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SPECTROPHOTOMETRIC RESEARCH INTO INTERACTION NICKEL (II) WITH 1- (2-PYRIDYLAZO) -2- HYDROXY -4-MERCAPTOFENOL AND AMINOPHENOLS

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Abstract:*The complexation reaction of nickel with 1-(2-pyridylazo)-2- hydroxy-4-mercaptophenol (PAGMP) in the presence of aminophenols (AP) was studied by spectrophotometric methods. Out of aminophenols there were used 2-(N,N-dimethylaminomethyl)-4-methylphenol and 2-(N, N-dimethylaminomethyl)-4-chlorophenol. We revealed optimum conditions for formation and extraction of differently ligand compounds and determined proportions of components in the complexes. It found that the complexes were formed in a weakly acidic environment (pH 3.5-6.5). The maximum in the light absorption spectrum is observed at* λ*=620-650 nm. The molar coefficient of light absorption is* $\varepsilon=(2.3\text{-}2.5)\times10^4$*. The optimal condition for the formation and extraction of these compounds is the concentration* $(1.2\text{-}1.5)\times10^{-3}$ *M PAGMP and* $(2.4\text{-}2.6)\times10^{-3}$ *M AP. The maximum optical density is obtained within 15 minutes. Nickel MLC extracts with PAGMP and AF are subject to Beer's law at concentrations of 0.5–18* μ*g/ml. Proceeding from the results obtained, the photometric methods for the determination of nickel in water and soil were developed.*

Keywords: *nickel, 1-(2-pyridylazo)-2- hydroxy-4-mercaptophenol, aminophenol, extraction-photometric method, detection limit, determination.*
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Introduction

Among organic reagents successfully used in analytical chemistry, it is possible to single out azooxy compounds. However, most of these reagents coordinate metal ions at the expense of oxy-groups following which the selectivity of the reagents is reduced. Introduction into molecule reagents heteroatoms - S, - N, -O, capable of selectively coordinating metal ions, can significantly increase the selectivity of the reagents [1,2]. Nickel finds extensive application in alloys. Its alloys with copper do not corrode, so they are used in the chemical industry, marine shipbuilding, aircraft construction, in the manufacture of alloyed and special steels, in coinage (Ni+Cu), for the manufacture of pendulums, scales of measuring instruments, etc. All of these factors make it necessary to determine ever smaller quantities of nickel in various objects.

Nickel belongs to the elements for the determination of which many methods have been developed [2-6]. Oximes are widely used for a number of tasks in the analytical chemistry of nickel. Dioximes are known as reagents for the spectrophotometric determination of nickel, monooximes are less selective [1]. In search of better reagents for determination of metals with ionic radius of 0.07-0.08 nm (copper, nickel, cobalt), 1-(2-pyridylazo)-2- hydroxy-4-mercaptophenol (PAGMP, H_2L) were investigated. The aim of the present work is to study the complexation of nickel (II) with PAGMF and aminophenols (AP).

Experimental part

Reagents and solutions. A solution of nickel(II) (0.1mg/ml) was prepared by dissolving in water $NiCl_2\times6H_2O$. The titer of the solution was set gravimetrically as dimethylglyoxymate [3]. We used 0.01M solutions of PAGMF and AP in chloroform. PAGMP was used in the freshly distilled form. From AP, 2-(N,N-dimethylaminomethyl)-4-methylphenol (AP_1), 2-(N,N-dimethylamino-methyl)-4-chlorophenol (AP_2) were used. The purified chloroform was used as an extractant. The ionic strength of solutions equal to $\mu=0.1$ was kept constant by injection of the calculated amount of KCl. In order to create necessary acidity of solutions, we used 1M HCl solution. All the reagents were qualified partially or chemically.

Apparatus. The optical density of the organic phase was measured by CPK-2 and SPF-26. Value of pH of aqueous phase was controlled by device I-120.2 with glass electrode. The IR-spectra were taken with spectrophotometer "Bruker".

Methods. 0.1-0.8 ml at intervals of 0.1 ml of the stock solution of nickel, 2.5 ml of 0.01M PAGMF solution and 0.8-1.0 ml AF were injected into graduated test tubes with sealed plugs. The required pH value was set by adding 1M HCl solution. The volume of the organic phase was brought to 5 ml with chloroform and the aqueous phase to 20 ml with distilled water. After 5 min the organic layer was separated and its optical density was measured at room temperature on a KFC-2 at 600 nm.

