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SPECTROPHOTOMETRIC STUDY OF NICKEL (II) COMPLEXES WITH 2-HYDROXYTHIOLPHENOL AND ITS DERIVATIVES IN THE PRESENCE OF HYDROPHOBIC AMINES

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Abstract: *The complexation reactions of nickel (II) with 2-hydroxythiophenol and its derivatives (2,5-dihydroxythiophenol, 2-hydroxy-5-chlorothiophenol, 2-hydroxy-5-bromothiophenol, and 2-hydroxy-5-iodothiophenol) in the presence of hydrophobic amines were studied by spectrophotometric methods. Aniline and N, N-dimethylaniline were used as hydrophobic amines. It found that mixed-ligand complexes (MLC) are formed in a weakly acidic medium (pH 2.3-8.0). The maximum in the light absorption spectrum of the complexes is observed at* λ *= 620-650 nm. The molar coefficients of light absorption are* $(4.1\text{-}4.3)\times10^4$*. The best extractants were chloroform, dichloroethane and carbon tetrachloride. With a single extraction with chloroform, 97.8-98.4% of nickel is extracted in the form of MLC. Based on the data obtained, photometric methods for the determination of nickel in various objects were developed.*

Keywords: *nickel, 2-hydroxythiophenol, 2,5-dihydroxythiophenol, 2-hydroxy-5-chlorothiophenol, 2-hydroxy-5-bromothiophenol, 2-hydroxy-5-iodothiophenol, aniline.*

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Introduction

Many chelating reagents belonging to different classes of compounds and containing N, O, or S as donor atoms are suitable for the photometric determination of nickel [1]. It is known that by improving the analytical parameters of the reaction of mixed-ligand complexes (MLC) formation, they have found wide application in the photometric determination of elements [2-5].

According to the hypothesis of analogies, reactions with reagents of the R-SH type are possible for ions of elements that form sulfides which are poorly soluble in water [6]. It was of interest to study the interaction of nickel with 2-hydroxythiophenol (HTP) and its derivatives {2,5-dihydroxythiophenol (DHTP), 2-hydroxy-5-chlorothiophenol (HCTP), 2-hydroxy-5-bromothiophenol (HBTF) and 2-hydroxy -5-iodothiophenol (HITP)} and hydrophobic amines (Am) {aniline (An) and N, N-dimethylaniline (dAn)}.

Experimental part

Reagents and solutions. A solution of nickel (II) (0.1 mg / ml) was prepared by dissolving in water $NiCl_2 \times 6H_2O$. The titer of the solution was established by the gravimetric method in the form of dimethylglyoxime [2]. Solutions with a concentration of 0.1 mg / ml were obtained by diluting the stock solution.

Hydroxythiophenol derivatives (L) were purified by reprecipitation from ethanol solutions by adding water and then by distillation. We used 0.01 M solutions of L chloroform. Purified chloroform was used as an extractant.The ionic strength of solutions equal to μ = 0.1 was maintained constant by introducing a calculated amount of KCl. To create the required acidity of the solutions, a 1M

NaOH solution was used. All reagents used were of analytical grade. or x. h.

Apparatus. The optical density of the organic phase was measured on KFK-2 and SF-26. The pH value of the aqueous phase was monitored using an I-120.2 instrument with a glass electrode. The NMR spectra of the reagents were recorded on a Bruker pulse Fourier spectrometer (Germany), at an operating frequency of 300.18 MHz, in deuterated benzene at room temperature. The relative abundance of protons of various structural groups was determined by integrating the corresponding resonance absorption bands. IR spectra were recorded on a Bruker spectrophotometer.

