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RESEARCH INTO COMPLEX FORMATION OF COBALT(II) AND NICKEL (II) WITH 2-HYDROXY-5-NITROTHIOPHENOL AND DIPHENYLGUANIDINE

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The interaction of cobalt (II) and nickel (II) with 2-hydroxy-5-nitrothiophenol (HNTPh) and diphenylguanidine (DPG) produces chloroform-extracted mixed-ligand complexes (MLC) with the ratio Me (II): GNTF: DPG = 1: 2: 2. Conditions for extraction-photometric determination of cobalt (II) nickel (II) are revealed. It found that MLC are formed in a weakly acid medium (pH_{opt} = 4.4 ÷ 7.8). Note that all studies were performed with $CHCl_3$ (extraction ratio R = 98.3-98.6%). To generate and extract MLC, (1.2-2.0) × 10-3 M HNTPh and (2.0-2.5) × 10-3 M DPG are required. Maximum in the absorption spectrum for cobalt (II) nickel (II) MLC is observed at 560 nm and 480 nm, respectively. The following chemical analytical parameters were calculated: extraction constants (Kex = 10.56-11.64), stability constants (β = 8.59-9.62), distribution constants (K_D = 1.86-1.92), coefficients extraction (R = 98.3-98.6%) with molar coefficient of light absorption being equal to (2.64 - 3.30) × 104.

Key words: *cobalt, nickel, 2-hydroxy-5-nitrothiophenol, diphenylguanidin, chlorophorm*

INTRODUCTION

Many metal ions form colored binary particles with hydroxythiophenols, such as 2-hydroxy-5- halogenthiophenols (HTPh) and 2,6-dithophenols (DTPh) [1,2]. Besides, the color and extraction properties of these species can be improved by adding auxiliary compounds [3-4]. The following ion-associated reagents were used for liquid liquids extraction of $[X(HTPh)_2]^{z-}$ or $[X(DTPh)_2]^{z-}$ (z = 2 or 3; X-metal): Aromatic Amines{aniline, N-methylaniline, N,N-dimethylaniline, *o*-toluidine, *m*-toluidine, *p*-toluidine, 3,4-xylidine, 2,4-xylidine, and 2,5-xylidine} [5-14], diphenylguanidine and triphenylguanidine [15], diphenylamine and triphenylamine [16]. The previous article dealt with the formation and extraction of the complex in a system containing Co (II) and Ni (II), 2-hydroxy-5-iodothiophenol (HITP), diphenylguanidine (DPG) and triphenylguanidine (TPG) [17].

Below-cited are results of systematic research into a similar extraction chromogenic system which involves 2-hydroxy-5-nitrothiophenol (HNTPh) in place of HITP.

MATERIALS AND METHODS

Reagents and instruments. The initial solution of Ni (II) and Co (II) was prepared through dissolving $NiCl_2 \times 6H_2O$ and $Co(NO_3)_2 \times 6H_2O$, respectively, in distilled water. Process solutions of Me (II) (0.1 mgL^{-1}) were prepared by appropriate dilution of the stock solution. The concentration of cobalt and nickel solutions was adjusted gravimetrically [18].

HTPh was synthesized according to the procedure [19]. Chloroform solutions of HTPh (0.01 mol L^{-1}) and DPG (0.025 mol L^{-1}) were used. To create the optimum acidity, 0.1 mol L^{-1} solutions of HCl, NaOH, or ammonium acetate {CH_3COOH+CH_3COONH_4 (pH 4-12)} buffers were applied.

The absorbance of the extracts was measured through the use of a SF 26 spectrophotometer and KFK 2 photo-colorimeter. Glass cells with optical paths of 5 to 10 mm were used. pH of aqueous phase was measured using an I-120.2 potentiometer with a glass electrode.