Investigation of PAGMP. PAGMP is a dark green crystalline substance, poorly soluble in water and acids, partially soluble in alkalis, acetone, well soluble in chloroform. Light absorption curves of 4×10-4 M PAGMF solutions at different pH values are shown in Fig.1. Depending on the acidity of the medium, PAGMF can exist in three forms: H_2R, HR^-, HR^{2-}.

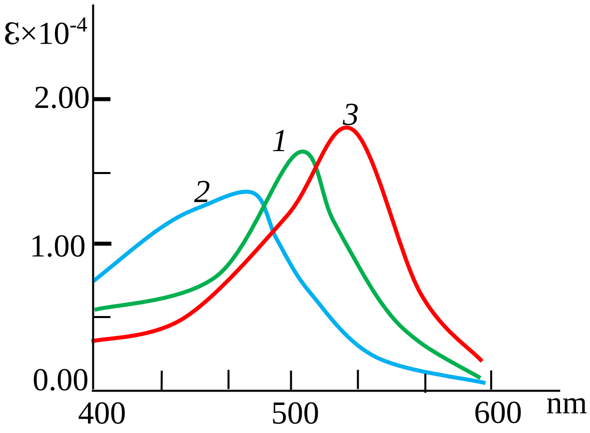
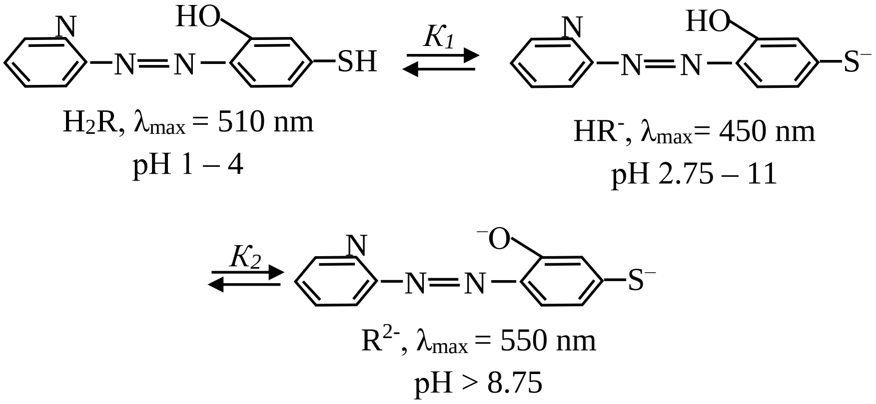


Fig. 1. Light absorption of 4×10-4 M PAGMF solutions at different pH *1* – 1.2-2.75; *2* – 4.42-8.70; *3* – 10.70-12.76.



The first proton of the sulfhydryl group is detached at pH>2.75; the second proton of the oxy-group at pH>8.5. The light absorption curves of the molecular and ionic forms noticeably differ from each other.

Values of the reagent's dissociation constants calculated by the isobestic point method and calculated by the graphical method (Fig. 2) at μ=0.1 and chloroform concentration of 40% by volume agree well with each other. Some properties of PAGMP are shown in Table 1. PAGMP appears to be in protonated forms in the strongly acidic solutions.

Numerous studies of PAGMPs have shown that they exist in organic solutions as monomers.

Results and discussion

Determination of the complex charge sign. The interaction of nickel (II) with PAGMF produced colorful complexes, insoluble in non-polar organic solvents. The charge of homogeneous ligand complexes (HLC) was determined by electro-migration of ions.

