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STUDY OF REACTION OF NICKEL (II) WITH 2,4-TYAZOLIDINDYON COMPLEX AND ITS DERIVATIVES**A.Z. Zalov, K.A. Kuliyeu, K.R. Aliyeva**

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Abstract: Spectrophotometric methods are used to study the reaction of the nickel complex with 2,4-tyazolidindion (L) and its derivatives {5-(gasilyliden) -2,4-tyazolidindion (L_1), 5-(2-chlorbenzyliden) -2,4-tyazolidindion (L_2), 5-(2-hydroxybenzyliden) -2,4-tyazolidindion (L_3), 5-(4-hydroxybenzyliden)-2,4-tyazolidindion (L_4), 5-(2-Brom-5-methoxybenzyliden)-2,4-tyazolidindion (L_5). With a single extraction, the chloroform is extracted by 97.8-98.4 % of the nickel. The study of the dependence of complexation on pH showed that the optimal range of acidity, at which the optical density is maximum and constant, is at pH 2.3-8.0. The optimal condition for the formation and extraction of the nickel complex of these compounds is $(2.0-2.5) \times 10^{-4}$ M concentration L. Nickel complexes with L are stable in water and organic solvents and do not decompose for three days, and after extraction for more than a month. The maximum optical density is achieved within 5 minutes. The maximum analytical signal during the complex of nickel L is observed at 470-482 nm/ molar absorption coefficients $(1.37-1.61) \times 10^4$. The comprehensive form of nickel is Ni^{2+} while the number of protons supplanted by it from one molecule L turned out to be equal to 1. Nickel and its complexes in the organic phase are not polymerized and are in a monomeric form ($\gamma = 1.01-1.07$). The proposed method under already established optimal conditions is used to determine Ni (II) in wastewater, bottom deposits, as well as in oil and oil products.

Keywords: nickel, 2,4-tyazolidindion, chloroform, oil, oil products

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Introduction

For the photometric determination of nickel, many chelating reagents belonging to different classes of compounds and containing N, O, or S as donor atoms are suitable [1]. Nickel is one of the metals that have chromophoric properties; therefore, among the numerous photometric methods for the determination of nickel, there are both methods based on the use of colored reagents with chromophore groups, as well as methods that use colorless reagents.

It is known that by improving the analytical parameters of the reaction of the formation of mixed ligand complexes, they have found wide application in the photometric determination of elements [2-17].

For the extraction-spectrophotometric determination of nickel (II), dimethylglyoxime

was proposed [1]. A sensitive and selective method has been developed for the spectrophotometric determination of nickel (II) in alloys and in water [5, 12, 13]. Spectrophotometric methods have been proposed for the determination of nickel in some ecological and biological objects with 2-hydroxythiophenol [13], azomercaptophenol {1-(2-pyridylazo)-2-hydroxy-4-mercaptophenol, 1-(5-chloro-2-pyridylazo)-2-hydroxy-4-mercaptophenol, 1-(5-bromo-2-pyridylazo)-2-hydroxy-4-mercaptophenol, 1-(5-iodo-2-pyridylazo)-2-hydroxy-4-mercaptophenol} in the presence of aminophenols in various natural materials [18,19].

It was of interest to study the interaction of nickel with 2,4-thiazolidinedione (L) and its derivatives {5-(benzylidene)-2,4-

thiazolidinedione (L_1), 5-(2-chlorobenzylidene)-2,4-thiazolidinedione (L_2), 5-(2-hydroxybenzylidene)-2,4-thiazolidinedione (L_3),

5-(4-hydroxybenzylidene)-2,4-thiazolidinedione (L_4), 5-(2-bromo-5-methoxybenzylidene)-thiazolidine-2,4-dione (L_5).

Experimental part

Reagents and solutions. To prepare the stock solution (1 mg/mL) of nickel, 3.9296 g of freshly recrystallized $NiCl_2 \cdot 6H_2O$ was dissolved in water, 2 drops of conc. H_2SO_4 and diluted in a volumetric flask with water to 1 L [1]. Solutions with a concentration of 0.1 mg/ml were obtained by diluting the stock solution.