Methods. Graduated tubes with ground-in stoppers were injected with 0.1–0.8 ml, with an interval of 0.1 ml of the initial nickel solution, 2.5 ml of a 0.01 M solution of L, and 0.8–1.0 ml of Am. The required pH value was set by adding 1M NaOH solution. The volume of the organic phase was brought to 5 ml with chloroform, and the volume of the aqueous phase - 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 dıscussıon

L are dibasic (HTP, HCTP, HBTF and HITF, H_2L) and tribasic weak acid (DHTP, H_3L) and, depending on the pH of the medium, can exist in molecular and anionic forms. Their estimates of the ionization constants obtained by potentiometric titration are as follows: DHTP (pK_1 = 6.33; pK_2 = 8.82; pK_3 = 12.78), HTP (pK_1 = 5.8; pK_2 = 10.9), HCTP (pK_1 = 5.1; pK_2 = 10.6), HBTF (pK_1 = 5.05; pK_2 = 10.4), HITF (pK_1 = 5.0; pK_2 = 10.2). The dependence of the ionization constant (pKa) for L on the ionic strength of the solution in the range μ = 0-1 is linear and corresponds to the following equation:

DHTP: $pK_1 = 6.88 - 0.77\sqrt{\mu}$; $pK_1 = 9.76 - 0.77\sqrt{\mu}$; $pK_2 = 11.73 - 0.63\sqrt{\mu}$.
HTP: $pK_1 = 5.65 - 0.53\sqrt{\mu}$; $pK_2 = 11.21 - 0.35\sqrt{\mu}$.
HCTP: $pK_1 = 5.28 - 0.56\sqrt{\mu}$; $pK_2 = 10.73 - 0.40\sqrt{\mu}$.
HBTF: $pK_1 = 5.26 - 0.67\sqrt{\mu}$; $pK_2 = 10.53 - 0.42\sqrt{\mu}$.
HITF: $pK_1 = 5.15 - 0.46\sqrt{\mu}$; $pK_2 = 10.36– 0.51\sqrt{\mu}$.

The synthesized compounds were determined by physicochemical methods: IR and NMR spectroscopy (Table 1) [7,8]:

Table 1. Results of IR and NMR spectroscopy studies

Reagents	IR spectrum (KBr, ν, см^{-1}):	Spectrum ^{1}H NMR (300,18 MHz, C_6D_6, δ, ppm):
HTP	3470(OH), 2580(SH), 1580 (C_6H_5)	5.48 (s, 1H, OH), 3.47 (s, 1H, SH), 7.28 (s, 2H, Ar−H), 6.95 (s, 1H, Ar−H), 6.92 (s, 1H, Ar−H).
DHTP	3460 (OH), 2570 (SH), 1555 (C_6H_5)	5.24 (s, 2H, OH), 3.38 (s, 1H, SH), 7.11 (s, 2H, Ar−H), 6.95 (s, 1H, Ar−H).).
HCTP	3458 (OH), 2568 (SH), 1535 (C_6H_5)	5.70 (s, 1H, OH), 3.35 (s, 1H, SH), 7.05 (s, 2H, Ar−H), 6.42 (s, 1H, Ar−H).
HBTP	3462 (OH), 2563 (SH), 1532 (C_6H_5)	5.72 (s, 1H, OH), 3.31 (s, 1H, SH), 7.09 (s, 2H, Ar−H), 6.38 (s, 1H, Ar−H).
HITP	3460 (OH), 2571 (SH), 1539 (C_6H_5)	5.69 (s, 1H, OH), 3.28 (s, 1H, SH), 7.12 (s, 2H, Ar−H), 6.34 (s, 1H, Ar−H).

Influence of pH of the aqueous phase. The study into 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. At a solution pH of 8, MLC extraction is practically not observed which is apparently associated with an increase in free molecules of hydrophobic amines. On the other hand, in the case of DHTP, the concentration of the non-extractable complex $[NiL_2]^{4-}$ in an aqueous solution increases, since the dissociation at the second -OH group continues to increase. The dependence of optical density on pH is shown in Fig. 1. The presence of one maximum of optical density within the indicated pH range confirms the assumption of the formation of one complex compound.

