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Study of oil emulsion and oil dispersion properties of quaternary ammonium salts and their use as analytical reagents

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ABSTRACT

The oil-collecting and oil-dispersing properties of a quaternary ammonium salt (QAS) formed by octadecane and cis-9-octadecenoic acid with triethanolamine, as well as its use as an analytical reagent for the extraction-photometric determination of nickel in the form of a mixed-ligand complex (MLC) with 2-hydroxy-5-chlorothiophenol (L) and QAS in water were studied. The maximum absorption of MLC Ni(II)-L-QAS is observed at λ = 500-510 nm (pHop. 3.5–5.9). The molar absorption coefficients range from $(2.58\text{-}2.71)\times10^4$. Beer's law is followed within the range of 1.0–90 μg ml–1 of Ni(II). The methods we proposed were applied under already established optimal conditions for the determination of nickel in various objects: in wastewater and bottom sediments of metallurgical plants, in samples of natural soils, and also in samples of food products.

1. Introduction

In recent years, increasing attention has been directed toward the application of natural polymers in fields such as biomedicine, pharmaceuticals, and dermatology, owing to their biocompatibility, biodegradability, and functional versatility. Among these, polysaccharides and proteins have emerged as dominant candidates for biomedical use, as they are inherently non-toxic to human tissues. In particular, proteins such as sericin have demonstrated the ability to support skin regeneration and healing, making them highly attractive for therapeutic applications [1, 2]. The paper investigates the process of obtaining dialdehyde carboxymethyl cellulose (DCMC) with high molecular weight and aldehyde content by periodate oxidation under microwave irradiation. The effect of periodate oxidation time, sodium periodate concentration and pH value of the solution during microwave treatment on the molecular weight, aldehyde group content and DCMC yield was investigated. The obtained samples were analyzed using various methods, including chemical analysis, FTIR spectroscopy, thermogravimetric analysis, AFM and NMR [1].

Environmental pollution has become a major global challenge, mainly resulting from industrial, agricultural, pharmaceutical, and transportation activities that release toxic substances into water and soil. These pollutants — including organic compounds, pharmaceutical residues, heavy metals, and petroleum derivatives — seriously affect human health and ecosystems. Among various remediation methods, adsorption is one of the most efficient and eco-friendly techniques, especially when using bio-based and biodegradable materials. Thus, developing environmentally compatible adsorbents and dispersants remains a key focus in green chemistry research [3–6].

With the continuous growth of industry, pollution of the biosphere is constantly increasing with the development of industry. Technologies implemented in many of its branches are associated with the formation of wastewater containing Zn, Pb, Cd, Ni, Cr and other metals. A significant amount of these metals enters water bodies with wastewater from non-ferrous metallurgy, especially galvanic shops. Motor transport is also a powerful source of environmental pollution [7].

As with other water basins in the world, oil pollution of the Caspian Sea is a serious environmental problem caused by both oil production and transportation and the discharge of industrial and domestic waste. This has a

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negative impact on the flora and fauna of the sea, as well as on the quality of water and bottom sediments [8].

Oil-based substances that reflect sunlight prevent the absorption of energy by water. Removal of such stains is especially necessary for the vital activity of marine inhabitants, since more than one hundred species of fish, 95% of the world population of sturgeon, live in the Caspian Sea [9]. Surface-active substances used to remove thin layers of oil from the surface of the water are divided into oil dispersants and oil skimmers [10-15]. The concentration of such hazardous substances as chromium, iron, aluminum, manganese, etc., should be below the established standard values [16-19]. The development of modern industrial society creates the need for prompt and reliable control of the content of heavy metals that have toxic properties and inevitably enter the environment. The content of nickel is one of the main indicators in the chemical analysis of natural water. At the same time, an increased concentration of nickel is one of the main reasons for the unpleasant taste of water and the negative impact on human health.

Nickel (II) has chromoform properties and gives color reactions with many reagents. According to the complexing properties of Ni (II), mainly Fe (II), Co (II), Cu (II) and platinum metals are similar. Many chelating reagents containing donor N, O or S atoms are suitable for the photometric determination of it. Dioxymes, oxyazo compounds, and thiocarboxylic acids are most often used [20, 21]. The most important photometric methods for the determination of nickel (II) are reactions with dioxins and dithizones [22]. Complexation of Ni (II) with α-ketoglutaric acid [23], 2-[(2-mercaptophenylimino)methyl]phenol [24], 1-azobenzene-3-(3-hydroxyl-2-pyridyl) triazene [25], azo derivatives of chromotropic acid -2- (2-hydroxy-3-sulfo-5-nitrophenyllazo) naphthalene-1,8-dihydroxy-3,6-disulfonatriya and 2- (2-hydroxy-3,5-disulfophenyllazo) naphthalene-1,8-dihydroxy-3,6-disulfonatriya [26], di-2-pyridyl ketone with benzoylhyddisulfide [27], 1-nitroso-2-naphthol-3,6-disulfonic acid [28], with 1,3-bis (3,5-dimethylpyrazol1yl) propane [29], an extraction-spectrophotometric technique is proposed eniya with pyridoxal-4-phenyl-3-thiosemicarbazone [30]. Three-component complexes of Ni(II) with 1,10-phenanthroline and thi-palsyllic acid [31], with 2,2-dipyridyl and dinitrobenzenazosalicylic acid [32], with 2-phenyl4,6-diamino-1, 3,5-triazine and 8-hydroxyquinoline [33], with the complexes of a number of carboxymethyleneeamines: iminodiacetic, 2-hydroxyethyliminodi-acetic, nitrilotiacetic acid and malonic acid [34]. The interaction of Ni (II) with 2,2,1,3,4-tetrahydroxy-3,1-sulfo-5- nitrobenzene in the presence of cationic surfactantscetyl pyridinium chloride, cetylpyridinium bromide and cetyltrimethylammonium bromide [35]. An extraction-spectrophotometric method for the determination of Ni (II) in various samples was developed [36-38].