RESULTS AND DISCUSSION

At pH 5-6, the cobalt and nickel complex {Me- HNTPh –DPG (Me= Co or Ni)} is not extracted with the help of inert organic solvents. To determine signs of complex charge, ion exchange chromatography was used: AV17 anionexchanger in chloroform absorbs a part of solution; the chromatographic column is coloured in orange while Ni (II) and Co (II) are not detected in the filtrate. The anion complex is extracted in the presence of a hydrophobic amine, wherein the complex stability increases, and the colour becomes more saturated. To neutralize the charge of the anion complex, diphenylguanidine is used as a hydrophobic amine which transforms into a diphenylguadininic ion in the acidic medium, that is,

$$\underset{\text{(DPG)}}{(C_6H_5NH)_2C{=}NH + H^+} \Longleftrightarrow \underset{\text{(DPGH+)}}{(C_6H_5NH)_2C{=}NH_2^+}$$

Influence of pH of the aqueous phase. The spectra of mixed ligand complexes (MLC) Me (II)-HNTPh-DPG extracted in chloroform are shown in Fig.1. The light absorption spectra of Me (II) complexes with HNTPh and DPG were studied in a wide range of medium pH. Complexes of cobalt and nickel are formed and extracted at pH 2.2-8.4 and 2.5-10.8, respectively. It could be seen that Co(II)- HNTPh - DPG species are extracted to a greater extent at pH values in the range of 5.5–7.8. Ni(II) complexes are extracted at lower pH: 4.4–6.7.

Choice of Extractant. To extract the complexes, $CHCl_3$, CCl_4, C_6H_5Cl, C_6H_6, $C_6H_5CH_3$, $C_6H_4(CH_3)_2$, *iso*-C_4H_9OH, C_4H_9OH, *iso*- $C_5H_{11}OH$, $C_2H_4Cl_2$ were used. The extractability of the complexes was evaluated by the distribution coefficient and the degree of extraction. The best extractants were $CHCl_3$, $C_2H_4Cl_2$ and C_6H_5Cl. All further studies were carried out with $CHCl_3$ (extraction ratio (R,%) R=98.3-98.6%). The concentration of cobalt and nickel in the organic phase was determined using 2-nitroso-1-naphthol and dimethylglyoxime [20] respectively, through the photometric measurements after the back extraction, and the aqueous phase was determined by the difference.

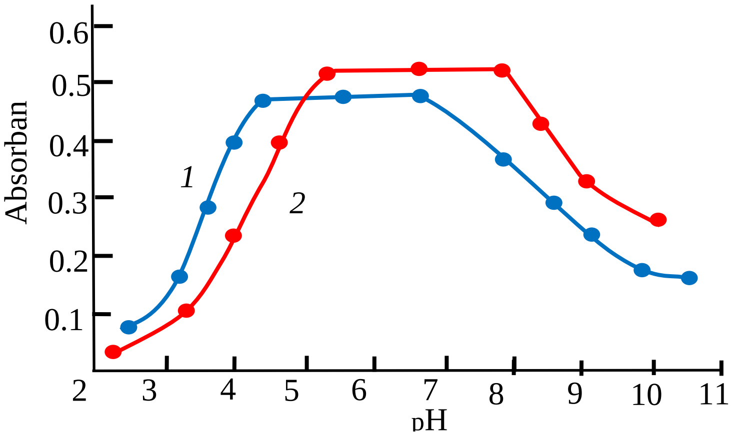


Fig. 1. Absorbance of mixed ligand complexes as a function of the pH of the aqueous phase: Ni- HNTPh-DPG (curve 1) and Co-HNTPh-DPG (curve 2). $C_{Co(II)} = 1.7\times10^{-5}$ mol L^{-1}, C_{Ni}=3.5 × 10^{-5} mol L^{-1}, C_{HNTPh} = (1.2 - 2.0)×10^{-3} mol L^{-1}, C_{DPG} = (2.0 - 2.5) ×10^{-3} mol L^{-1}, λ = 490 and 590 nm.

Electronic Absorption Spectra. The spectra of the MLC of Me (II)-HNTPh-DPG extracted in chloroform are shown in Fig.2. Co-HITP-DPG cobalt complex absorption is higher than that of the Ni-HITP-DPG nickel complex. The maximum values are recorded at 560 nm for Co (II) compounds and at 480 nm for Ni (II) compounds.

All colour reactions were very contrast, since the initial solutions are colourless (λ_{max} (HNTPh) = 284 nm). Thus, the bathochromic shift is 276-196 nm. The contrast of the reactions was high, i.e. initial reagents are colourless, and the complexes are intensely coloured.

Effect of reagent concentration. The effect of shaking time. The optimum concentrations of HNTPh and DPG are shown in Table 1. For the formation and extraction of MLC, (1.2-2.0) × 10^{-3} M HNTPh and (2.0-2.5) × 10^{-3} M DPG are required.