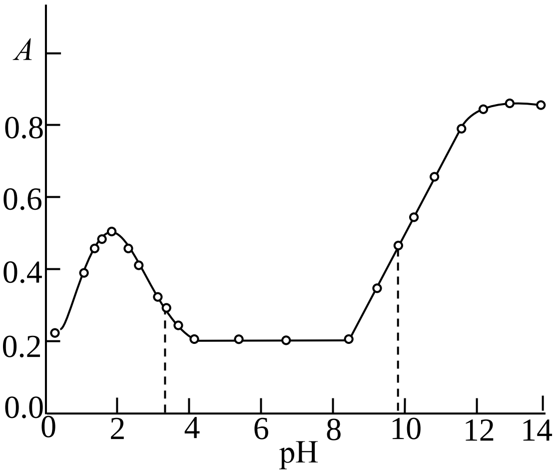


Fig. 2. Optical density - pH curve of 4×10-4 M PAGMP solutions; λ=540 nm

Table 1. Some properties of PAGMP

Reagent form	Maximum light absorption, nm	$\varepsilon \times 10^{-4}$ at λ_{max}	Isobestic point, nm	pK_{Diis}
H_2R	510	1.75±0.01	474	3.41±0.18(calcul.) 3.30 (graph)
HR^-	450	1.46±0.01		
HR^{2-}	550	1.98±0.01	482	10.00±0.12(calcul.) 9.87 (graph.)

In the study of HLC electromigration in a U-shaped tube with two taps, we observed the movement of bluish-green ions towards the positive pole and concluded that the stained Ni(II)-PAGMP complexes were anions. When AF is introduced into the system, the transition of the anionic complexes into the organic phase in the form of a mixed-ligand complex (MLC) is observed. From AP, 2-(N,N-dimethylaminomethyl)-4-methylphenol (AP_1), 2-(N,N-dimethylaminomethyl)-4-chlorophenol (AP_2) were used.

Influence of the pH of the aqueous phase. The optimum acidity interval at which the optical density is maximum and constant is at pH 3.5 - 6.5 (pH 2.3 - 8.5). At the solution pH >9, the MLC extraction is practically not observed which seems to be due to the increase in the amount of free AF molecules and the formation of hydrolyzed forms of nickel (II). The dependence of the optical density on pH is shown in Fig. 3. The presence of a single optical density maximum within the indicated pH limits confirms the assumption of the formation of a single complex compound.

Choice of extractant. The non-aqueous solvents used for MLC extraction are chloroform, 1,2-dichloroethane, carbon tetrachloride, benzene, chlorobenzene, toluene, xylene, *iso*-butanol and *iso*-pentanol. The nature of the organic solvent has a strong influence on the degree of extraction (*R*). The extractability of the complexes was evaluated by the distribution coefficient and the degree of extraction. The solvation ability of the solvent increases in parallel with the increase of its dielectric constant (*D*). An increase in solvation capacity of the solvent leads to an increase in the extractability of Ni(II)-PAGMP-AP. The degree of extraction increases in the series hexane < dioxane < cyclohexane < carbon tetrachloride = benzene < toluene < chloroform < dichloroethane < chlorobenzene (Table 2).

Chloroform, dichloroethane and chlorobenzene turned out to be the best extractants for the extract complexes. Further research was carried out with chloroform. The nickel content in the organic phase was determined photometrically with dimethylglyoxime after reextraction [1], and in the aqueous phase - by difference.

Influence of ligand concentration and holding time. Nickel MLCs are formed in the presence of a large excess of complexing reagents. The optimal condition for the formation and extraction of these compounds is the concentration (1.2-1.5)$\times 10^{-3}$ M PAGMP and (2.4-2.6)$\times 10^{-3}$ M AP. Nickel MLC extracts with PAGMP and AP obey Beer's law at concentrations of 0.5–18 µg / ml.

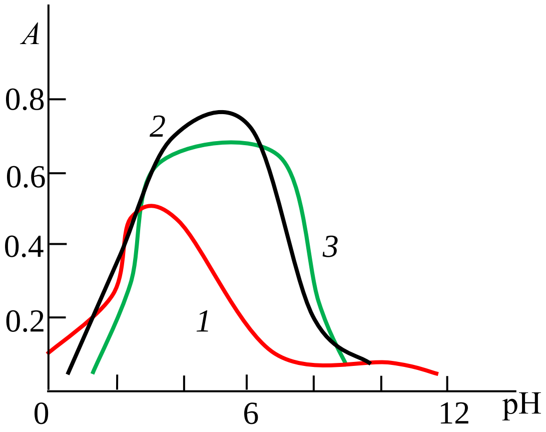


Fig. 3. Effect of aqueous phase pH on the formation and extraction of Ni(II) MLCs with PAGMP and AP.

$C_{Ni(II)} = 3.4\times10^{-5}$ M, PAGMP = 1.2×10^{-3} M, $C_{AP} = 2.5\times10^{-3}$ M; KFK-2, ℓ=0.5 cm.
1-PAGMP; 2-Ni-PAGMP-AP_1; 3-Ni-PAGMP-AP_2.

