DETERMINATION OF THE OPTIMAL CONDITIONS FOR THE SPECTROPHOTOMETRIC DETERMINATION OF Ni(II) IONS USING 7-BROMO-2-NITROSO-1-OXYNAPHTHALENE-3,6-DISULFOXIDE

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Abstract. In this article, the spectrophotometric detection method of complexing Ni(II) ion with 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulfoacid (BNOKS-S, S-3,6) (HR) with organic analytical reagent developed and studied optimal conditions: λ_{max} = 640, pH = 6,5, universal buffer, reagent - buffer - Ni(II) - distilled water, BNOKS-S with 0.05% relative to $T_{Ni^{2+}}$=10 μg/25ml, 1.0 ml of S-3.6 reagent was found to be sufficient. The area of obedience to the Bouguer-Lambert-Behr law was determined to be 1.0-17.5 μg/25 ml. Absorption spectra were studied: sensitivity according to Sendel was 0.0011 μg/cm2, contrast $\Delta\lambda$ = 100 nm.

Keywords: Ni(II) ion, 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulfoacid, organic analytical reagent.

Introduction

Today, as a result of the continuous development of the industry, including the operation of enterprises, factories, and plants, many chemical compounds and toxic metals that cause environmental pollution are contaminating rainwater and wastewater as ecotoxicants[1]. These ecotoxicants include copper, cadmium, nickel, and other heavy metals. It is important to identify metals from sources contaminated with these metals and to reduce their amount or even completely clean them [2]. The optimal conditions for spectrophotometric determination of Ni(II) ions using D-penicillamine have been investigated[3].

This study outlines simultaneous spectrophotometric techniques for quantifying Ni^{2+} ions using 1-(2-pyridylazo)2-naphthol (PAN) in micellar media. The ligand and its metal complexes, namely Ni^{2+}-PAN, were rendered water-soluble with the neutral surfactant Triton X-100, eliminating the need for organic solvent extraction[4]. The spectrophotometric technique was employed to investigate the interaction between Ni(II) and Cu(II) ions with three different ligands: ethyl 4-(4-hydroxyphenyl)-6-

methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Ligand 1), 4-(1H-benzimidazol-2-yl)phenol (Ligand 2), and 2-(3-phenylamino-4,5-dihydro-1,2-oxazol-5-yl)phenol (Ligand 3). These interactions were studied under conditions of 0.01 M ionic strength and 28°C in a 70% dioxane-water mixture[5,6]. The compound 2-(2-Thiazolylazo)-p-cresol (TAC) undergoes a reaction with Ni(II) ions within the pH range of 6–10, resulting in the formation of a blue complex in a 1:2 ratio. The stability constant of this complex is determined to be 5.0 ± 0.3[7,8]. A method was developed for the extraction and concentration of nickel in the form of its dimethylglyoxime complex using triethylamine. The nickel-enriched triethylamine extract was then separated and evaporated. The resulting residue was dissolved in chloroform (50 µL), and the absorbance at 380 nm was measured. A spectrophotometric procedure for the determination of nickel was established, with a limit of detection of 0.020 µg/mL (n = 10; P = 0.95) [9,10].

Reagents and solutions. Standard solutions of Nickel(II) salts with a titer of T_{Ni}^{2+} = 1000 mg/ml were prepared using accurately weighed portions of analytical grade metallic mineral salts. "Clean" and "chemically clean" brand reagents were used in the experiment. An accurately weighed portion of nickel(II) salt was placed in a 1000 mL volumetric flask and dissolved in distilled water. Small concentration solutions were prepared by serial dilution of the prepared standard solution. Reagent solutions are prepared by dissolving 0.0500 g of reagent with distilled water in a 100 ml volumetric flask.

Methods. Spectrophotometric studies of colored solutions were carried out on an EMC-31PC-UV spectrophotometer. The pH of the solutions was controlled using a pH meter (pH-150M OAO, Gomelsky zavod izmirtelnyx priborov) with an accuracy of ±0.05 pH units.

Experimental part

One of the main conditions of the complex formation reaction is the environment of the solution. Therefore, buffer solutions with different pH values were used to obtain reproducible results. For this, 5 ml of a universal buffer solution with a pH value of 2.46 to 11.2, 0.05% 7-bromo-2-nitroso-1-oxynaphthalene-3,6-disulfoacid (BNOKS-S, S-3,6) is added to a 25 ml measuring flask reagent solution, 1.0 ml of 40 µg/ml Ni(II) solution was added and diluted by adding distilled water up to the mark of the flask. The optical density of the complex compound solution was measured in an EMC-31PC-UV spectrophotometer, at a wavelength of λ_{max}=640 nm, in cuvettes with an absorption thickness of ℓ=1.0 cm. The obtained results are presented in Figure 1.