Recently, sorption methods have become widespread for determining trace elements. Imidazolate framework structures attract special attention as effective sorbents, not only due to their high specific surface area, but also due to the possibility of obtaining them under "mild" conditions [39].

Microconcentrations of Cu^{2+}, Fe^{3+} and Zn^{2+} ions, which are close in their chemical properties to Ni2+ ions, in water and industrial alloys were determined with high accuracy using spectrophotometry [31,40-43]. Atomic absorption spectrophotometry was used to determine heavy metals (Mn, Co, Fe, K, Ni, Na and Cd) in vegetables (beans, cowpeas and broad beans) [44]. A comparative analysis of free and total inorganic ion content was carried out in thirty wine samples divided into alcoholic, non-alcoholic and wines based on fruit juices using three analytical methods: titrimetric, spectrophotometric and chromatographic [45]. A highly sensitive and selective molecularly imprinted polymer was synthesized for solid-phase extraction and concentration of trace amounts of ascorbic acid. The parameters affecting the separation and synthesis of the polymer such as solvent volume, equilibration time, selectivity and capacity of the material, time, pH, etc. were tested by ultraviolet spectroscopy. The linear range of the method was investigated and the data obtained showed linearity in the range of 0.4-9.0 mg/L [46]. A simple and selective spectrophotometric method for the determination of nickel(II) was developed using 5-(2-bromo-5-methoxybenzylidene)thiazolidine-2,4-dione and 4,5-dihydroxy-3,6-dinitrosonaphthalene-2,7-disulfonic acid as a chromogenic reagent forming a yellow-brown complex. Nickel(II) complexes are formed in the pH range of 7.1-8.9 and 67-9.2, respectively [47]. The development of coordination chemistry is associated with the development of new methods for the synthesis and study of coordination compounds, as well as the search for new ligands for obtaining compounds with desired properties.

Like the analytical reagents mentioned above [20-47], 2-hydroxy-5-chlorothiophenol has S and O donor atoms included in the –OH and –SH functional groups located in the ortho position. The presented article is devoted to the study of oil-collecting and oil-dispersing properties of quaternary ammonium salt (QAS), formed by octadecane (8-DA) and cis-9-octadecenoic acid (8 -cis-9-DA), with triethanolamine (TEA) nickel in the form of a mixed-ligand complex (MLC) with 2-hydroxy-5-chlorothiophenol (H_2L, L) and QAS in various objects.

2. Materials and Methods

2.1. Reagents and Apparatus

Preparation stock solution. The standard solution (1mg / ml) Ni (II) was prepared by dissolving in water an exact linkage $(NH_4)_2Ni(SO_4)_2 \times 6H_2O$ in water containing 2 ml conc. H_2SO_4 [39]. In the work, 0.01 M solutions of L and QAS in chloroform were used. Working solutions with a concentration of 0.1 mg/ml were obtained by diluting the stock solution. To create the required pH value, fixanal HCl (pH 1-2) and acetate-ammonia ($CH_3COOH+CH_3COONH_4$) buffer solutions (pH 3-11) were used. All reagents used were of at least analytical grade. All the substances used in the work were of analytical reagent grade (a.r.) and chemically pure (c.p.).

Apparatus. The optical density of the organic phase was measured on a KFK-2 (in Russian). Spectrophotometric studies of colored extracts were carried out on a SF-26 spectrophotometer (in Russian). The pH value of the solutions was controlled using an I-130 ion meter (Russia) with a glass electrode. IR spectra were recorded on a Bruker spectrophotometer (Germany). The 1H NMR spectra of the substances in Asetone-D_6 were recorded on a Bruker SP-300 instrument [300.13 for (1H)], with HMDS used as the internal standard. Elemental analysis for C, H, N and S atoms was performed on a Carlo Erba 1106 elemental analyzer. Chromatographic separation was carried out on a glass column (l=20 cm, d=2 cm) filled with silica gel of grade L 100/160. The eluent was a hexane-ethyl acetate mixture in a 9:1 ratio.

2.2. General procedure

Method. 0.1-0.9 ml of Ni(II) solution, 2.3 ml of 0.01 M solution L, 2.2 ml 0.01 M QAS and 2.6 ml of 1 M HCl solution were added to the test tubes. The volume of the organic phase was brought to 5 ml with CH_3Cl, the total volume was brought to 25 ml with distilled water. After 10 min, the extract was separated and the light absorption was measured at 540 nm (l = 0.5 cm) in KFK-2.

2.2.1. Determination of nickel in sewage water and bottom sediments

1L taken for analysis of waste water is evaporated to obtain a precipitate, do not boil. The precipitate was dissolved in 5 ml of HNO_3, was transferred to a 50 ml flask and diluted to the mark with water.

2.2.2. Determination of nickel in plant samples

A weighed portion of the air-dried food product (30-60 g) was dissolved in a mixture of HNO_3 and HCl acids in a 1:1 ratio. The solution was evaporated to dryness and the resulting residue was ashed at 300ºC. The ash was dissolved in 5 ml of 1 M H_2SO_4 and the volume of the solution was brought to 100 ml with distilled water. Nickel was determined in aliquot parts of the solution using the method we proposed.