Table 2. Dependence of the extraction degree on dielectric constant of the solvents

Solvent	***D***	R, %	
		AP_1	AP_2
Hexane	1.89	92.06	93.15
Dioxane	2.00	92.09	93.78
Cyclohexane	2.05	93.51	94.61
Carbon tetrachloride	2.23	94.17	95.93
Benzene	2.23	94.17	95.93
Toluene	2.29	96.38	96.62
Chloroform	5.10	98.20	98.80
Dichloroethane	10.36	98.39	98.90
Chlorobenzene	10.30	98.42	98.91

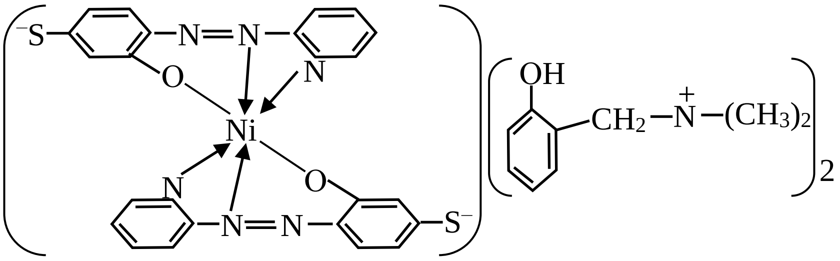
MLC of nickel with PAGMP and AP are stable in aqueous and organic solvents and do not decompose within three days, and after extraction - for more than a month. The maximum optical density is reached within 8-15 minutes.

Absorption spectra. The maximum analytical signal during the complexation of nickel with PAGMP and AP is noticeable at 620–650 nm. PAGMP absorbs maximum at 530 nm. During complexation, a bathochromic shift of the maximum of light absorption by 90-120 nm is observed. The contrast of the reactions is high: PAGMP is red, and the complexes are bluish-green. Colored chloroform extracts of PAGMP - AP associates absorb maximum light at 550-580 nm. The molar absorption coefficients are (2.3-2.5) × 10^4 L mol^{-1} cm^{-1}.

Composition and structure of the complexes. The stoichiometry of the complexes under study was established by the methods of equilibrium shift and relative yield [7]. In the composition of MLC for 1 mole of nickel 2 moles of PAGMP and AP. It was established by the Nazarenko method that the complexing form of nickel is Ni2+ [8]. In this case, the number of protons displaced by him from one H_2L molecule turned out to be equal to 2.

The method of coordination of nickel with ligands was established using IR spectroscopy. The complexes were isolated as described above, then tablets were pressed with potassium bromide in a ratio of 1: 200, and samples of PAGMP and AP were prepared in the same way. The spectra were recorded in the range 400-3600 cm^{-1}. The difference in the spectra of PAGMP and the Ni-PAGMP-AP system is indicative of the strong interaction. The bands of stretching vibrations of the OH group (3600-3200 cm^{-1}) which are observed in the spectrum of PAGMP, are absent in the complex. The absence of this band in the spectrum of the nickel complex with PAGMP and AF indicates the formation of a Ni-O chemical bond. The absorption band of PAGMP at 1395 cm^{-1} (-N = N-) is shifted to 1315 cm^{-1} which indicates the formation of a coordination bond of the nickel atom with this group. The observed decrease in the intensity of the band at 2580 cm^{-1} in the PAGMP spectrum is illustrative of the participation of the –SH group in the formation of the complex in the ionized state. It is assumed that PAGMP is a tridentate ligand coordinated with Ni^{2+} through the nitrogen atom of the azo group ($\upsilon_{N=N}$ = 1395 cm^{-1}), bottom nitrogen (υ_{C-N} = 1290 cm^{-1}; υ_{C-N} = 1170 cm^{-1}) and an atom oxygen (δ_{C-O} = 1250). The detection of absorption bands at 1370 cm^{-1} points at the presence of protonated AP [9, 10].