The 2,4-thiazolidinedione derivatives (L) were purified by re-precipitation from ethanol solutions by adding water and then by distillation. We used 0.01 M solutions of L chloroform while purified chloroform was used as an extractant.

The ionic strength of the solutions, equal to $\mu=0.1$, was maintained constant by introducing the calculated amount of KCl. A 1

M NaOH solution was used to create the required acidity of the solutions. All reagents used were of analytical grade. or x. h.

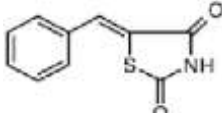
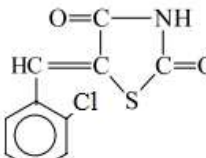
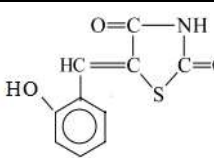
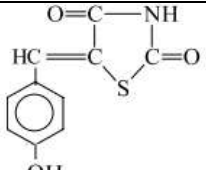
Methods. In graduated test tubes with ground stoppers, 0.1–0.8 ml, with an interval of 0.1 ml of the initial copper solution, and 2.5 ml of 0.01 M solution L were introduced. The required pH value was set by adding 1 M NaOH solution. The volume of the organic phase was adjusted to 5 ml with chloroform, and the volume of the aqueous phase was adjusted to 20 ml with distilled water. After 5 minutes, the organic layer was separated and its optical density was measured at room temperature on KFK-2 at 490 nm.

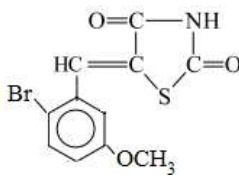
Results and discussion

L are monobasic (L_1 , L_2 , L_3 and L_5) and dibasic weak acids (L_4) and, depending on the pH of the medium, can exist in molecular and

anionic forms. Some characteristics of the studied reagents are presented in Table. 1.

Table 1. Some characteristics of the analyzed reagents

Reagent	Structural formula	Composition of neutral	pK ₁	pK ₂	pH existence of in neutral form
L_1		HL	8.65	-	0-8 ($\lambda=340$ nm)
L_2		HL	8.45	-	0-8 ($\lambda=345$ nm)
L_3		HL	6.35	10.10	0-8 ($\lambda=349$ nm)
L_4		H ₂ L	6.46	10.26	0-5 ($\lambda=351$ nm)

L ₅		HL	8.47	-	0-5(λ=354 nm)
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Choice of extractant. The complexes were extracted using non-aqueous solvents: chloroform, 1,2-dichloroethane, carbon tetrachloride, benzene, chlorobenzene, toluene,

xylene, isobutanol, isopentanol, and diethyl ether. The extractability of the complexes was evaluated by the distribution coefficient (D) and the degree of extraction (R%) [7]:

$$D = \frac{[Ni]_{org.}}{[Ni]_{aq.}}, \quad R = \frac{100 \times D}{D + \frac{V_{aq}}{V_{org}}}$$

The best extractants were chloroform, dichloroethane and carbon tetrachloride. As for a single extraction with chloroform, 97.8-98.4% of nickel is extracted. Further studies were carried out with chloroform.

Influence of the pH of the aqueous phase. The study of the dependence of complexation on pH showed that the optimal range of acidity, at which the optical density is

maximum and constant, is at pH 2.3-8.0. In a solution pH of 8, the extraction of complexes is practically not observed. The dependence of optical density on pH is shown in Fig.1. The presence of one optical density maximum within the indicated pH ranges confirms the assumption of the formation of one complex compound.