2.2.3. Determination of nickel in soils

A 1.0 g sample is finely ground in an agate mortar and calcined in a muffle furnace for 4 hours. After cooling, the sample is processed and dissolved in a graphite cup in a mixture (3:1:3 = HCl:HNO_3:HF) of 15 ml conc. HCl, 5 ml conc. HNO_3 and 15 ml conc. HF at 50-70ºC to remove excess hydrogen fluoride. Another 8 ml of conc. HNO_3 are added to the solution three times and evaporated each time to 5-6 ml. After this, the solution is transferred to a 100 ml measuring flask and its volume is brought up to the mark with distilled water. Nickel is determined in aliquots of the solution using the methods we proposed.

2.2.4. General procedure for the synthesis of QAS

For the synthesis of QAS, the reaction of 8-DA and TEA in a molar ratio of 1:1 was carried out under laboratory conditions at 80-90°C (70-80°C in the case of 8 -cis-9-DA and TEA) for 1 day with intensive stirring. The reaction scheme is as follows:

$R\text{-}COOH + N(C_2H_4OH)_3 \rightarrow [R\text{-}COO^- N^+H(C_2H_4OH)_3]$ (R = $-C_{17}H_{35}$, $-C_{17}H_{33}$).

8-DA: Yield 0.89 g (68.9%). IR spectrum (KBr) ν, cm^{-1}: 3244 (-OH); 2955, 2915, 2848 (-CH); 2633, 2533 (NH); 1557, 1466 (COO-) [48]. 1H NMR (Asetone-D_6), δ, ppm: 0.89 (t., 3H, CH_3), 1.29 (24H, CH_2), 1.59 (m., 2H, CH_2CH_2COO), 2.27 (t., 2H, CH_2COO), 2.74 (t., 6H, CH_2N), 3.58 (t., 6H, CH_2OH), 4.13 (s., 4H, OH, NH). Found, %: C 63.09, H 13.17, O 20.32, N 3.42. Calculated, %: C 62.34, H 13.25, O 20.78, N 3.64.

8 -cis-9-DA: Yield 0.85 g (65.8%). IR spectrum (KBr) ν, cm^{-1}: 3245 (-OH); 2954, 2922, 2853 (-CH); 2677, 2572 (NH); 1457, 1400 (COO-) 721-665 (-CH=CH-). [36]. 1H NMR (Asetone-D_6), δ, ppm: 0.89 (t., 3H, CH_3), 1.30 (20H, CH_2), 1.58 (m., 2H, CH_2CH_2COO-), 2.09 (t., 2H, CH_2CH=), 2.24 (t., 2H, CH_2COO), 2.83 (t., 6H, CH_2N), 3.63 (t., 6H, CH_2OH), 4.60 (s., 4H, OH, NH), 5.36 (m., 2H, CH=CH). Found, %: C 63.14, H 12.70, O 20.10, N 4.06. Calculated, %:62.83, H 12.85, O 20.94, N 3.66.

The IR spectrum of 8-DA (a) 8 -cis-9-DA (b), and 1H NMR spectrum of 8 -cis-9-DA (c) is represented in Figure 1.

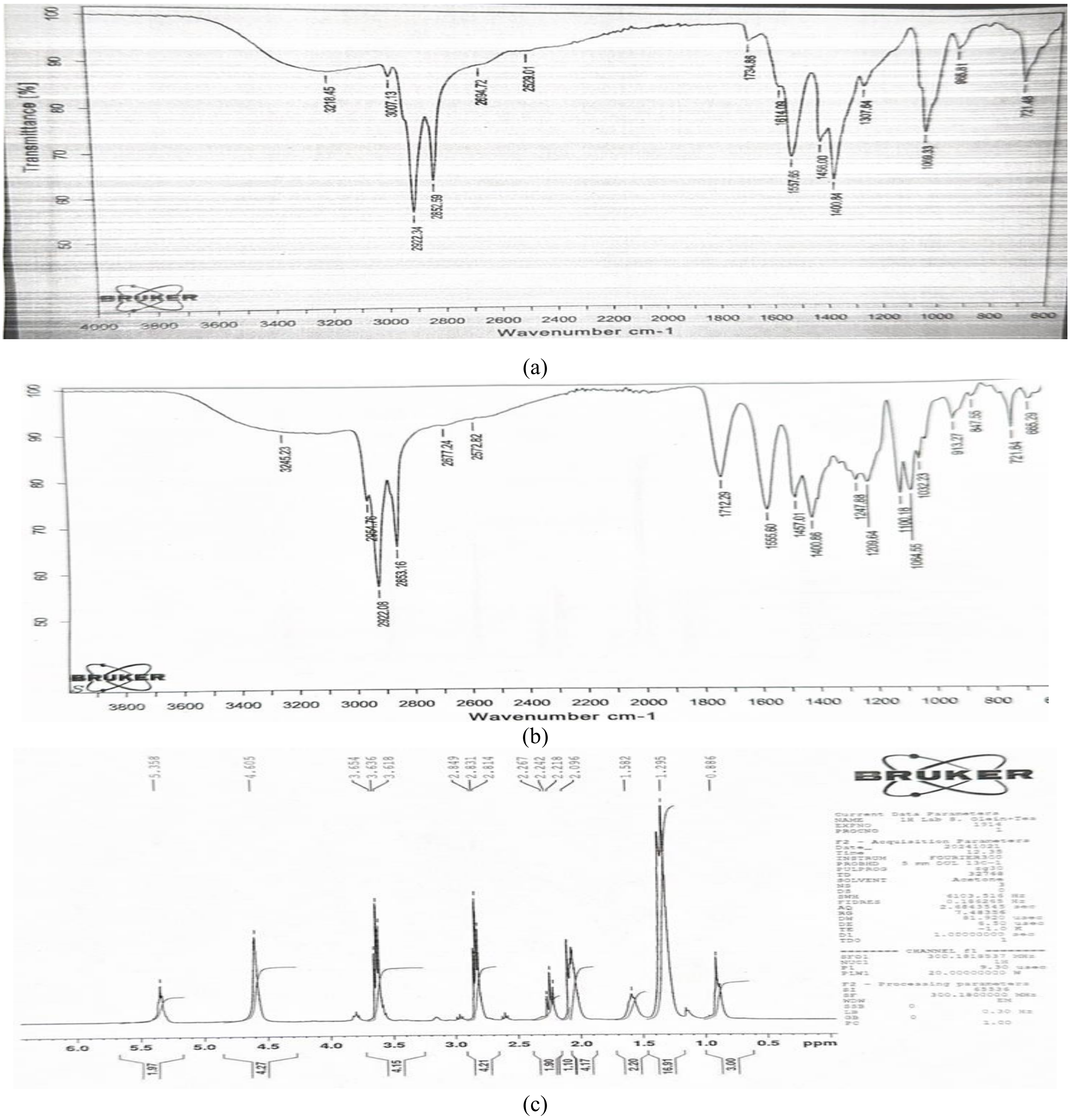
2.2.5. Study of surface activity and oilcollecting and dispersing properties of QAS