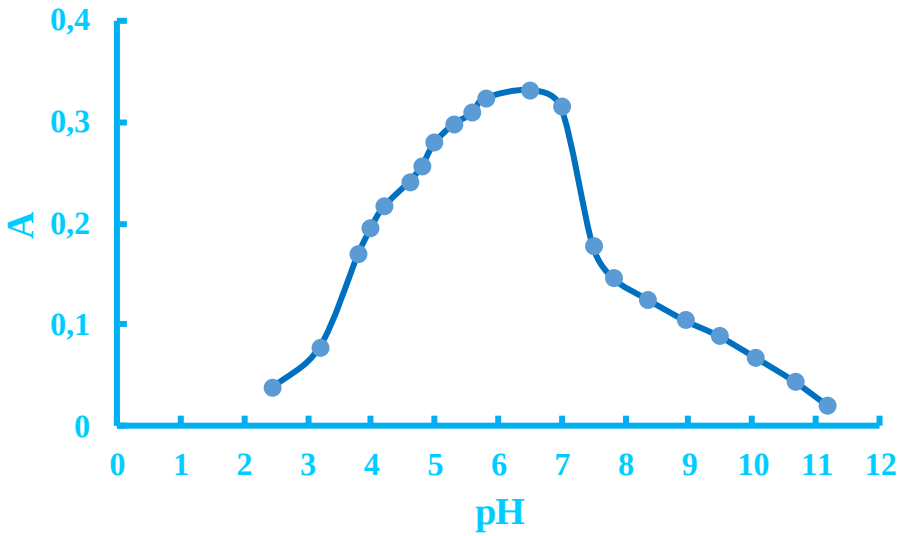

Figure 1. Graph of dependence of optical density on solution environment. (λ_{max}=640 nm, ℓ=1,0 cm, n=5)

According to the results presented in Figure 1, the highest optical density of the complex compound was observed in the range of pH 5.8-7.0. The highest optical density was chosen at pH=6.5. During the study, the effect of buffer solutions of different compositions with the same pH=6.5 on the light absorption of the color complex was studied. The results are presented in Figure 2.

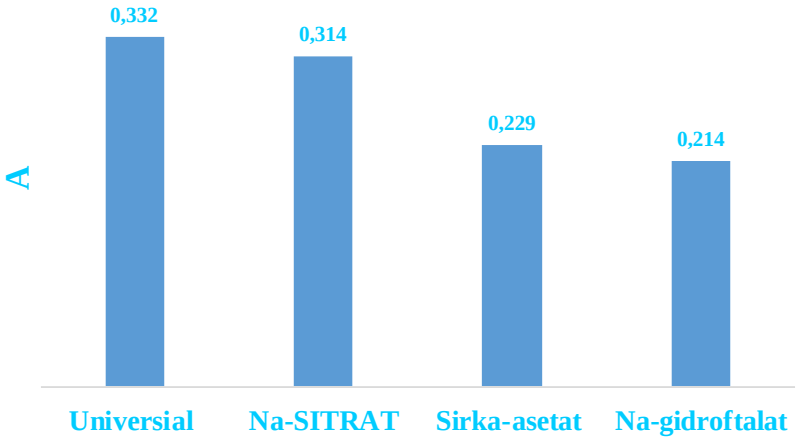

Figure 2. Graph of dependence of optical density on buffer solution content. (λ_{max}=640 nm, ℓ=1,0 cm, pH=6,5, n=5)

According to the results presented in Figure 2, the color complex showed the highest optical density under the influence of a universal buffer solution[11,12]..

During the research, the time dependence of the formation of a colored complex was studied. According to the obtained results, the fact that the optical density of the formed colored complex did not change for 4 hours indicates that there is enough time for the analysis. When the order of pouring was studied, reagent-buffer-Ni(II)-distilled water produced the highest optical density in the order. To form a complete complex

of the metal ion, the amount of reagent is taken in excess, for this purpose, the dependence on the amount of reagent was studied (Fig. 3). According to the research results, it was found that 1.0 ml of BNOKS-S, S-3.6 reagent with 0.05% $T_{Ni^{2+}}$ =10 μg/25ml is enough.

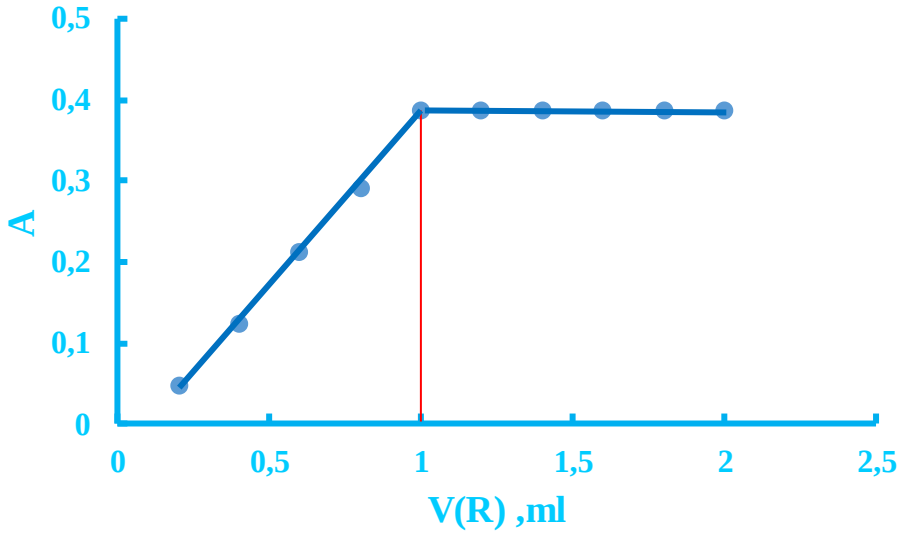

Figure 3. The graph of the dependence of the optical density of the complex compound on the amount of added reagent. λ_{max}=640 nm, ℓ=1,0 cm, pH=6,5, n=5).

