

Təbiət elmləri

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EXTRACTION-SPECTROPHOTOMETRIC DETERMINATION OF COBALT(II) IN DIFFERENT OBJECTS

Keywords: Cobalt, chloroform, solvent extraction, spectrophotometry

Mixed-ligand complexes of cobalt (II) with *o*-hydroxythiophenol derivatives (*o*-HTPD) {2-hydroxythiophenol and its derivatives (2-hydroxy-5-chlorothiophenol, 4-hydroxy-3-thiolsulfonic acid, 4-hydroxy-3-thiolbenzoic acid, 4-nitro-2-hydroxythiophenol} in the presence of hydrophobic amines (Am). have been studied by spectrophotometry. The optimal conditions for the formation and extraction of mixed-ligand compounds have been found and the ratios of components in the complexes have been determined: (pH 1.9-5.1, $(1.3-1.5) \times 10^{-3}$ mol mL⁻¹ HTPD, $(1.2-1.5) \times 10^{-3}$ mol mL⁻¹ Am, $\lambda_{\max} = 540-560$ nm, Co(II): *o*-HTPD: Am=1:2:2). The Beer's law was applicable in the range of 0.05-3.2 µg/mL. The effect of foreign ions and reagents on the extraction was studied. A procedure has been developed for extraction – spectrophotometric determination cobalt in different objects.

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KOBALTIN (II) MÜXTƏLİF OBYEKTŁƏRDƏ EKSTRAKSİYALI SPEKTROFOTOMETRİK TƏYİNİ

Açar sözlər: kobalt, xloroform, həllediciəkdə ekstraksiya, spektrofotometriya

Kobaltın (II) *o*-hidroksitiofenol törəmələri (*o*-HTT) {2-hidroksitiofenol və onun törəmələri (2-hidroksi-5-xlorotiofenol, 4-hidroksi-3-tiolsulfo turşu, 4-hidroksi-3-tiolbenzoy turşusu, 4-nitro-2-hidroksitiofenol} və hidrofob aminlərlə (Am) əmələ gətirdiyi müxtəlifliqandlı kompleksləri spektrofotometrik üsulla tədqiq edilmişdir. Müxtəlifliqandlı kompleks birləşmələrinin əmələ gəlmə və ekstraksiya şəraiti və kompleksdə komponentlərin molyar nisbəti müəyyən edilmişdir: (pH 1.9-5.1, $(1.3-1.5) \times 10^{-3}$ mol ml⁻¹ *o*-HTT, $(1.2-1.5) \times 10^{-3}$ mol ml⁻¹ Am, $\lambda_{\max} = 540-560$ nm, Co(II): *o*-HTT: Am=1:2:2). Ber qanunu 0.05-3.2 mkq/ml intervalında özünü doğruldur. Kənar

ionların və reagentlərin ekstraksiyaya təsiri öyrənilmişdir. Kobaltın müxtəlif obyektlərdə ekstraksiya-spektrofotometrik təyini metodikası işlənilmişdir.

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ЭКСТРАКЦИОННО-СПЕКТРОФОТОМЕТРИЧЕСКОЕ ОПРЕДЕЛЕНИЕ КОБАЛЬТА (II) В РАЗЛИЧНЫХ ОБЪЕКТАХ

Ключевые слова: кобальт, хлороформ, экстракция растворителем, спектрофотометрия

Разнолигандные комплексы кобальта (II) с производными о-гидрокситиофенола (о-ПГТ) {2-гидрокситиофенол и его производными (2-гидрокси-5-хлортиофенол, 4-гидрокси-3- тиолсульфоновой кислоты, 4-гидрокси-3-тиолбензойной кислоты, 4-нитро-2-гидрокситиофенола} в присутствии гидрофобных аминов (Ам) изучены спектрофотометрически. Найдены оптимальные условия образования и экстракции разнолигандных соединений, определены соотношения компонентов в комплексах: (рН 1.9-5.1, $(1.3-1.5) \times 10^{-3}$ мол мл⁻¹ о-ПГТ, $(1.2-1.5) \times 10^{-3}$ мол мл⁻¹ Ам, $\lambda_{\max} = 540-560$ нм, Со(II): о-НТПД: Ам=1:2:2). Закон Бера применим в интервале 0.05-3.2 мкг/мл. Изучено влияние посторонних ионов и реagensов на экстракцию. Разработана методика экстракции – спектрофотометрического определения кобальта.