Absorption spectra. The maximum analytical signal during the complexation of nickel with L and Am is observed at 520-550 nm (Fig. 1). L absorb maximum at 278-283 nm. During complexation, a bathochromic shift of the absorption maximum by 239-267 nm is observed. The contrast of the reactions is high: the initial reagents are almost colorless, and the complexes are red. Colored chloroform extracts of hydroxythiophenolate-amine associates absorb maximum at 325-335 nm. A sharp difference in the values of the maximums of light absorption of complexes and associates allows us to conclude that the resulting compounds are MLCs with a mixed coordination sphere. The molar absorption coefficients are $(4.1\text{-}4.3)\times10^4$.

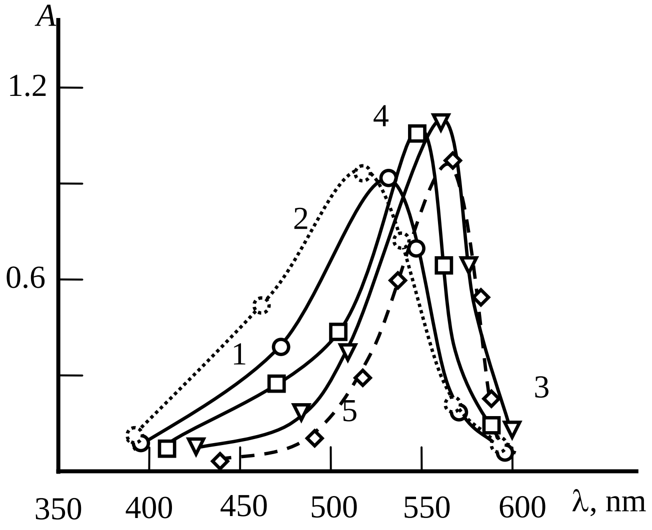

Fig. 1. Absorption spectra of nickel complexes with L and An.
1. Ni-HTP-An; 2. Ni-DHTP-An; 3. Ni-HCTP-An; 4. Ni-HBTP-An; 5. Ni-HITP-An
$C_{Ni} = 3.4\times10^{-5}$ M; $C_L = (0.8\text{-}1.0)\times10^{-3}$ M; $C_{\text{Aн}} = (1.0\text{-}1.2)\times10^{-3}$ M, SP-26, $\lambda = 590$ nm, $\ell = 1.0$ cm

The choice of the extractant. Non-aqueous solvents were used for MLC extraction: chloroform, 1,2-dichloroethane, carbon tetrachloride, benzene, chlorobenzene, toluene, xylol, isobutanol, and isopentanol. The best extractants were chloroform, dichloroethane and carbon tetrachloride. With a single extraction with chloroform, 97.8-98.4% of nickel is extracted in the form of MLC. Further studies were carried out with chloroform. The copper content in the organic phase was determined photometrically – with dimethylglyoxime after re-extraction [2], and in the water phase – by the 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 $(0.8\text{-}1.0)\times10^{-3}$ M concentration of L and $(1.0\text{-}1.2)\times10^{-3}$ M - Am.

MLC of nickel with L and Am 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.

Composition and structure of the complexes. The stoichiometry of the complexes under study was established by the methods of equilibrium shift and relative yield [9]. In the composition of the MLC, there are two moles of L and Am per mole of metal. Using the Nazarenko method, it was established that the complexing form of nickel is Ni^{2+} [10]. In this case, the number of protons displaced by it from one molecule L turned out to be equal to 2 (Fig. 2).