QAS is an amber-colored solid that freezes at room temperature. Its solutions with a concentration of 0.025%, 0.05%, 0.75%, 0.1% form a colloidal solution in water, and are readily soluble in ethyl and isopropyl alcohols. The surface activity of QAS solutions of various concentrations at 20±5°C was determined using a tensiometer at the water-air interface (Table 1).

As can be seen from Table 1, 8-DA exhibited high surface activity due to a decrease in surface tension from 59.8 mN·m^{-1} to 33.3 mN·m^{-1} (and for 8 -cis-9-DA from 65.6 mN·m^{-1} to 29.4 mN·m^{-1}). QAS was studied as an oil collector and oil dispersant when treating the surface of water turbid with an oil layer 0.17 nm thick [49]. The effectiveness of this reagent was studied in laboratory conditions on waters of varying degrees of mineralization using a sample of Balakhan light oil. The reagent was used both in pure form and as a 5% aqueous solution. A decrease in the area of the initial oil layer due to reagent penetration into oil-contaminated water determines its effectiveness. The oil accumulation coefficient (*K*) is a value characterizing this effect. *K* is calculated as the ratio of the initial area of the oil layer to the area of the oil slick formed under the influence of the reagent.

As can be seen from Table 2, 8-DA exhibits oil-accumulating capacity in distilled water in both forms of reagent application. K_{max} is 16.2 in undiluted reagent form and 18.6 $mN\cdot m^{-1}$ in the form of a 5% solution. The reagent exhibits oil accumulation in undiluted product form in drinking water (K_{max}=16.6), oil accumulation in the first hours in the form of a 5% solution and oil dispersibility after the 6th hour (K_D=80.0% in the case of the maximum dispersion rate).

As an oil collector-dispersant, the reagent exhibits a mixed effect in both forms of application in sea water (K_{max}=11.4, K_D=85.0%). 8 -cis-9-DA in the form of a 5% solution has an oil-accumulating capacity of K_{max}=13.6 $mN\cdot m^{-1}$. The reagent exhibits a mixed effect as an oil collector dispersant in drinking and sea water (K_{max}=7.4 and 11.4; K_D=75.0 and 78.9%, respectively). The reagents retains its effect for 4 days. From a comparison of QAS, it is clear that 8 -DA is superior in its ability to disperse oil in drinking and sea water (8 -DA: 81.7; 8 -cis-9-DA: 75.1%).



(a)

(b)

(c)

Fig. 1. IR spectrum of 8-DA (a), 8-cis-9-DA (b) and 1H NMR spectrum of 8-cis-9-DA (c).

Table 1. The value of the surface activity of QAS at the air-water interface (t=20±5°C)

ω,%		0.00025	0.0005	0.00075	0.001	0.0025	0.005	0.0075	0.01	0.025	0.05	0.075	0.1
σ, mN·m^{-1}	8-DA	59.8	52.0	48.5	46.4	41.4	38.2	36.5	35.4	33.9	33.6	33.4	33.3
	8 -cis-9-DA	65.6	55.7	50.0	46.1	36.9	33.5	32.2	31.0	30.1	29.4	29.7	29.4

Table 2. Results of studies of oil-gathering and oil-dispersing capacity of QAS

Reagent	**The state of supply of reagent to the oil surface**	**Distilled water**		**Drinking water**		**Sea water**	
		τ, hour	**K (K$_D$, %)**	**τ, hour**	**K (K$_D$, %)**	**τ, hour**	**K (K$_D$, %)**
8-DA (Balakhany oil, thickness 0.17 mm)	Undiluted product	0	7.4	0	7.4	0	6.0
		1.0-	11.6	1.0-	16.6	1.0-	8.3
		6.0	16.2	5.0	7.4	5.0	Dis.89.0%
		36.0-	13.2	24.0-	is destroyed	24.0-	Dis.79.9%
		48.0		48.0		72.0	
		72.0		60.0		86.0	
	5% aqueous dispersion	0	6.9	0	7.4	0-4.0	11.4
		1.0-	18.6	5.0-	Dis.80.0%	20.0-	Dis.85.0%
		40.0	16.2	20.0	Dis.75.4%	44.0	Dis.78.2%
		72.0		43		60-84	
8 -cis-9-DA (Ramana oil, thickness 0.17 mm)		0	8.2	0	7.4	0-3.0	11.4
		1.0-	13.6	6.0-	Dis.75.0%	36.0-	Dis.81.7%
		46.0	10.2	24.0	Dis.78.9%	48.0	Dis.75.1%
		72.0		42.0		72.0	

3. Results and Discussion

3.1. The choice of the extractant

Various organic solvents like $CHCl_3$, CCl_4, $C_2H_4Cl_2$, C_6H_5Cl, C_6H_6, $C_6H_5–CH_3$, $C_6H_4(CH_3)_2$, *iso*- C_4H_9OH, C_4H_9OH, *iso*- $C_5H_{11}OH$ and their mixes were tried for extraction. The distribution coefficient and the level of extraction were used to assess the extractability of the complexes. By extracting the complexes with chloroform, the layers quickly separated and the molar absorption coefficient reached its maximum value. After a single extraction with $CHCl_3$ at a ratio of aqueous and organic phases of 4:1, 97.6-98.5% of nickel is recovered in the form of a MLC.