Proceeding from the data obtained, the composition of the extracted complexes can be represented by the formula $[NiL_2](APH^+)_2$:



The mechanism of MLC formation can be represented as follows: L at pH 2.3-8.5 forms a compound insoluble in chloroform with an excess of nickel ions. AR is added to such a system, a compound is instantly formed, soluble in chloroform, intensely colored in a bluish-green color. Calculations have shown that MLCs in the organic phase do not polymerize and are in the monomeric form (γ = 0.94–1.07).

Equilibrium and extraction constants. It can be assumed that the processes occur during complexation:

$$Ni^{2+} + 2H_2L \leftrightarrow [NiL_2]^{2-} + 4H^+$$

$$[NiL_2]^{2-} + 2A\Phi H^+ \leftrightarrow [NiL_2](A\Phi H)_2$$

The equilibrium constant of the reaction is

$$K_p = \frac{\{[NiL_2](A\Phi H)_2\}_{(org)}}{\{[NiL_2]^{2-}\}_{(aq)}\{[A\Phi H^+]^2\}_{(aq)}} = \lg\frac{A_x}{A_o - A_x} \times \frac{1}{[A\Phi]^2}$$

Since the distribution coefficient (D) is

$$D = \lg\frac{A_x}{A_o - A_x}, \quad \text{то} \quad K_{eq} = \frac{D}{[A\Phi H^+]^2}$$

Taking the logarithm of the last expression, we get
$\lg K_{eq.} = \lg D - 2\lg[A\Phi H^+]$
Extraction constants were calculated using equations
$\lg K_{ex} = \lg D - 2\lg[L^{2-}] - 2\lg[HA\Phi^+]$.

The results of calculating the equilibrium constant and the extraction of the reaction are shown in Table 3.

Based on the equations of the calibration curves, the limit of photometric detection and the limit of quantitative determination of nickel in the form of MLC were calculated [11]. Table 3 shows the analytical characteristics of nickel complexes with PAGMP and AP.

The influence of foreign ions. To assess the applicability of MLC for the separation and determination of nickel, the interfering effect of foreign ions was studied. It revealed that a large amount of alkaline, alkaline-earth elements, REE, F^-, Cl^-, Br^-, SO_3^{2-}, SO_4^{2-}, NO_2^-, NO_3^- and $C_2O_4^{2-}$ do not interfere with the nickel determination. The interfering effect of Fe (III) was removed with oxalic acid; Ti (IV) - sodium fluoride or tyron; Hg (II) sulfite ion; Nb (V) and Ta (V) - with oxalic acid, and Mo (VI) and W (VI) - with sodium fluoride and oxalic acid. When using a 1% solution of ascorbic acid, the determination does not interfere with Mn (VII), V (IV), Nb (V), Cr (VI), Mo (VI), and Fe (III). When a 0.01 M solution of oxalic acid is used, V (IV), Nb (V), Ta (V), Cr (III), Mo (VI), W (VI), and Fe (III) do not interfere with the determination.

Table 3. Characteristics of MLC nickel with PAGMP and AP

Parameter	$[NiL_2](A\Phi_1H)_2$	$[NiL_2](A\Phi_2H)_2$
pH range of education and extraction	0.7-8.5	0.5-8.4
pH range of maximum extraction	3.8-6.5	3.5-6.0
Molar absorptivity (L $mol^{-1}cm^{-1}$)	2.3	2.5
Degree of extraction (*R,%*).	98.20	98.80
Organic solvent	($CHCl_3$)	($CHCl_3$)
Extraction time (min)	8	15
λ_{max} (nm)	620	650
λ (nm)	90	120
Coefficient polymerization (γ)	0.94	1.07
Equilibrium constant ($\lg K_e$)	7.69	8.44
The extraction constant ($\lg K_{ex}$)	9.69	9.26
Stability constant (β)	10.67	10.28
The equation of calibration curves	$y = 0.086+0.0072x$	$y = 0.062+0.0084x$
Correlation coefficient	0.9981	0.9984
Beer's law range (μg ml^{-1})	0.5-16	0.5-18
Limit of detection (ng mL)	9.2	8.73
Limit of quantification (ng mL)	28.4	26.5
Sandell's sensitivity (ng mL)	2.40	2.29

Table 4 shows the data which makes it possible to compare different methods for the nickel determination. It can be seen that PAGMP has advantages over other reagents: the maximum of light absorption is shifted to the long-wavelength region of the spectrum [12-17], the molar coefficient of light absorption is much higher than the molar coefficients of light absorption of other complexes [12-14, 16], the pH of the reaction is shifted to a more acidic region [12, 15], which increased the selectivity.