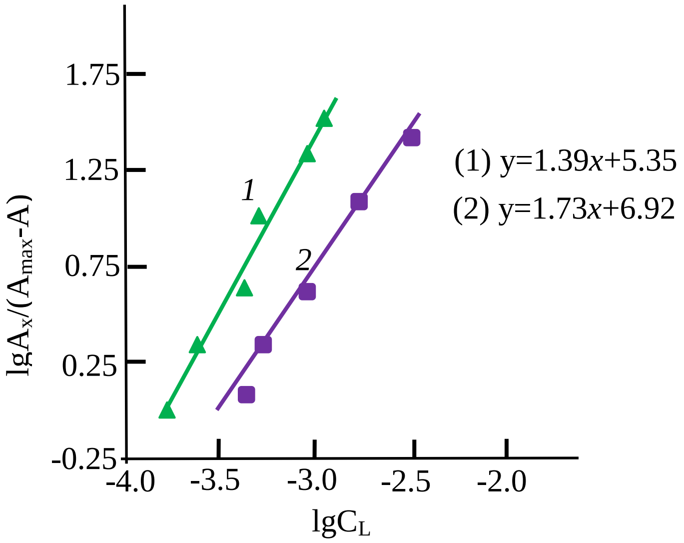

Fig.2. Determination of the HITP-to-Ni (straight line 1) and the An-to-Ni (straight line 2) molar ratios by the mobile equilibrium method. $C_{Ni} = 3.4\times10^{-5}$ M, $C_L = 1.2\times10^{-3}$ M, $C_{An} = 2.0\times10^{-3}$ M, λ=550 nm, KFK-2, ℓ=0.5 cm.

An intense absorption band appears in the IR spectra of the Ni-HTP-An complex in the region of 950-960 cm-1, which is absent in the spectra of the reagent. This band is due to the stretching vibration of the metal-ligand bond. The disappearance of a pronounced band at 2580 cm^{-1}, observed in the HTP spectrum, suggests that the –SH groups are involved in the formation of the complex. The observed decrease in the intensity of the absorption band in the region of 3200-3600 cm^{-1} with a maximum at 3460 cm^{-1} and the appearance of a wide band in the region of 3050-3160 cm^{-1} reveals that the -OH group is involved in the formation of coordination bonds in an ionized state. The detection of absorption bands at 1370 cm^{-1} indicates the presence of protonated aniline [7, 8].

The calculations showed that MLC in the organic phase does not polymerize and is in the monomeric form (γ = 0.94–1.07) [11].

Table 2 shows the main spectrophotometric characteristics of MLC nickel.

Table 2. Characteristics of MLC nickel with L and Am

Соединение	pH		λ, nm	$\varepsilon\times10^{-4}$	$lgk_{eq.}$	lgk_{ex}	$lg\beta_k$	Working range μg/ml
	Education and extraction	Opt.						
$[Ni(DHTP)_2]$ $(AnH)_2$	3.4-8.0	3.8-6.5	620	4.3	3.69	9.69	10.67	0.2-20
$[Ni(HTP)_2]$ $(AnH)_2$	3.0-7.5	3.5-6.0	630	4.2	3.49	9.26	10.28	0.2-20
$[Ni(HCTP)_2]$ $(AnH)_2$	2.3-6.5	2.6-5.3	650	4.0	3.51	9.31	9.75	0.2-13
$[Ni(HCTP)_2](DAnH)_2$	2.4-6.6	2.7-5.4	648	4.1	3.57	9.55	9.77	0.2-15
$[Ni(HBTP)_2](AnH)_2$	1.9-6.2	2.4-4.8	655	3.8	3.43	9.71	9.45	0.2-15
$[Ni(HITP)_2]$ $(AnH)_2$	1.5-5.5	2.1-4.3	660	3.6	3.82	10.26	9.08	0.2-12

Based on the data obtained, the composition of the extracted complexes can be represented by the formula $[NiL_2)](AmH)_2$. The mechanism of MLC formation can be represented as follows: L at pH 1.5-8.0 forms a compound insoluble in chloroform with an excess of nickel ions. Am is added to such a system, an intensely colored compound, soluble in chloroform, is instantly given.

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

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

$$[NiL_2]^{2-} + 2AmH^+ \leftrightarrow [NiL_2](AmH)_2$$

The equilibrium constant of the reaction is

$$K_p = \frac{\{[NiL_2(AmH)_2)_{orq}}{\{[NiL_2]^{2-}\}_{aq}\{[AmH^+]^2\}_{aq}} = \lg\frac{A_x}{A_0 - A_x} = D$$

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

Taking the logarithm of the last expression, we get

$$\lg K_{eq} = \lg D - 2\lg[AmH^+]$$

The extraction constants were calculated using the equations

$$\lg K_{ex} = \lg D - 2\lg[H_2L] - 2\lg[\text{AмH}^+].$$

The results of calculating the equilibrium constant and the extraction of the reaction were shown in Table. 2.