The degree of extraction (*R*) of MLC was calculated by formula:

$$R = \frac{100D}{D+1}$$

where *D* is the distribution coefficient. Until the phase volume ratio reaches a 20:1, the extent of extraction remains constant. Organic solvents used for extraction of Ni(II) can be arranged on the basis of their extraction coefficient values as $CHCl_3 > CCl_4 > C_2H_4Cl_2 > C_6H_5Cl > C_6H_5–CH_3 > C_6H_6 > C_6H_4(CH_3)_2 > C_4H_9OH >$ *iso*-$C_4H_9OH >$ *iso*- $C_5H_{11}OH$. $CHCl_3$ was found to be the best extracting solvent; hence, it was selected for extraction throughout the work. The content of nickel in the organic phase was determined photo- metrically by dimethylglyoxime [20, 21] after stripping, and in the aqueous phase by the difference.

3.2. Electronic absorption spectra

The absorption maxima of the ternary Ni(II)-L QAS complexes lie in the range of 500-510 nm (Table 1). The calculated molar absorptivities (λ_{max}) belong to the interval (2.23-2.41)×10^4.

3.3. Determination of optimal conditions for complex formation

Influence of the pH of the Aqueous Phase. The effect of pH on the formation of Ni(II)- L-QAS complex was studied, in order to find a suitable pH that can be adopted in the determination of Ni(II) (Figure 2). The optimal acidity range, at which the optical density is maximum and constant, is at $pH_{opt.}$ 3.5 – 5.9 (pH 2.1 – 9.2). Up to pH 2.1, the extraction of Ni(II) is enhanced. A further increase in acidity leads to a gradual decrease in extraction, which is obviously due to a decrease in the concentration of the ionized form L. At pH > 9.2 of the solution, the extraction of MLC is practically not observed, which is apparently due to an increase in free QAS molecules and the formation of hydrolyzed forms of Ni(II).

1- Ni(II)-L-8-DA, *2*-Ni(II)-L-8 -cis-9-DA. $C_{Ni(II)} = 2.035\times10^{-5}$ M, $C_L = C_{QAS}$=1.3.0·10^{-3} M, λ=490 nm, ℓ=0.5 cm

Effect of reagent concentration. Complete extraction is achieved at reagent concentrations not lower than 1.3×10^{-3} M H_2L and (1.2-1.5)×10^{-3} M QAS.

The adherence to Beer's law was studied by measuring the absorbance value of the series of solutions containing different concentrations of the metal ion. A linear calibration graph drawn between absorbance and the metal ion concentration indicates that Ni(II) may be determined in the range 0.2-18 μg ml^{-1} (Table 3).

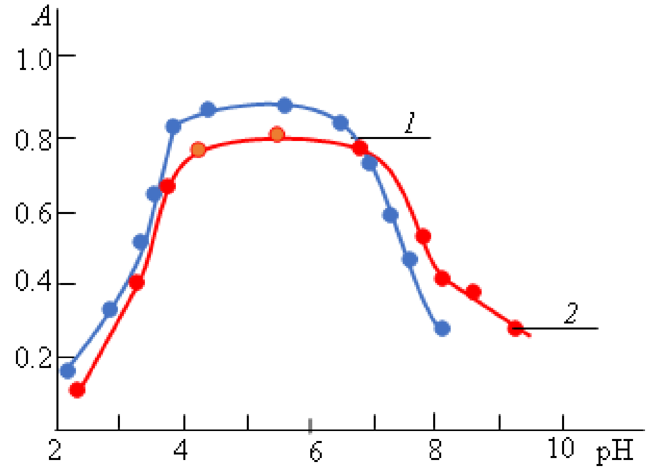


Fig. 2. Absorbance of MLC as a function of the pH of the aqueous phase.

Effect of phase volume ratios. The degree of Ni(II) extraction as MLC does not depend on the aqueous to organic phase volume ratio in a wide range (from 5:5 to 100:5), which allows for simultaneous concentration and photometry of Ni(II). Thus, a 20-fold increase in the aqueous phase volume relative to the organic phase does not affect the completeness of extraction.

Effect of time. The color appears almost immediately after adding the reagents. The optical density of the extracts is stable for at least 48 hours. The optimal shaking time is 5 minutes.

3.4. Stoichiometry of the complexes

The molar ratios of the MLC components were determined by the equilibrium shift method and the Asmus method [50]. The results show a complex composition of 1:2:2 (Ni(II):L:QAS). It has been established that Ni (II) in the complexes is present in the form of Ni^{2+} [51]. According to the experiments the complexes exist in monomeric form in the organic phase, polymerization ratios are 1.03 - 1.15 [52].