MLC II (II) with HNTPh and DPG are stable in aqueous and organic solvents and do not decompose for two days or for a month after extraction. The effect of shaking time in the course of extraction is shown in Fig. 3. Extracts of Ni-system (curve *1*) and Co-system (curve *2*) are produced within approximately 2 minutes and 4 minutes respectively. A longer shaking time does not affect absorption.

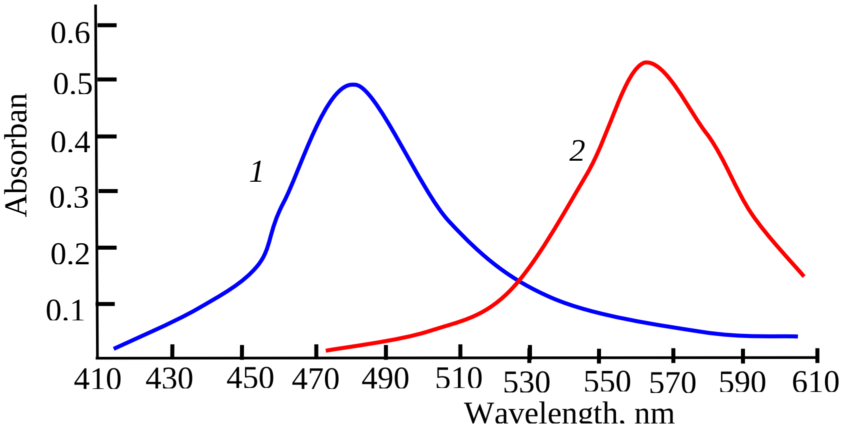


Fig. 2. Absorption spectra of the complexes: Ni-HNTPh-DPG (curve 1) and Co-HNTPh-DPG (curve 2). $C_{Co(II)}$ = 1.7×10^{-5} mol L^{-1}, $C_{Ni(II)}$ =3.5 × 10^{-5} mol L^{-1}, C_{HNTPh} = (1.2 - 2.0)×10^{-3} mol L^{-1}, C_{DPG} = (2.0 - 2.5) ×10^{-3} mol L^{-1}, pH 4.5-6.8 and pH 5.5-7.9, λ = 490 and 590 nm.

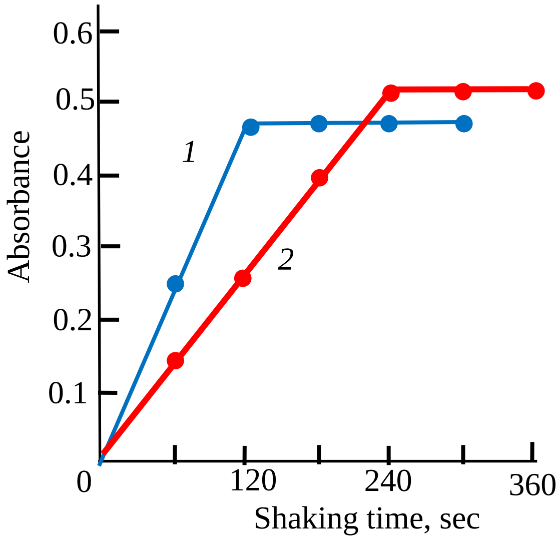


Fig. 3. Effect of shaking time on the absorbance: Ni- HNTPh-DPG (curve 1) and Co- HNTPh-DPG (curve 2). $C_{Co(II)} = 1.7\times10^{-5}$mol L^{-1}, $C_{Ni(II)}$ =3.5 × 10^{-5} mol L^{-1}, C_{HNTPh} = (1.2 - 2.0)×10^{-3} mol L^{-1}, C_{DPG} = (2.0 - 2.5) ×10^{-3} mol L^{-1}, pH 4.5-6.8 and pH 5.5-7.9, λ = 490 and 590 nm.