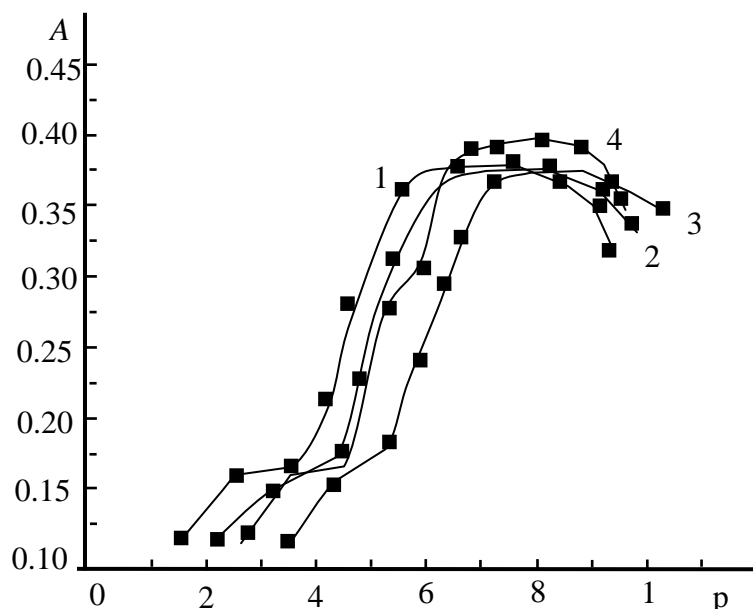


Fig.1. Effect of aqueous phase pH on the formation of Ni-L complexes.

1- Ni-L₁; 2- Ni-L₂; 3- Ni-L₃; 4- Ni-L₄;
 $C_{Ni(II)} = 3.44 \times 10^{-5}$ M, $C_L = 2.5 \times 10^{-3}$ M, $\lambda = 540$ nm.

Influence of ligand concentration and holding time. The optimal condition for the formation and extraction of nickel complexes of these compounds is $(2.0-2.5) \times 10^{-4}$ M

concentration L.

Nickel complexes with L are stable in aqueous and organic solvents and do not decompose within three days, and after

extraction - more than a month. The maximum optical density is reached within 5 minutes.

Absorption spectra. The maximum analytical signal during the complex formation of nickel with L is observed at 470–488 nm (Fig. 2). L maximally absorb at 340–354 nm. During complex formation, a bathochromic shift

of the light absorption maximum by 130–134 nm is observed. The reaction contrast is high: the starting reagents are almost colorless, while the complexes are yellowish-brown.

The molar absorption coefficients are $(1.37-1.61) \cdot 10^4$.

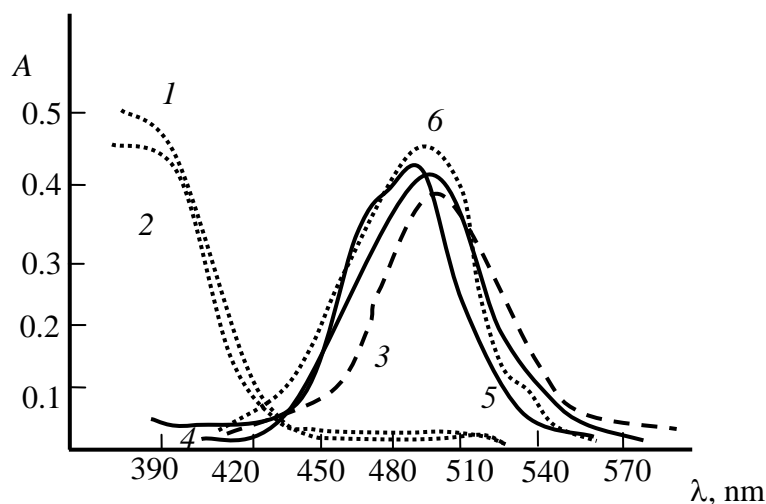


Fig. 2. Absorption spectra of chloroform extracts of reagents and complexes with Ni-L

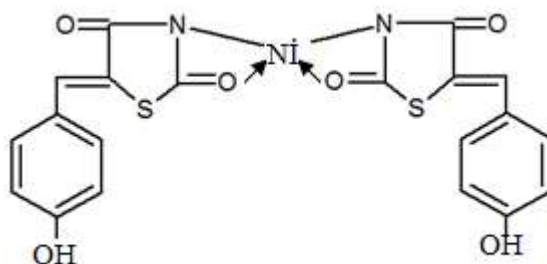
1-L₁; 2-L₂; 3-Ni-L₁; 4. Ni-L₂; 5. Ni-L₃; 6. Ni-L₄.