Results and Discussion

Bouguer-Lambert-Behr law. 1.0 ml of 0.05% BNOKS-S, S-3,6 aqueous solution, 5.0 ml of universal buffer solution pH=6.5, variable amount of 5 μg/ ml of Ni(II) standard solution was diluted to the mark of the flask by adding distilled water. By mixing the solutions, their optical densities were measured in comparison to the reference solution. The obtained results are presented in Figure 4.

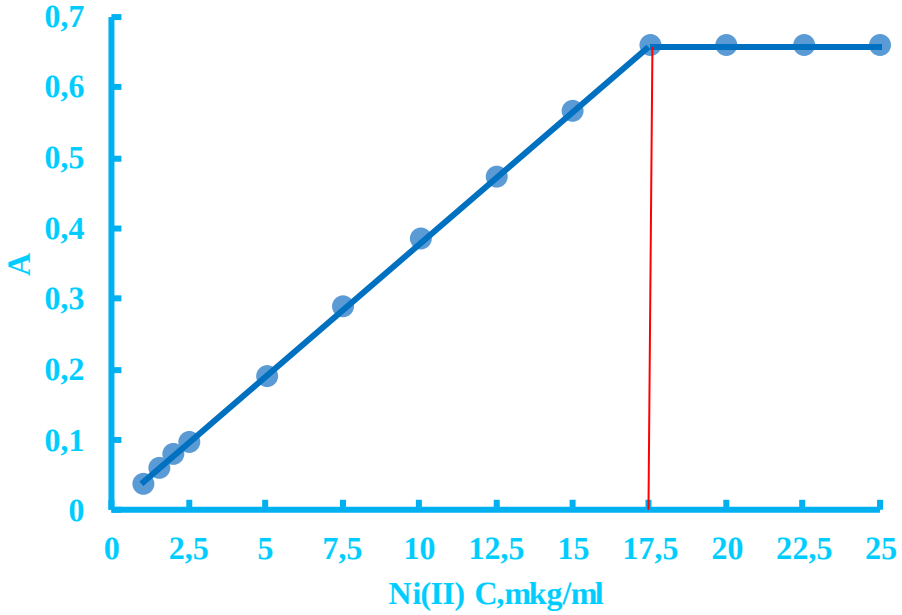

Figure 4. Graph of dependence of the optical density on the amount of added Ni(II). (λ_{max}=640 nm, ℓ=1,0 sm, pH=6,5, n=5)

According to the obtained results, compliance with the Bouguer-Lambert-Beer law was observed in the range of 1.0-17.5 μg/25ml. At higher concentrations, there was a deviation from the linear relationship[13,14].

Absorption spectra. UV-visible spectra were obtained under optimal conditions using the BNOKS-S,S-3,6 reagent and the complex formed with Ni(II) (Table 1). According to the method's sensitivity by Sendel, the light absorption per unit area (0,001 of mkg/sm^2) was calculated using the following formula:

$$S.b.s = \frac{Q \cdot l \cdot 0{,}001}{A \cdot 25} = \frac{15 \cdot 1 \cdot 0{,}001}{0{,}568 \cdot 25} = 0{,}0011 mkg/sm^2$$

Where: S.b.s - Sensitivity according to Sendel, Q - Concentration of the absorbed metal ion, l - Path length of the cuvette, A - Optical density.

Table 1. Spectral characterization of the BNOKS-S,S-3,6(HR) and its complex with Ni(II) (MeR) (C_{Ni2+}= 15 mkg, λ_{max}=640 nm, ℓ=1,0 cm, pH=6,5, n=5)

Complex color	pH	Maximum wavelength, nm		Δλ	C_{Ni}^{2+}, mkg/ 25 ml	C_{Ni}^{2+}, mol/l	Ā	Sensitivity according to Sendel, μg/cm²
		MeR	HR					
Kompleks Ni(II) bilan R (ℓ=1,0 sm, n=5)								
Blue-green	6,5	640	540	100	15	$1{,}02 \cdot 10^{-5}$	0,568	0,0011

Based on the results obtained during the research, it is evident that the reaction exhibits both a considerable contrast (Δλ=100 nm) and high sensitivity (S.b.s=0.0011 μg/cm²).

Conclusion

The optimal conditions for the formation of the complex of Ni(II) ions with the BNOKS-S,S-3,6 reagent were determined. The range of applicability according to the Bouguer-Lambert-Beer law is 1.0-17.5 μg/25ml, with a sensitivity (S.b.s) of 0.0011 μg/cm² and a wavelength shift (Δλ) of 100 nm.

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