The mechanism of formation of the MLC can be represented as follows. Ni^{2+} ions, when circulating with two H_2L molecules, provide divalent anionic complexes, which are extracted by two molecules of protonated QAS (Figure 3).Complexation occurs with the displacement of one proton from one H_2L molecule. Therefore, the complexes can be considered as ionic associates between divalent anionic chelates $[Ni(HL)_2]^{2-}$ and two QAS species: $[Ni(HL)_2](QAS)_2$. The disappearance of the pronounced absorption bands in the region of 3200-3600 cm^{-1} with a maximum at 3460 cm^{-1}, observed in the spectrum of H_2L, indicates the participation of the -OH group in the formation of the complex. The observed decrease in the intensity of the absorption band in the region of 2580 cm^{-1} shows that the -SH group is involved in the formation of the coordination bond in the ionized state. The detection of the absorption band at 2380 cm^{-1} indicates the presence of protonated QAS [48]. The singlet observed in the spectrum of the H_2L ligand at 5.48 ppm, associated with the phenol group OH, disappears in the spectrum of the complex. This shows that the -OH group is involved in the complex formation. The intensity of the signal observed at 3.28 ppm, associated with the thiol group –SH decreases by a factor of two and slightly shifts to a weaker field (3.43 ppm).

Table 3. Optical characteristics, precision and accuracy of the spectrophotometric determination of Ni(II) with L and QAS

Compound	**pH$_{for}$**	**pH$_{op.}$**	**λ_{max} (nm)**	**$\varepsilon\cdot 10^{-4}$**	***R***	**C_L, M**	**C_{QAS}, M**	**V_{aq}: V_{org}**	**Working range, μg ml^{-1}**
Ni(II)-L-8-DA	2.1-8.1	3.5-5.8	500	2.58	97.6	1.3×10^{-3}	1.2×10^{-3}	100:5	0.2-18
Ni(II)-L-8 -cis-9-DA	2.2-9.2	3.6-5.9	510	2.71	98.5	1.3×10^{-3}	1.5×10^{-3}	90:5	0.25-16

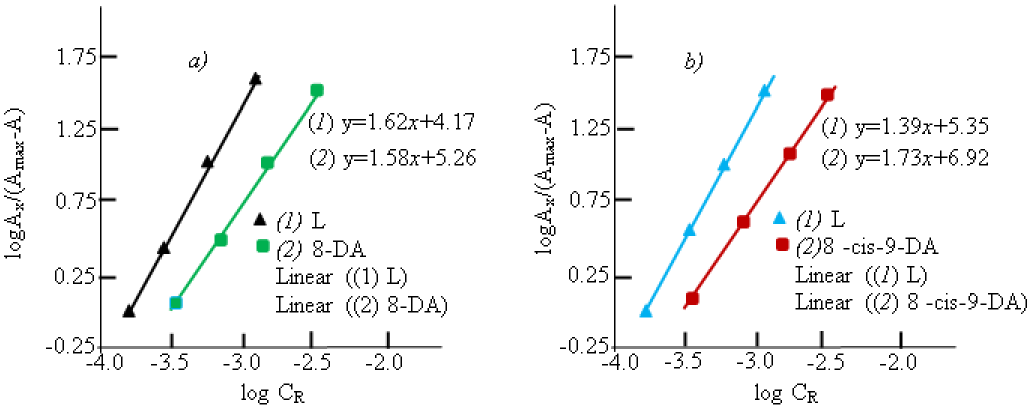
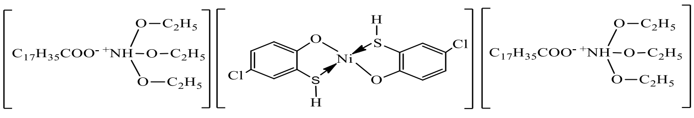
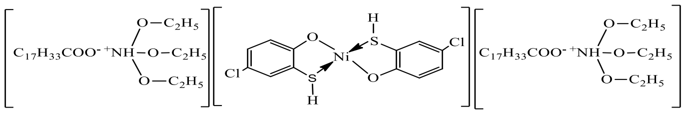


Fig. 3. Determination of the H_2L-to-Ni (straight line 1) and the QAS-to-Ni (straight line 2) molar ratios by the mobile equilibrium method. $C_{Ni(II)} = 2.035\times10^{-5}$ M, $C_L = C_{QAS}=1.0\cdot10^{-3}$ M, λ=490 nm, KFK-2 ℓ=0.5 cm.

Based on the data obtained, the composition of the isolated complexes can be represented by the formula $[NiL_2](QASH^+)_2$:





The stability constant of the Ni(II)-L-QAS complexes was calculated and found to be logβ = 8.91-10.37 at room temperature.

3.5. Equilibrium constants

Several processes should be taken into account for the system of $[NiL_2]^{2-}$, $(QASH^+)_2$, H_2O and $CHCl_3$:

I) Association in the aqueous phase between anionic chelate, $[Ni(HL)_2]^{2-}$, and the $(QASH^+)_2$, with the equilibrium constants $\{[Ni(HL)_2](QASH^+)_2\}$:

$$[Ni(HL)2]2-(aq) + 2QASH+(aq) \leftrightarrow [Ni(HL)2](QASH)2\ (aq) \quad (1)$$

$$\beta = \frac{[Ni(HL)_2](QASH)_2}{[Ni(HL)_2]^{2-}[(QASH^+)_2]} \quad (2)$$

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

$$[Ni(HL)2](QASH)2\ (aq) \leftrightarrow [Ni(HL)2](QASH)2\ (org) \quad (3)$$

with the distribution constants

$$K_D = \frac{[Ni(HL)_2](QASH)_2\ (org)}{\{[Ni(HL)_2]^{2-}[(QASH^+)_2]\}(aq)} \quad (4)$$