$C_{Ni(II)} = 3.44 \times 10^{-5}$ M; $C_L = 2.5 \times 10^{-3}$ M, $pH_{opt} = 4.5-8.3$, $C\Phi-26$, $\ell = 1.0$ cm.

Composition and structure of complexes. The stoichiometry of the analyzed complexes was determined by the methods of equilibrium shift and relative yield [20]. The calculations performed show that the composition of complexes contains two moles of L per mole of nickel. Using the Nazarenko method, it was found that Ni^{2+} is the complexing form of nickel [18]. In this case, the number of protons displaced by it from one molecule L turned out to be equal to 1.

The performed calculations showed that nickel complexes with L in the organic phase do not polymerize and are in the monomeric form ($\gamma = 1.01-1.07$) [13].

Based on the ratio of components in the resulting complexes and the number of displaced protons in the ionic form of nickel, one can imagine the probable structure of the complexes on Ni-L.

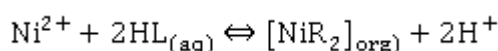


Scheme 2. Main spectrophotometric characteristics of rlc nickel.

Table 2. Characterization of nickel complexes with L.

Compound	pH range complexation	pH range of maximum extraction	λ , nm	$\epsilon \cdot 10^{-4}$	$\lg K_{eq}$	$\lg k_{ex}$	$\lg \beta$	Interval compliance with Beer's law, mkg/mL
Ni-L ₁	2.4-8.5	6.6-8.0	470	1.37	6.97	12.46	10.32	0.2-20
Ni-L ₂	2.5-8.3	6.7-7.8	474	1.48	6.85	12.70	10.45	0.2-20
Ni-L ₃	2.5-8.0	6.2-7.3	472	1.54	6.72	12.54	10.39	0.2-13
Ni-L ₄	2.5-7.5	6.4-7.4	480	1.61	6.73	12.10	10.35	0.2-15
Ni-L ₅	2.6-8.5	6.4-7.5	482	1.75	6.77	13.02	10.61	0.2-15

Equilibrium and extraction constants. occur during complex formation:
It can be assumed that the following processes



The equilibrium constant of the reaction is

$$K_p = \frac{[NiL_2][H^+]^2}_{[Ni^{2+}][HL]_{aq}^2} = \lg \frac{A_x}{A_0 - A_x}$$

Since the distribution coefficient (D) is

$$D = \frac{A_x}{A_0 - A_x}$$

then

$$K_p = \frac{D}{[HL]^2}$$

Taking the logarithm of the last expression, we get

$$\lg K_p = \lg D - 2 \lg [HL]$$

Extraction constants were calculated from the equations

$$\lg K_{ex} = \lg D - 2pH - 2 \lg [HL].$$

To determine the stability constant, the line crossing method was used

$$\beta_{s.c.} = \frac{C_k}{(C_{Ni} - C_k) \times (C_L - nC_k)^n}$$

The results of calculating the equilibrium constant, extraction and stability of the reaction were given in Table 2.

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 complexes were calculated [20]. Table 3 shows the analytical characteristics of

some nickel complexes with L.

The dependences found by us are consistent with the literature data, indicating that with an increase in pK_1 of the complexing reagents, the strength of the complex compounds formed by them and the pH of the complex formation increase as well.

Table 3. Analytical characteristics of nickel complexes with L.