The developed technique was applied to determine nickel (II) in various objects.

Determination of nickel in drinking water. Drinking water samples were taken from the territory of Gedabek region of Azerbaijan. Water samples (2 *l*) were evaporated to a volume of 50 ml with the addition of 0.1 M HCl solution to pH 4.3-6.8. An aliquot of 20 ml was introduced into an extraction vessel, an $n \times 10^3$ excess of PO_4^{3-} was added to mask Fe^{3+}, and the nickel content was determined using the developed methods. The nickel content is found according to a previously constructed calibration graph.

The correctness of the proposed method was tested on a model mixture containing the most common components of waters in a natural sample. In drinking water, the Ni^{2+} content was $(3.5 \pm 0.5) \times 10^{-3}$ µg in a 20 ml sample. The results of the determination of nickel are shown in Table 5.

Determination of nickel in soil. The developed methods were applied to determine nickel in soil taken from the Caspian zone.

A sample of soil finely ground in an agate mortar (0.5 g) was calcined in a muffle furnace for 3 h. After cooling, the sample was processed and dissolved in a graphite cup at a temperature of 50–60°C with a mixture of 16 ml of concentrated HF, 5 ml of concentrated HNO_3 and 15 ml of concentrated HCl. To remove excess hydrogen fluoride, 8 ml of concentrated HNO_3 were added to the solution three times and evaporated each time to 5–6 ml. After that, the solution was transferred into a 100 ml volumetric flask and the volume was brought to the mark with distilled water. Nickel content was determined in aliquots of the solution using the developed methods. The results are shown in Table. 6.

Table 4. Comparative characteristics of methods for the determination of nickel (II)

Reagent*	pH (solvent)	λ, nm	$\varepsilon \times 10^{-4}$	Linear range of gradation graphs, µg mL^{-1}	Ref.
DMG	8-12 ($CHCl_3$)	470	1.56	0.26-2.10	[12]
CATK	6.0 (C_6H_6)	400	1.40	0.4-10	[13]
MCCATC	6.0 (C_6H_6)	410	1.67	0.1-12	[14]
TCQ	8.7 -9.5 (C_6H_6)	522	3.17	0.02-0.70	[15]
PPTC	4.0-6.0 (C_6H_6)	430	1.92	0.5-50	[16]
HBB	4.0 (C_6H_6)	497	2.85	0.01-0.10	[17]
PAGMP-AP_1	2.6-8.5($CHCl_3$)	620	2.30	0.5-16	This work
PAGMP -AP_2	2.3-8.4($CHCl_3$)	650	2.50	0.5-18	

Note: DMG – Dimethylglyoxime, CATK – Carbazolecarboxaldehyde-3-thiosemicarbazone, MCCATC – 7-methyl-2-chloroquinoline-3-carbaldehyde thiosemicarbazone, TCQ – Thiazole-2-carbaldehyde-2-quinolylhydrazone, PPTC – Pyridoxal-4-phenyl-3-thiosemicarbazone, HBB – 4-hydroxybenzaldehyde-4-bromo phenylhydrazine

Table 5. Results of extraction-photometric determination of nickel (II) in a model system (M.S.) and drinking water (**D. W.**) ($n = 6$; $p = 0.95$)

Reagent (s)	Ni^{2+} content in the sample (Vsample = 20 ml), µg		$\bar{X} \pm \frac{t_p \times S}{\sqrt{n}}$
	Introduced	Found	

	M. S.	D.W.	M. S.	D. W.	M. S.	D. W.
PAGMP-AP$_1$	0.02	0.018	0.02 ±0.05	0.035 ±0.009	0.02±0.04	0.035 ± 0.005
PAGMP -AP$_2$	0.02	0.019	0.02 ±0.03	0.036 ±0.007	0.02±0.04	0.036 ± 0.005
Dimethylglyoxime	0.02	0.019	0.02 ±0.04	0.036 ±0.002	0.02±0.04	0.036 ± 0.003

Table 6. Results of determination of nickel in soil (section depth 10–20 cm, $n = 3$, $p = 0.95$)

Method	$\bar{x}$, % · 10−3	$\bar{x} \pm \frac{t_p \times S}{\sqrt{n}}$
PAGMP-AP$_1$	4.607 ± 0.57	4.607 ± 0.43
PAGMP -AP$_2$	4.505 ± 0.22	4.505 ± 0.61
Dimethylglyoxime	4.476 ± 0.41	4.476 ± 0.35

The developed methods for determining the nickel content in various natural objects were monitored by dimethylglyoxime methods [1].