1. Introduction

Cobalt is a transition element of great industrial importance due to its valuable alloying, coloring, magnetic, catalytic, and electroplating properties. It also has biological significance due to its ability to be the active site of coenzymes, i.e. gram. vitamin B12 [1]. A wide variety of photometric reagents are known for the determination of cobalt [2-11].

Oxyphenolate complexes of cobalt are insoluble in chloroform, while mixed ligand complexes with hydrophobic amines and aminophenols are readily soluble in various organic solvents [5-11]. In this regard, o-hydroxythiolphenols (HTPD) are very promising reagents, which contain one hydroxyl and one sulfohydyl group and are a sulfur-containing analogue of mononuclear polyphenols with the replacement of one oxygen atom by sulfur atoms. The present work is devoted to the study of the complexation reaction of Cobalt (II) with o-hydroxythiolphenols (о-HP) in the presence of hydrophobic amines (Am). From o-hydroxythiophenols {2-hydroxythiophenol (GTP) and its derivatives (2-hydroxy-5-chlorothiophenol (HCTP), 4-hydroxy-3-thiolsulfonic acid (HTSA), 4-hydroxy-3-thiobenzoic acid (NTBA), 4-nitro-2-hydroxythiophenol (GNTF)} in the presence of hydrophobic amines (Am. in

the presence of hydrophobic amines (Am). Aniline (An) and N-methylaniline (mAn) were used as hydrophobic amine.

2. Experimental

Reagents. A stock solution (1mg / mL) of cobalt (II) was prepared by dissolving in water an exact linkage $\text{CoSO}_4 \times 7\text{H}_2\text{O}$ in water containing 2 ml conc. H_2SO_4 , and diluted with water to 1 liter [12]. The concentration of the cobalt solution was adjusted gravimetrically [2,4]. Solutions of HTPD and Am in chloroform (0.01M) were used. HTPD were synthesized according to the procedure [14]. Their purity was verified by melting point determination and paper chromatography. To create the optimal acidity, 0.1M solutions of KOH and HCl or $\text{CH}_3\text{COOH} + \text{CH}_3\text{COONH}_4$ buffers were applied. The extractant was purified chloroform.

Apparatus. The absorbance was recorded with a KFK-2 photocolormeter and a SF 26 spectrophotometer (USSR), equipped with 5 and 10 mm pathlength cells. pH was measured using an I-120.2 potentiometer with a glass electrode. IR spectra were recorded on a spectrophotometer "Bruker" (Germany).

General Procedure for the Determination of Cobalt (II). Portions of stock solutions of Cobalt (II) varying from 0.1 to 1.0 mL with a 0.1-mL step, a 2.2 mL portion of a 0.01 M solution of HTPD, and a 2.5 mL portion of a 0.01M solution of Am were placed in to calibrated test tubes with ground-glass stoppers (the volume of the organic phase was 5 mL). The required value of pH was adjusted by adding 1M HCl. The volume of the aqueous phase was increased to 20 mL using distilled water. In 8 minnute after the complete separation of the phases, the organic phase was separated from the aqueous phase and the absorbance of the extracts was measured on KFK-2 at room temperature and 540 nm ($\ell=0.5\text{cm}$).

Charge of the complexes. The binary complexes Co(II)-HTPD , cannot be extracted in chloroform or other slightly polar organic solvents. Experiments with KU-2 and AV-17 ion-exchangers showed that these species are charged negatively. Electroneutral ternary complexes can be formed in the presence of Am.

3. Results and Discussion

Choice of organic solvent. The following organic solvents were tested for the extraction of these complexes: chloroform, 1,2-dichloroethane, carbon tetrachloride, benzene, toluene, xylene, *iso*-butanol, and *iso*-pentanol. Chloroform was found to be the most effective. At the optimum conditions this solvent provides degrees of extraction $R=95.9-99.1\%$ (Table 1).