III) Equilibrium constant (K_{eq}) of the reaction

$$K_{eq} = \frac{\{[Ni(HL)_2](QASH)_2\}(org)}{\{[Ni(HL)_2]^{2-}\}(aq)([(QASH^+)_2]\}(aq)} = log\,\frac{A_x}{A_o - A_x} = D \quad (5)$$

$$K_{eq} = \frac{D}{[(QASH)^+]^2} \quad (6)$$

Where, A_x is the optical density for this experiment; A_o – optical density at complete binding of cobalt ion into a colored complex; *D* – distribution coefficient. Taking logarithm of the last expression, we get

$$logKeq = lgD - 2log[QASH+] \quad (7)$$

IV) Extraction of the ternary complexes from water into chloroform

$$[Ni(HL)2]2-\ (aq)\ + 2QASH+(aq) \leftrightarrow [Ni(HL)2](QASH)2\ (org) \quad (8)$$

org with the extraction constants

$$K_{ex} = K_D + \beta = \frac{\{[Ni(HL)_2](QASH)_2\}(org)}{\{[Ni(HL)_2]^{2-}\}(aq)([(QASH^+)_2]\}(aq)} \quad (9)$$

For the Ni(II)-L-QAS-H_2O-CH_3Cl systems, the values of the stability constants (logβ), distribution constants ($logK_D$), equilibrium constant ($logK_{eq}$) and extraction constant ($logK_{ex}$) were calculated, respectively, using Eqs. (2), (4), (7) and (9). All calculations were carried out at a probability of 95%. The obtained values are presented in Table 4.

3.6. Effect of interference ion

Various methods are proposed for the determination of nickel in anhydrous solutions. In order to evaluate the selectivity of those methods, we studied the effect of many foreign cations, as well as some complexing anions, on the results of determination of nickel by H_2L and QAS. The experiments were carried out according to the recipe, according to which the calibration curves were established, with the only difference that a certain amount of the corresponding ions was introduced into the solution other than Ni(II). The tolerance limit was set for ions that do not cause a deviation of more than +2% in the value of optical density for Ni(II)-L-QAS complexes (Table 5). It can be assumed that large amounts of alkaline ions, alkaline-earth ions, Cl^-, Br^-, SO_3^{2-}, SO_4^{2-}, NO_2^-, NO_3^- and $C_2O_4^{2-}$. Tartrate, citrate, F^-, I^-, CN^-, thiourea do not interfere determination of Cu(II). Co(II), Cu(II), Fe(II, III), V(IV,V), W(VI), Mo(VI), Ti(IV) and Mn(II) interfere determination of Ni(II). Interference of Fe(III) eliminated oxalic acid; Ti(IV) - tiron or sodium fluoride; Hg(II) ion -sulfit; Nb(V) and Ta(V) - oxalic acid, and Mo(VI) and W(VI) - sodium fluoride and oxalic acid. A comparison of photometric methods for determining Ni(II) with known reagents and L with QAS was carried out (Table 6). The methods for determining Ni(II) with L with QAS have a number of advantages over other reagents: the maximum of optical density is shifted to the long-wave region of the spectrum [13,21,53,54,56,57], the values of the molar absorption coefficient are higher than those of other complexes [21, 54-57], the complexation reaction occurs in a more acidic medium [54,56], which increases selectivity.

4. Analytical application

The methods we proposed were applied under already established optimal conditions for the determination of nickel in various objects: in wastewater and bottom sediments of metallurgical plants, in samples of natural soils, and also in samples of food products (Tables 7-9).

Table 4. Values of the stability constants (logβ), distribution constants ($logK_D$), equilibrium constant ($logK_{eq}$) and extraction constant ($logK_{ex}$) for the Ni(II)-L-QAS-H_2O-CH_3Cl systems

Extraction system	logβ	$logK_D$	$logK_{eq}$	$logK_{ex}$
Ni(II)-L-8-DA-H_2O-CH_3Cl	10.18±0.32	3.45±0.4	9.17±0.82	13.63±0.33
Ni(II)-L-8 -cis-9-DA-H_2O-CH_3Cl	8.39±0.41	3.51±0.4	8.69±0.51	11.90±0.27

Table 5. Influence of interfering ions on the determination of Ni(II) as MLC with H_2L and QAS (30.0 µg Ni added)

Foreign ions	**Molar exsess of the ion**	**Masking agent**	**Ni found (RSD (%))**	
			Ni(II)-L-8-DA	**Ni(II)-L-8 -cis-9-DA**
Mo(VI)	41	NaF	29.04 (6)	29.04 (6)
W(VI)	40	NaF	29.80 (5)	92.77 (6)
Cr(VI)	73		29.91 (4)	30.04 (6)
Cr(III)	59		29.80 (3)	29.08 (4)
Co(II)	57		29.82 (4)	30.07 (6)
Fe(II)	72		29.25 (6)	31.01 (2)
Fe(III)	50	$C_2O_4^{2-}$	30.04 (6)	31.04 (5)
V(IV)	48		30.55 (5)	31.02 (3)
V(V)	58		29.25 (3)	30.06 (6)
Cd(II)	25		29.86 (6)	30.22 (6)
Cu(II)	50	Thiourea	30.17 (7)	31.45 (2)
Al(III)	100		29.08 (4)	30.06 (6)
Zn(II)	60		30.04 (6)	30.82 (3)
Zr(IV)	76		30.18 (5)	30.51 (5)
Ta(V)	81	$C_2O_4^{2-}$	29.04 (6)	30.04 (6)
Ti(IV)	80	NaF	30.17 (7)	31.34 (4)
Nb(V)	70	$C_2O_4^{2-}$	29.91 (5)	29.27 (7)
Hg(II)	37	$Na_2S_2O_3$	29.86 (6)	30.04 (6)

Table 6. Comparative characteristics of methods for determining Ni(II)