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NİKELİN (II) 1-(PİRİDİLAZO)-2-HİDROKSİ-4-MERKAPTOFENOL VƏ AMİNOFENOLLA QARŞILIQLI TƏSİRİNİN SPEKTROFOTOMETRİK TƏDQİQİ

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Spektrofotometrik üsullarla nikelin (II) 1-(piridilazo)-2-hidroksi-4-merkaptofenol və aminofenolla kompleksəmələgəlmə reksiyası tədqiq edilmişdir. Aminofenol kimi 2(N, N-dimetilaminometil) -4- metilfenol (AF_1) *və 2(N, N-dimetilaminometil)-4-xlorfenol* (AF_2) *istifadə edilmişdir. Müxtəlifliqandlı komplekslərin əmələgəlmə və ekstraksiya şəraiti tədqiq edilmiş və komplekslərdə komponentlərin nisbəti müəyyən edilmişdir (Ni(II):L:AF =1:2:2). Müxtəlifliqandlı komplekslər zəif turş mühitdə əmələ gəlir* (pH_{opt}*=3.5-6.5). İşıqudma spektrində maksimum 620-650 nm-də müşahidə olunur. Molyar işıqudma əmsalı* ε *=(2.3-2.5)×* 10^4*. Müxtəlifliqandlı kompleksin əmələgəlməsi üçün (1.2-1.5)×*10^{-3} *L və ((2.4-2.6)×*10^{-3} *M AF qatılıq tələb* olunur. *Maksimum optiki sıxlıq 15 dəq müddətində əmələgəlir. Ber qanununa tabe olma volframın 0.5 - 18 mkq/ml qatılıq intervalında özünü doğruldur. Alınan nəticələr əsasında suda və* torpaqda nikelin təyini üçün *fotometrik metodikalar işlənib hazırlanmışdır.*

Açar sözlər: nikel, 1-(piridilazo)-2-hidroksi-4-*merkaptofenol, aminofenol, ekstraksiyalı*-fotometrik metod, *təyinetmə həddi*

СПЕКТРОФОТОМЕТРИЧЕСКОЕ ИССЛЕДОВАНИЕ ВЗАИМОДЕЙСТВИЯ НИКЕЛЯ (II) С 1-(2-ПИРИДИЛАЗО)-2- ГИДРОКСИ -4-МЕРКАПТОФЕНОЛОМ И АМИНОФЕНОЛАМИ

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Спектрофотометрическими методами изучена реакция комплексообразования никеля с 1-(2-пиридилазо)-2-гидрокси-4-меркаптофенолом (ПАГМФ) в присутствии аминофенолов. Из аминофенолов использованы 2-(N,N-диметиламинометил)-4-метилфенол и 2-(N, N-диметиламинометил)-4-хлорфенол. Найдены оптимальные условия образования и экстракции разнолигандных соединений и установлены соотношения компонентов в комплексах. Установлено, что разнолигандные комплексы (РЛК) образуются в слабокислой среде ($pH_{\text{опт}}$ *3.5-6.5). Максимум в спектре светопоглощения наблюдается при* λ*=620-650 нм. Молярный коэффициент светопоглощения равен* ε *=(2.3-2.5)×* 10^4*. Оптимальным условием образования и экстракции этих соединений является концентрация (1.2-*

1.5)$\times 10^{-3}$ *моль/л ПАГМФ и (2.4-2.6)*$\times 10^{-3}$*моль/л АФ. Максимальная оптическая плотность достигается в течение 15 мин. Экстракты РЛК никеля с ПАГМФ и АФ подчиняются закону Бера при концентрациях 0.5–18 мкг/мл. На основании полученных результатов разработаны фотометрические методы определения никеля в воде и почве.*

Ключевые слова: *никель, 1-(2-пиридилазо)-2- гидрокси -4-меркаптофенол, аминофенол, экстракционно-фотометрический метод, предел обнаружения, определение.*