Parameter	Ni-L ₁	Ni-L ₂	Ni-L ₃
Equation of calibration curves	$y = 0.055 + 0.0668x$	$y = 0.056 + 0.0502x$	$y = 0.059 + 0.052x$
Correlation coefficient	0.9981	0.9984	0.9989
Linear range of calibration curves, $\mu\text{g/mL}$	0.06-3.2	0.04-3.5	0.04-3.6
Limit of detection ng/cm^3	9.2	8.70	8.50
Limit of quantitative determination, ng/cm^3	33	36	33
Sensitivity, ng/cm^2	2.41	2.31	2.19

Table 4 shows the data that make it possible to compare the analytical characteristics of nickel determination methods with some already known [18,21–26] methods. It can be seen that L has advantages over other reagents: the light absorption maximum is

shifted to the long wavelength region of the spectrum [22, 23, 25], the molar light absorption coefficient is much higher than the molar light absorption coefficients of other complexes [21, 23], the reaction pH shifts to a more acidic region [21 -26], which increased the selectivity.

Table 4. Comparative characteristics of methods for determination of nickel (II) with already known reagents.

Reagents*	pH (extractant)	λ , nm	$\varepsilon \times 10^{-4}$	Linear range of calibration curves, mkg/mL	Ref.
DMG	8-12 (CHCl_3)	470	1.56	0.26-2.10	[21]
ECTSC	6.0 (C_6H_6)	400	1.40	0.4-10	[22]
MCCTSC	6.0 (C_6H_6)	410	1.67	0.1-12	[23]
PPTSC	8.7 -9.5 (C_6H_6)	522	3.17	0.02-0.70	[24]
ΠΦТСК	4-6(C_6H_6)	430	1.92	0.5-50	[25]
HBBPH	4 (C_6H_6)	497	2.85	0.01-0.10	[26]
BPHMP-AP	1.2-8.4 (CHCl_3)	635	4.60	0.20-18	[18]
ИПГМΦ-AP	2.3-8.4 (CHCl_3)	650	4.40	0.15-20	
Ni-L ₃	2.5-8.0(CHCl_3)	472	1.54	0,2-13	
Ni -L ₄	2.5-7.5(CHCl_3)	480	1.61	0,2-15	
Ni -L ₅	2.6-8.5(CHCl_3)	482	1.75	0,2-15	

Note: DMG-Dimethylglyoxime, ECTSC-N-ethyl-3-carbazolecarboxaldehyde-3-thiosemicarbazone, MCCTSC -7-methyl-2-chloroquinoline-3-carbaldehyde thiosemicarbazone, TKCHG-Thiazol-2-carbaldehyde-2-quinolyldiazine, PPTSC-pyridoxal-4-phenyl-3-thiosemicarbazone, HBBPH-4-hydroxybenzaldehyde-4-bromophenylhydrazine, BPHMP - 1-(5-bromo-2-pyridylazo)-2-hydroxy-4-mercaptophenol, IPHMP - 1-(5-iodine-2-pyridylazo)-2-hydroxy-4-mercaptophenol, AP - 2,6-bis(N,N-dimethylaminomethyl)-4-methylphenol.

Influence of foreign ions. To assess the applicability of the extracts of the complexes for the separation and determination of Ni(II), the interfering effect of foreign ions was studied (Table 5). The determination of Ni(II) with L is not interfered with by ions of alkaline, alkaline earth elements, and REE. The interfering effect of ions was eliminated by changing the pH of the medium with the help of masking agents and by using extraction. The interfering effect of

Nb(V), Ta(V), Ti(IV) was eliminated with an increase in pH and assisted by fluoride ion. Interfering influence of Ti(IV) - ascorbic acid, Cu(II) - thiourea, and Mo(VI) and Nb(V) - oxalate ion. When using a 0.01 M EDTA solution, Ti(IV), V(IV), Nb(V), Ta(V), Mo(VI) do not interfere with the determination. In an ammonium acetate buffer, Mn^{2+} binds more strongly to EDTA than to GAMF, which is used to mask it in the determination of Ni.