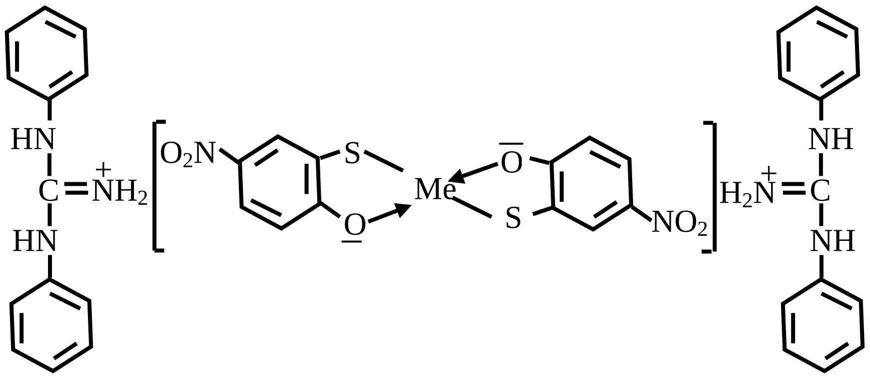
Table 1. Optimum operating conditions

Extraction system	Extraction time, s	pH	C_{HITP}, mol L^{-1}	C_{Am}, mol L^{-1}	λ_{max}, nm	$\Delta\lambda_{max}$, nm
Ni(II)- HNTPh-DPG-H_2O-$CHCl_3$	120	4.5-6.8	1.2×10^{-3}	2.0 × 10^{-3}	480	196
Co(II)- HNTPh-DPG-H_2O-$CHCl_3$	240	5.5-7.9	2.0×10^{-3}	2.5×10^{-3}	560	276

Composition of complexes and suggested formulae. The molar ratios between the components of the MLC were found by several methods: relative yield method, straight line method and equilibrium shift method [21]. The results suggest the complex composition of 1:2:2 (Me : HNTPh : DPG).

The results obtained, some conclusions [3-5] and our previous experience with identical systems suggest that Co (II) was oxidised to Co (III) by the atmospheric oxygen during the complex formation. İn acidic medium, HNTPh exhibit reducing properties. Hence, we are inclined to suggest the following formula of the ternary compound: $[Me(HNTPh)_2](DPGH)_2$; in the formula DPG is in its protonated form ($DPGH^+$).

The existence of clearly defined absorption bands at 2410 - 2415 cm^{-1} in the IR-spectrum of the complex indicates the coordination of the DPG in the protonated form [22]. The disappearance of the band at 2580 cm^{-1}, characteristic for the spectrum of HNTPh, and the appearance of corresponding bands in the spectrum of the complex which are shifted toward lower frequency, suggests that sulphur atoms are involved in complex formation. The observed decrease in the intensity of the absorption bands at 3200-3600 cm^{-1} with a maximum at 3460 cm^{-1} and the appearance of a broad band in the region of 3050-3150 cm^{-1} shows the involvement of hydroxyl group in the formation of a coordination bond. Proceeding from the obtained data, we propose the following structure for the extracted ternary complex:



Equilibrium constants. Several processes should be taken into account in the system of $[Me(HNTPh)_2]^{2-}$, $(DPGH^+)_2$, water and chloroform:

1) Association in the aqueous phase between anionic chelate, $[Me(HNTPh)_2]^{2-}$, and the hydrophobic amines cation, $(DPGH^+)_2$, with the equilibrium constants

$$\beta = \frac{[M(HITP)_2](AmH)}{[M(HITP)_2]^{2-} \times [(AmH^+)_2]}$$

2) Distribution of the complexes between the aqueous and the organic phase

$$\{[Me(HNTPh)_2](\ DPGH)_2\}_{aq} \leftrightarrow \{[Me(HNTPh)_2](\ DPGH)_2\}_{org}$$

with the distribution constants

$$K_D = \frac{[Me(HNTPh)_2](DPGH)_{org}}{[[Me(HNTPh)_2](DPGH)_2]_{aq}}$$

3) Extraction of the ternary complexes from water into chloroform

$$[Me(HITP)_2]^{2-}{}_{aq} + (DPGH^+)_{2\ aq} \leftrightarrow \{[Me(HNTPh)_2](DPGH)_2\}_{org}$$

with the extraction constants

$$K_{ex} = K_D + \beta = \frac{[[Me(HNTPh)_2](DPGH)]_{org}}{[[Me(HNTPh)_2]^{2-}]_{aq} \times [(DPGH^+)_2]_{aq}}$$

The constants of the distribution K_D were determined by comparison of the absorbance values obtained after single extraction under the optimum conditions (A_1) and the triple extraction (A_3): $K_D = A_1/(A_3–A_1)$. The extraction constants were calculated by the equation $K_{ex} = \beta + K_D$. The obtained values are shown in Table 2.