Table 1. Optical characteristics, precision and accuracy of the spectrophotometric determination of Co(II) with HTPD and Am

Compound	The pH range of maximum extraction	R,%	λ_{\max} (nm)	Molar absorptivity ($L \cdot mol^{-1} \cdot cm^{-1}$) $\epsilon \cdot 10^{-4}$	lgK_{eq}	$lg\beta$	lgK_{ex}	Working range / $\mu g \cdot 5cm^{-3}$
Co-HTP-An	3.5-5.8	98.4	545	2.58	8.46	13.67	10.56	0.5-70
Co-HTP-mAn	3.6-5.9	98.5	550	2.71	8.49	13.62	10.58	0.5-80
Co-HXTP-An	3.0-5.1	96.9	552	2.89	7.96	12.85	10.61	0.5-85
Co-HXTP-mAn	3.1-5.2	97.4	560	2.97	7.98	12.83	10.65	0.6-80
Co- HTSA -An	2.5-4.1	95.9	540	3.12	7.78	12.55	10.72	0.5-100
Co- HTSA -mAn	2.3-3.8	97.8	548	3.38	7.81	12.64	10.77	0.6-95
Co- HTBA -An	2.4-3.5	96.7	556	3.49	7.41	11.94	10.83	0.5-80
Co- HTBA -mAn	2.2-3.4	98.3	552	3.55	7.58	12.20	10.85	0.5-85
Co- HNTP -An	1.9-3.1	99.1	558	3.69	6.38	10.24	10.89	0.6-80
Co- HNTP -mAn	1.8-2.9	98.5	560	3.72	6.65	10.17	10.92	0.5-80

Influence of the pH of the Aqueous Phase. The effect of pH on the formation of Co(II)- HTPD -Am complex was studied, in order to find a suitable pH that can be adopted in the determination of cobalt(II). The absorbance was found to be maximum in the pH range 1.8-5.8. Extraction of Co(II) enhanced with the increase in the acidity of the initial solution; the further increase in acidity lead to the gradual decrease of recovery, which was obviously associated with a decrease in the concentration of the ionized form of HTPD. Probably, it is present in the solution in the nondissociated state. At $pH \geq 7.6$, the complexes were hardly extracted, obviously because of the decrease in the degree of Am protonation.

Absorption maxima, reagents concentrations, molar absorptivities and effect of time. The absorption maxima (λ_{\max}) of the ternary Co(II)-HTPD-Am complexes lie in the range of 540-560 nm (Table I). All colour reactions are very contrast since the initial solutions are colourless (λ_{\max} HTPD = 275-280 nm). Complete extraction is achieved at reagent concentrations not lower than $(1.3-1.5) \times 10^{-3} \text{ mol mL}^{-1}$ (HTPD) and $(1.2-1.5) \times 10^{-3} \text{ mol mL}^{-1}$ (Am). Co(II) concentration ranges in which the Beer's law is obeyed are listed in Table 1. The calculated molar absorptivities (ϵ_{\max}) belong to the interval $(2,58-3,71) \times 10^4 \text{ mL}$

$\text{mol}^{-1} \text{cm}^{-1}$. Colour develops almost immediately after the reagents addition. The absorbance of the extracts is stable for at least 48 hours. The optimum shaking time is 10 min.

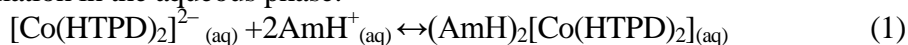
Stoichiometry of the Complexes and the Mechanism of Complexation.

The molar ratios of the components of the ternary complexes were established by the equilibrium shift method and the method of Asmus [15]. The results show a complex composition of 1:2:2 (Co(II):HTPD:Am). The formation of ternary complexes can be presented in the following way. When cobalt interact with two molecules of HTPD, they form doubly-charged anionic complexes, which are extracted with two molecules of protonated Am. Using the Nazarenko's method [11], we found that the cobalt complexation form is Co^{2+} . Hence, the complexes can be regarded as ion associates between doubly charged anionic chelates $[\text{Co}(\text{HTPD}^{2-})_2]^{2-}$ and two protonated Am species: $(\text{AmH}^+)_2[\text{Co}(\text{HTPD})_2]$. The stability constant of Co(II)-HTPD-Am complexes was calculated and found to be $\lg\beta = 10.17-13.67$ at room temperature.