Based on the equations of the calibration curves, the photometric detection limit and the quantitative determination limit of nickel in the form of MLC were calculated [12]. Table 3 shows the analytical characteristics of some complexes of nickel with L and An.

Table 3. Analytical characteristics of MLC nickel with L and An

Parameter	Ni-DHTP-An	Ni-HTP-An	Ni-HCTP-An
Equation of calibration curves	*y* =0.086+0.0066x	*y* = 0.062+0.0073x	*y* = 0.093+0.0070x
Correlation coefficient	0.9981	0.9984	0.9989
Linear range of calibration curves, µg/ml	0.06-3.2	0.04-3.5	0.04-3.6
Detection limit ng/cm^3	9.2	8.70	8.50
Limit of quantitative determination, ng/cm^3	28.0	26.5	23.4
Sensitivity, ng/cm^2	2.41	2.31	2.19

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

The influence of foreign ions. To assess the applicability of RLC for the separation and determination of nickel, the interfering effect of foreign ions was studied. It was found that large amounts of alkaline, alkaline-earth elements, REE, F-, CI-, do not interfere with the determination of nickel. Citrates and tartrates, J^-, CN^-, $S_2O_3^{2-}$, thiourea interfere with the determination. The interfering influence of Fe (III) was eliminated 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 using a 0.01M solution of oxalic acid, V (IV), Nb (V), Ta (V), Cr (III), Mo (VI), W (VI) and Fe (III) do not interfere with the determination.

The comparison of methods for the determination of Ni (II) with known reagents and L in the presence of amines. Table 4 shows the data allowing to compare the analytical characteristics of the photometric methods developed by us for the determination of Ni (II) with some already known methods.

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

Reagent	pH	λ_{max}	ε	Working range µg/ml
Dimethylglyoxime [1]	12	470	-	0.26-2.1
N-ethyl-3-carbazolecarboxaldehyde-3-thiosemicarbazone [13]	6.0	400	1.11	-
7-Methyl-2-chloroquinoline-3-carbaldehyde-thiosemicarbazone [14]	6.0	410	1.67×10^2	-
Thiazole-2-carbaldehyde-2-quinolylhydrazone [15]	8.7-9.5	522	7.17×10^4	0.7
Pyridoxal-4-phenyl-3-thiosemicarbazone [16]	4-6	430	1.92	0.5-5
4-Hydroxybenzaldehyde-4-bromophenylhydrazine [17]	4	497	12.85	0.01-0.1
DHTP-An	3.8-6.5	620	4.3×10^4	0.2-20
HTP-An	3.5-6.0	630	4.2×10^4	0.2-20
HCTP-An	2.6-5.3	650	4.1×10^4	0.2-13
HBTP-An	2.4-4.8	655	3.8×10^4	0.2-15
HITP-An	2.1-4.3	660	3.6×10^4	0.2-12

The results of studies of the formation and extraction of Ni (II) MLCs with L and Am, the physicochemical and analytical characteristics of these compounds served as the basis for the development of new methods for the extraction-photometric determination of Ni (II) in magnesium and carnallite.

Determination of nickel in magnesium and carnallite. The developed analytical procedure was applied for the analysis of real samples. Four replicates of metallic magnesium and four samples of carnallite were subjected to the analysis. The results are shown in Table 5; their reliability was verified by the added–recovered method.