Table 2. Values of the extraction constants (K_{ex}), distribution constants (K_D), association constants (β) and recoveries (R%) for the Me(II)-HNTPh-DPG-water-chloroform systems

Extraction system	lg β	lgK_D	lg K_{ex}	R%
Co(II)- HNTPh-DPG-H_2O-$CHCl_3$	8.59±0.2	1.86±0.01	10.56±0.21	98.3
Ni(II)- HNTPh-DPG-H_2O-$CHCl_3$	9.62±0.01	1.92±0.02	11.64±0.03	98.6

Influence of interfering ions. To evaluate the complex applicability for photometric determination of cobalt and nickel, we examined the influence of foreign ions and reagents. The results showed that great excesses of alkali, alkali earth, and rare earth elements, as well as NO^{3-}, ClO^{4-}, SO_4^{2-} and CH_3COO^- do not interfere in the determination of cobalt and nickel with HITP and Am. Interference of most cations is masked through adding complexone III. Tartrate has the property of masking milligram quantities of Ta, Ti, W and Mo. Zr and Cu are masked by fluoride ion and thiourea, respectively.

Beer's law and analytical characteristics. The validity of the Beer's law was checked at optimum conditions (Table 1). Equations of the obtained straight lines and some important characteristics concerning the application of the ternary complexes for extractive-spectrophotometric determination of Co(II) and Ni(II) are listed in Table 3.

Table 3. Analytical characteristics

Characteristic	Co- HNTPh-DPG	Ni- HNTPh-DPG
Apparent molar absorptivity (ε), L mol^{-1} cm^{-1}	3.30×10^4	2.64×10^4
Adherence to Beer's law, μg mL^{-1}	0.8-20	0.7-18
Equation of straight line	0.1252X+0.054	0.149x+0.02
Coefficient of correlation	0.9993	0.9981
Sandell's sensitivity, ng cm^{-2}	0.00746	0.00745
Limit of detection (LOD), μg mL^{-1}	14	11
Limit of quantification (LOQ), μg mL^{-1}	45	36

Determination of nickel in magnesium and carnallite. The developed analytical procedure was used to analyze real samples. Two replicates of metallic magnesium and two samples of carnallite were subjected to analysis. Results are shown in Table 4; their reliability was verified by the added–recovered method.

Dissolution of magnesium. A 0.5-0.6 g sample of magnesium was carefully dissolved in 10 mL of HNO_3 (1:2) and the solution was evaporated to moist salts on an electric heater. After cooling, 3 mL of water were added to dissolve the salts and pH was adjusted to *ca*. 6 with NaOH. The solution was quantitatively transferred into a 25 mL calibrated flask; then distilled water was added to the mark.

Dissolution of carnallite. A *ca.* 2.0 g sample of carnallite was placed in a 50-mL beaker. 0.5 mL of a 3% NaF solution and 5 mL of HNO_3 (1:20) were added and the beaker was heated on an electric heater. After cooling, the obtained solution was neutralised with ammonia to pH 6 (universal paper indicator); then it was transferred through a filter paper into a 50 mL calibrated flask and diluted to the mark with distilled water.

Table 4. Determination of cobalt and nickel in metallic magnesium and carnallite

Sample	Found (%) $\times10^{-4}$		Added-recovered method					
	Ni	Co	Added (%) $\times10^{-3}$		Recovered (%) $\times10^{-3}$		RSD (%)	
			Ni	Co	Ni	Co	Ni	Co
1^a	7.65±0.01	5.12±0.03	1	1	1.765	6.123	1.4	1.3
2^a	7.64±0.02	5.09±0.01	1	1	1.764	6.094	1.5	1.2
1^b	8.71±0.01	5.81±0.04	1	1	1.871	6.812	1.3	1.4
2^b	8.69±0.02	5.35±0.02	1	1	1.869	6.351	1.8	1.5

Note: ^{a} – Metallic magnesium; *n*=6; P=95%; ^{b} – Carnallite; *n*=5; P=95%

CONCLUSIONS

1. Cobalt (II) nickel (II) form chloroform extractable complexes of mixed ligands with HNTPh and DPG. The anionic part of these complexes $[Me\ (HNTPh)_2]^{2-}$ provides intensive coloring (for the nickel complex λ_{max} = 480 nm and for the cobalt complex λ_{max} = 560 nm) and the bulk of the cationic part

($DPGH^+$), in turn, guarantees poor solubility in water.