Table 5. Influence of foreign ions on the determination of nickel with L. ($n=6$, $P=0.95$, $30 \mu\text{g}$ of Ni(II) was taken)

Ion	Molar excess of ion	Masking reagent	Found, μg (S_r)
Co(II)	50		30.0 (0.021)
Fe(II)	200		29.8 (0.037)
Cd(II)	200		29.6 (0.048)
Al(III)	180		30.0 (0.045)
Fe(III)	60	Oxalic acid	30.2 (0.046)
Zr(IV)	50		29.8 (0.033)
W(VI)	25	Oxalic acid	29.6 (0.053)
Hg(II)	40		30.2 (0.031)
Ti(IV)	30	Tyrone	29.6 (0.027)
V(IV)	20	Tyrone	29.6 (0.052)
Mo(VI)	10	Sodium fluoride	30.4 (0.055)
Cr(III)	120		29.8 (0.041)
Nb(V)	50	Sodium fluoride	30.1 (0.057)
Ta(V)	50	Sodium fluoride	30.1 (0.038)
UO_2^{2+}	50		29.2 (0.040)

Analytical Applications

The proposed method, under already established optimal conditions, was applied to determine Ni(II) in wastewater, bottom sediments, as well as in oil and oil products.

Determination of nickel (II) in water (Table 4). For analysis, we took 1 liter of waste water, evaporated until a precipitate was obtained, without bringing to a boil. The precipitate was dissolved in 5 ml of HNO_3 , transferred to a flask ca. 50 ml and diluted with water to the mark. An aliquot of the resulting solution was taken, transferred to a separatory funnel, adjusted to pH 2.8–6.0, and 2.0–2.5 ml of 0.01 M L was added. The volume of the organic phase was adjusted to 5 ml with chloroform, and the total volume was adjusted to 25 ml with distilled water. The mixture was shaken for 5 min. After phase separation, the light absorption of the extracts was measured with a KFK-2 at 540 nm in a cuvette with a

thickness of 0.5 cm. The content of Ni(II) was found from a calibration curve.

Determination of nickel(II) in oil and oil products Baku (Table 4). For analysis, 40 gr of the test fuel was placed in a porcelain cup and burned in accordance with GOST 1461 - 75. Then the porcelain cup with ash was placed in a muffle at a temperature of $550 \pm 20^\circ \text{C}$ and kept at a temperature of 1 hour. After cooling, 5 ml of HCl (1:1) was added to the dish, the mixture was boiled to dryness, and 0.5 g of anhydrous Na_2CO_3 was added to it. Then the cup was placed for 2–3 min in a muffle heated to 800°C . After cooling, the alloy in the cup was dissolved in distilled water, filtered into a flask with a cap. 50 ml and diluted with water to the mark. An aliquot of the resulting solution was taken and the content of Ni(II) was determined as indicated in the case of determination in water.

Table 6. Results of determination of nickel(II) in sewage, bottom sediments, oil and oil products of Baku ($n = 6$, $P = 0.95$)

Object of analysis		Introduced mg/L	Found, mg/kg		Relative standard deviation
			With additive	$\bar{x} \pm \frac{t_p \cdot S}{\sqrt{n}}$	
Waste water	Sample 1	3.0	3.36	0.36 ± 0.09	0.07
	Sample 2	6.0	7.28	1.28 ± 0.05	0.04

Bottom sediments	Sample 1	4	5.85	1.85±0.13	0.07
	Sample 2	5	6.73	1.73±0.17	0.04
Oil		10	16.19	(6.17±0.14)·10 ⁻⁵	0.08
Fuel oil		10	12.68	(2.68±0.22)·10 ⁻³	0.03
Hydron		10	14.32	(4.32±0.17)·10 ⁻³	0.07

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NİKELİN(II) 2,4-TIAZOLIDİNDİON VƏ ONUN TÖRƏMƏLƏRİ İLƏ KOMPLEKSƏMƏLƏGƏLMƏ REAKSİYASININ TƏDQIQI