Reagent*	**pH**	**λ, nm**	**$\varepsilon \times 10^{-4}$**	**Linear range of calibration curves, µg ml^{-1}**	**Reference**
PPTC	4-6	430	4.28	0.5-5.0	[53]
DMG	12	470	1.11	0.26-2.1	[21, 54]
ECTC	6.0	600	1.70	0.04–3.8	[55]
MCTC	1.6-2.9	410	7.17	0.8-3.4	[56]
TCQH	8.7-9.5	572	1.92	0.1-0.78	[56]
HBBH	4	497	1.72	1.2-2.4	[57]
L-8-DA	3.5-5.8	500	2.58	0.2-18	This work
L-8 -cis-9-DA	3.6-5.9	510	2.71	0.25-16	This work

*PPTC – Pyridoxal-4-phenyl-3-thiosemicarbazon, DMG – Dimethylglyoxime, ECTC – N-ethyl-3-carbazolecarboxaldehyde-3-thiosemicarbazone, MCTC – 7-methyl-2-chloroquinoline-3-carbaldehydethiosemicarbazone, TCQH– Thiazole-2-carbaldehyde-2-quinolylhydrazone, HBBH – 4-hydroxybenzaldehyde-4-bromophenylhydrazin.

Table 7. Determination results of nickel (II) with H_2L and QAS in the sewage water and bottom sediments (n= 6, P = 0.95)

Analysis object	**Added, µg/kg**	**Found, µg/kg**	**RSD (%)**	$\bar{X} \pm \frac{t_p \cdot S}{\sqrt{n}}$
Sewage water				
Sample 1	2.0	2.55	7.6	0.55±0.06
Sample 2	5.0	6.01	7.4	1.09±0.12
Bottom sediments				
Sample 1	5.0	6.25	6.5	1.25±0.07
Sample 2	5.0	6.85	6.2	1.85±0.05

Table 8. Determination results of nickel (II) in food samples (n= 6, P = 0.95)

Analysis object	**Reagent**	**Found in sample, µg/kg**	**SD**	**RSD (%)**	$\bar{X} \pm \frac{t_p \cdot S}{\sqrt{n}}$
Beans	DMG	1.68	0.059	2.7	1.68±0.06
	L-8-DA	1.71	0.048	2.8	1.71±0.05
	L-8 -cis-9-DA	1.66	0.038	2.6	1.66±0.04
Oats	DMG	0.46	0.009	2.9	0.46±0.01
	L-8-DA	0.42	0.019	4.1	0.42±0.02
	L-8 -cis-9-DA	0.47	0.009	2.9	0.47±0.01
Peas	DMG	2.46	0.011	4.5	2.46±0.12
	L-8-DA	2.35	0.088	3.9	2.35±0.09
	L-8 -cis-9-DA	2.28	0.087	3.9	2.28±0.09

Table 9. Determination results of nickel (II) in soil samples (n= 6, P = 0.95)

Soil	Method	$\bar{X}$, mg/кg	RSD (%)	$\bar{X} \pm \frac{t_p \cdot S}{\sqrt{n}}$
Sod-podzolic sandy and sandy loamy soils	DMG	3.19	5.1	3.19±0.16
	L-8-DA	3.21	4.2	3.21±0.15
	L-8 -cis-9-DA	3.20	3.9	3.20±0.12
Sod-podzolic loam and clay soils	DMG	9.89	4.5	9.89±0.47
	L-8-DA	9.95	4.2	9.95±0.44
Gray forest soils	DMG	13.05	3.0	13.05±0.41
	L-8-DA	12.96	3.9	12.96±0.39
	L-8 -cis-9-DA	15.78	3.7	15.78±0.61
Black earth	DMG	15.72	3.5	15.72±0.58
	L-8-DA	11.64	4.7	11.64±0.57
	L-8 -cis-9-DA	11.55	4.5	11.55±0.55
Chestnut soils	DMG	11.75	4.2	11.75±0.55
	L-8-DA	11.55	4.5	11.55±0.55
	L-8 -cis-9-DA	11.75	4.2	11.75±0.55

5. Conclusion

The oil-collecting and oil-dispersing properties of a quaternary ammonium salt (QAS) formed by octadecane (8-DA) and cis-9-octadecenoic acid (8 -cis-9-DA) with triethanolamine (TEA) {triethanolammonium salt of 8-DA and triethanolammonium salt of 8 -cis-9-DA}, as well as its use as an analytical reagent for the extractionphotometric determination of nickel in the form of a mixed-ligand complex (MLC) with 2-hydroxy-5-chlorothiophenol (L) and QAS in water were studied.

The maximum absorption of MLC Ni(II)-L-QAS is observed at λ = 500-510 nm ($pH_{op.}$ 3.5–5.9). The molar absorption coefficients range from $(2.58\text{-}2.71)\times10^4$. With single extraction using chloroform, 99.1–99.3% of nickel is extracted as MLC. Beer's law is followed within the range of 1.0–90 μg ml^{-1} of nickel.

It can be assumed that large amounts of alkaline ions, alkaline-earth ions, Cl^-, Br^-, SO_3^{2-}, SO_4^{2-}, NO_2^-, NO_3^- and $C_2O_4^{2-}$. Tartrate, citrate, F^-, I^-, CN^-, thiourea do not interfere determination of Ni(II). Co(II), Cu(II), Fe(II, III), V(IV,V), W(VI), Mo(VI), Ti(IV) and Mn(II) interfere determination of Ni(II). Interference of Fe(III) eliminated oxalic acid; Ti(IV) - tiron or sodium fluoride; Hg(II) ion -sulfit; Nb(V) and Ta(V) - oxalic acid, and Mo(VI) and W(VI) - sodium fluoride and oxalic acid.

The methods we proposed were applied under already established optimal conditions for the determination of nickel in various objects: in wastewater and bottom sediments of metallurgical plants, in samples of natural soils, and also in samples of food products

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