2. The following equilibrium constants and analytical parameters are calculated: extraction constants (K_{ex} = 10.56-11.64), association constants (β=8.59-9.62), distribution constants (K_D =1.86-1.92), recovery factors (R=98.3-98.6%), molar absorptive capacities (ε=(2.64-3.30) ×10^4), Sendell sensitivity (SS= 0.00745-0.00746 ng cm^{-2}), detection limits (LOD = 11-14 μg mL^{-1}) and limits of quantitative evaluation (LOQ= 36-45 μg mL^{-1}). Analogous values of some above-mentioned characteristics obtained through the use of independent methods go to show the correctness of the experiments performed.

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ИЗУЧЕНИЕ РЕАКЦИИ КОМПЛЕКСООБРАЗОВАНИЯ КОБАЛЬТА (II) И НИКЕЛЯ (II) С 2-ГИДРОКСИ-5-НИТРОТИОФЕНОЛОМ И ДИФЕНИЛГУАНИДИНОМ

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При взаимодействии кобальта (II) и никеля (II) с 2-гидрокси-5-нитротиофенолом (ГНТФ) и дифенилгуанидином (ДФГ) образуются экстрагируемые хлороформом разнолигандные комплексы (РЛК) с соотношением Ме(II):ГНТФ:ДФГ=1:2:2. Найдены условия экстракционно-фотометрического определения кобальта (II) и никеля (II). Установлено, что РЛК образуются в слабокислой среде ($pH_{опт.}$4.4-7.8). Все исследования проводили с помощью $CHCl_3$. Для образования и экстракции РЛК требуется $(1.2\text{-}2.0)\times10^{-3}$ М ГНТФ и $(2.0\text{-}2.5)\times10^{-3}$ М ДФГ. Максимум в спектре светопоглощения для РЛК кобальта (II) и никеля (II) наблюдается при 560 нм и 480 нм соответственно. Рассчитаны следующие химико-аналитические параметры: константы извлечения ($K_{эх}$ = 10.56-11.64), константы устойчивости (β = 8.59-9.62), константы распределения (K_D = 1.86-1.92), коэффициенты извлечения (R = 98.3-98.6%), молярный коэффициент светопоглощения равен $(2.64 \div 3.30)\times 10^4$.

Ключевые слова: *кобальт, никель, 2-гидрокси-5-нитротиофенол, хлороформ*

KOBALT (II) VƏ NİKELİN (II) 2-HİDROKSİ-5-NİTROTİOFENOL VƏ DİFENİLQUANİDİNLƏ KOMPLEKSƏMƏLƏGƏLMƏ REAKSİYASININ TƏDQİQİ

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Kobalt (II) və nikel (II) 2-hidroksi-5-nitrotiofenol (HNTF) və difenilquanidin (DFQ) ilə qarşılıqlı təsirdə olaraq xloroformla yaxşı ekstraksiya olunan müxtəlif liqandlı kompleks birləşmə (MLK) əmələ gətirir (Me(II):HNTF:DFQ =1:2:2). Kobalt (II) və nikelin (II) ekstraksiya-fotometrik təyininin optimal şəraiti müəyyən edilmişdir. MLK zəif turş mühitdə əmələ gəlir ($pH_{opt.}$ 4.4- 7.8). Ən yaxşı ekstragent kimi $CHCl_3$ müəyyən edilmişdir. MLK əmələ gəlməsi və ekstraksiyası üçün (1.2-2.0)×10^{-3} M HNTF və (2.0-2.5)×10^{-3} M DFQ tələb olunur. Kobalt (II) və nikelin (II) MLK müvafiq olaraq 560 və 480 nm-də maksimum işıq udur. Bəzi kimyəvi- analitik sabitlər hesablanmışdır: tarazlıq sabiti (K_{ex} = 10.56-11,64) davamlılıq sabiti (β = 8.59-9.62), paylanma sabiti (K_D = 1.86-1.92) ekstraksiya dərəcəsi (R = 98.3-98.6%), molyar işıqudma əmsalı (ε = (2.64-3.30) ×10^4.

Açar sözlər: *kobalt, nikel, 2-hidroksi-5-nitrotiofenol, difenilquanidin, xloroform*