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Xülasə: Nikelin 2,4-tiazolidindion (L) və onun törəmələri {5-(benziliden)-2,4-tiazolidindion (L₁), 5-(2-xlorbenziliden)-2,4-tiazolidindion (L₂), 5-(2-hidroksibenziliden)-2,4-tiazolidindion (L₃), 5-(4-hidroksibenziliden)-2,4-tiazolidindion (L₄), 5-(2-brom-5-metoksibenziliden)-tiazolidin-2,4-dion

(L₅) ilə kompleksmələgəlmə reaksiyası spektrofotometrik metodla tədqiq edilmişdir. Xloroform ilə bir dəfəyə 97.8-98.4% nikel ekstraksiya olunur. Kompleks pH 2.3-8.0 intervalında əmələ gəlir. Alınmış kompleks birləşmələr şəkilində nikelin ekstraksiyası üçün optimal şərait reagentin (2.0-2.5) × 10⁻⁴ M qatılığıdır. Ni-L kompleksləri sulu və üzvi həlledicilərdə üç gün ərzində parçalanmır və ekstraksiyadan sonra bir aydan çox optiki sıxlıq sabit qalır. Komplekslərin maksimum optik sıxlığı 5 dəqiqə ərzində yaranır. Nikelin L ilə komplekslərinin maksimum analitik siqnalı 470-482 nm-də müşahidə olunur. Molyar udma əmsalları (1.37-1.61)×10⁴-dür. Nikelin kompleks əmələ gətirən ion forması Ni²⁺-dir. Bir L molekulundan sıxışdırılıb çıxarılan protonların sayı 1 olmuşdur. Ni-L kompleksləri üzvi fazada polimerləşmiş, monomer formadadır (γ=1.01-1.07). Təklif olunan üsul artıq müəyyən edilmiş optimal şəraitdə tullantı və qrunt sularında, o cümlədən neft və neft məhsullarında Ni(II)-nin təyini üçün tətbiq edilmişdir.

Açar sözlər: nikel, 2,4-tiazolidindion, xloroform, neft, neft məhsulu.

ИЗУЧЕНИЕ РЕАКЦИИ КОМПЛЕКСООБРАЗОВАНИЯ НИКЕЛЯ (II) С 2,4-ТИАЗОЛИДИНДИОНОМ И ЕГО ПРОИЗВОДНЫМИ

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Аннотация: Спектрофотометрическим методом изучена реакция комплексообразования никеля с 2,4-тиазолидиндион (L) и его производными - {5-(бензилиден)-2,4-тиазолидиндион (L₁), 5-(2-хлорбензилиден)-2,4-тиазолидиндион (L₂), 5-(2-гидроксибензилиден)-2,4-тиазолидиндион (L₃), 5-(4-гидроксибензилиден)-2,4-тиазолидиндион (L₄), 5-(2-бром-5-метоксибензилиден)-тиазолидин-2,4-дион (L₅)}. При однократной экстракции хлороформом извлекается 97.8-98.4 % никеля. Изучение зависимости комплексообразования от pH показало, что оптимальный интервал кислотности, при котором оптическая плотность максимальна и постоянна, находится при pH 2.3-8.0. Оптимальным условием образования и экстракции комплексов никеля этих соединений является (2.0-2.5)×10⁻⁴ M концентрация L. Комплексы никеля с L устойчивы в водных и органических растворителях и не разлагаются в течение трех суток, а после экстракции - больше месяца. Максимальная оптическая плотность достигается в течение 5 минут. Максимальный аналитический сигнал при комплексообразовании никеля с L наблюдается при 470-482 нм. Молярные коэффициенты поглощения составляют (1.37-1.61)×10⁴. Комплексообразующей формой никеля является Ni²⁺. При этом число протонов, вытесняемых им из одной молекулы L, оказалось равным 1. Комплексы никеля с L в органической фазе не полимеризуются и находятся в мономерной форме (γ=1.01-1.07). Предложенный метод при уже установленных оптимальных условиях применен для определения Ni(II) в сточных водах, донных отложениях, а также в нефти и нефтепродуктах.

Ключевые слова: никель, 2,4-тиазолидиндион, хлороформ, нефть, нефтепродукт.