Table 5. Determination of nickel in metallic magnesium and carnallite

Sample	Found (%) $\times10^{-4}$	Added-recovered method		
	Ni	Added (%) $\times10^{-3}$	Recovered (%) $\times10^{-3}$	RSD (%)
		Ni	Ni	Ni
1[a]	7.65±0.01	1	1.765	1.4
2[a]	7.64±0.02	1	1.764	1.5
3[a]	7.70±0.02	1	1.770	1.2
4[a]	7.63±0.02	1	1.763	1.3
1[b]	8.71±0.01	1	1.871	1.3
2[b]	8.69±0.02	1	1.869	1.8
3[b]	8.90±0.01	1	1.890	1.2
4[b]	8.81±0.01	1	1.881	1.5

Note: [a] – Metallic magnesium; *n*=6; P=95%; [b] – Carnallite; *n*=5; P=95%

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NİKELİN (II) 2-HİDROKSİTİOLFENOL, ONUN TÖRƏMƏLƏRİ VƏ HİDROFOB AMİNLƏRLƏ KOMPLEKSLƏRİNİN SPEKTROFOTOMETRİK TƏDQİQİ

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Nikelin (II) hidrofob amin iştirakında 2-hidroksitiofenol və onun törəmələrilə (2,5-dihidroksitiofenol, 2-hidroksi-5-xlorotiofenol, 2-hidroksi-5-bromotiofenol və 2-hidroksi-5-yodtiofenol) kompleksləri spektrofotometrik üsulla tədqiq edilmişdir. Hidrofob amin kimi anilin və N, N-dimetilanilin istifadə edilmişdir. Müəyyən edilmişdir ki, müxtəlifliqandlı komplekslər (MLK) zəif turşu mühitdə (pH 2.3-8.0) əmələ gəlir. Komplekslərin işıq udma spektrində maksimum λ = 620-650 nm-də müşahidə olunur. Molyar işıqudma əmsalı (4.1-4.3) × 10^4-dür. Ən yaxşı ekstragent kimi xloroform, dixloretan və karbontetraxlorid seçilmişdir. Xloroform ilə birdəfəyə 97.8-98.4% nikel MLC şəklində ekstraksiya olunur. Alınan məlumatlar əsasında müxtəlif obyektlərdə nikelin təyini üçün fotometrik üsullar işlənib hazırlanmışdır.

Açar sözlər: *nikel, 2-hidroksitiofenol, 2,5-dihidroksitiofenol, 2-hidroksi-5-xlorotiofenol, 2-hidroksi-5-bromotiofenol, 2-hidroksi-5-iyodotiofenol, anilin.*

СПЕКТРОФОТОМЕТРИЧЕСКОЕ ИЗУЧЕНИЕ КОМПЛЕКСОВ НИКЕЛЯ (II) С 2-ГИДРОКСИТИОЛФЕНОЛОМ И ЕГО ПРОИЗВОДНЫМИ В ПРИСУТСТВИИ ГИДРОФОБНЫХ АМИНОВ

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Реакции комплексообразования никеля (II) с 2-гидрокситиофенолом и его производными (2,5-дигидрокситиофенол, 2-гидрокси-5-хлортиофенол, 2-гидрокси-5-бромтиофенол и 2-гидрокси-5-йодотиофенол) в присутствии гидрофобных аминов исследовали спектрофотометрическим методом. В качестве гидрофобных аминов использовали анилин и N, N-диметиланилин. Установлено, что разнолигандные комплексы (РЛК) образуются в слабокислой среде (pH 2.3-8.0). Максимум в спектре поглощения света комплексов наблюдается при λ = 620-650 нм. Молярные коэффициенты поглощения света составляют (4.1-4.3)×10^4. Лучшими экстрагентами были хлороформ, дихлорэтан и четыреххлористый углерод. При однократной экстракции хлороформом 97.8-98.4% никеля извлекается в виде РЛК. На основе полученных данных разработаны фотометрические методы определения никеля в различных объектах.

Ключевые слова: *никель, 2-гидрокситиофенол, 2,5-дигидрокситиофенол, 2-гидрокси-5-хлортиофенол, 2-гидрокси-5-бромтиофенол, 2-гидрокси-5-йодотиофенол, анилин.*