,0–11,0. Стійкий комплекс утворюється за pH=7,0. З'ясовано оптимальні умови взаємодії 1-[(5-(3-нітробензил)-1,3-тіазол-2-іл)діазеніл]нафтален-2-олу з іонами Ni(II): співвідношення компонентів комплексної сполуки C (Ni(II)) : C (NBnTAN) = 1:2; кислотність середовища (pH=7,0); максимум полярографічного струму спостерігається при потенціалі $E_k^n = -0,751$ В. Запропоновано чутливу ($C_{\min} = 1,3 \cdot 10^{-7}$ М) та просту у виконанні методику вольтамперометричного визначення нікелю за піком комплексної сполуки на фоні 2 М NaCl при pH 7,0 з використанням 1-[(5-(3-нітробензил)-1,3-тіазол-2-іл)діазеніл]нафтален-2-олу з концентрацією – $4,0 \cdot 10^{-4}$ М, швидкість накладання напруги поляризації – 1,0 В/с. Визначенню нікелю (II) не заважають значні надлишки багатьох супутніх металів, за винятком: Co(II), Cu(II), Zn(II) і Mn(II). Заважаючий вплив цих іонів металів можна пояснити тим, що вони також утворюють комплекси із 1-[(5-(3-нітробензил)-1,3-тіазол-2-іл)діазеніл]нафтален-2-олом. Проте, оскільки у природних родовищах нікель і кобальт завжди є присутніми одночасно, ця методика не є придатною для визначення нікелю (II) у таких об'єктах, але може бути успішно використана для визначення останнього у Ni–Cd акумуляторах, а також у сплавах на основі Fe(III), Al(III), Pb(II), Cd(II). Проведено апробацію розробленої методики під час аналізу сплаву на основі алюмінію (А 1891). Для перевірки правильності розробленої методики вольтамперометричного визначення нікелю(II) з використанням NBnTAN як референтну методику використовували атомно-абсорбційне його визначення.

Ключові слова: 1-[(5-(3-нітробензил)-1,3-тіазол-2-іл)діазеніл]нафтален-2-ол, тіазоліазо-барвники, вольтамперометрія, нікель (II).

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