The disappearance of the pronounced absorption bands in the $3200-3600 \text{cm}^{-1}$ with a maximum at 3460cm^{-1} observed in the spectrum of HTPD, says that the -OH group is involved in the formation of the complex. The observed decrease in the intensity, absorption bands in the area 2580cm^{-1} shows that -SH group involved in the formation of coordination bond in the ionized state. Detection of the absorption bands at 2380cm^{-1} indicates the presence of a protonated aniline [13].

Several equilibrium processes are important when we describe quantitatively the formation and subsequent extraction of $(\text{AmH}^+)_2[\text{Co}(\text{HTPD})_2]$.

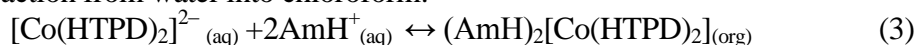
Formation in the aqueous phase:



Distribution:



Extraction from water into chloroform:



The equilibrium constants describing these processes are shown in Table 1.

The stability constant determined by crossed lines method. The sizes of equilibrium constant K_e calculated on a formula $\lg K_e = \lg D - 2\lg[\text{AmH}^+]$ were presented in table 1. Calculation of extent of polymerization of complexes was carried out on the equation [9-11]. The made calculations showed that ternary Co(II)-HTPD-Am complexes in an organic phase won't be polymerized and are in a monomeric form ($\gamma = 1,08-1,31$).

Effect of foreign ions and reagents. The effect of various ions and reagents on the extraction-spectrophotometric determination of $20 \mu\text{g}$ cobalt (II) is summarised in Table 2. It can be assumed that large amounts of alkaline ions, alkaline-earth ions, NH_4^+ , W(VI), Mo(VI), Cl⁻, $\text{S}_2\text{O}_3^{2-}$, F⁻, NO_3^- , SO_4^{2-} , PO_4^{3-} , tartrate,

citrate, oxalate and tiron; moderate amounts of Cr(VI), Cr(III), Zn(II) and Cd(II); and small amounts of Mn(II), Sn(II), Cu(II), Al(III), ascorbic acid and SCN^- are tolerable. Ni(II), Fe(II,III), V(IV,V), Ga(III), In(III), and Tl(III) interfere seriously at a ratio of 1:1 with respect to Co(II). However, the interfering effect of some of these ions can be reduced by masking with oxalate, citrate or EDTA (see Table 2). Co-HTP-An-water-chloroform system are given in Table 2.

Table 2. Effect of foreign ions on the extraction of 20 μg cobalt (II)

Foreign ions and reagents (FI)	mg	FI-to-Co ratio	Co found	R,%	Foreign ions and reagents (FI)	mg	FI-to-Co ratio	Co found	R, %
Citrate ³⁻	5	250	20.03	100.5	Fe(II)	0.5	2.5	19.25	85.0
Oxalate ²⁻	10	200	20.13	102.6	Fe(III)	0.5	2.5	20.90	118.0
Tartrate ²⁻	2.5	250	5.05	101.0		1.0 ^a	20	20.05	101.0
Ascorbic acid	0.5	25	5.15	103.1	V(IV)	0.05	2.5	20.55	111.0
EDTA	0.5	25	5.10	102.0	V(V)	0.05	2.5	19.25	85.0
CDTA	0.005	0.25	19.73	94.6	Cd ²⁺	0.2	10	19.86	97.2
Tiron	2.5	125	20.11	102.5	Cu ²⁺	0.06	3	20.17	103.4
SCN ⁻	0.025	10	20.13	102.6		1.2 ^b	6	20.04	100.8
Cl ⁻	20	100	20.20	104.0	Al ³⁺	5	250	20.08	101.6
S ₂ O ₃ ²⁻	10	200	19.92	98.5		10 ^c	500	20.02	100.3
F ⁻	10	500	20.20	104.0		10 ^d	500	20.13	100.4
NO ₃ ⁻	20	1000	20.02	100.3	Zn ²⁺	0.5	25	20.04	100.8
SO ₄ ²⁻	20	500	20.03	100.5		1.0 ^e	50	20.18	103.5
PO ₄ ³⁻	7	225	20.10	102.0	Zr(IV)	3.0	150	20.18	103.5
ClO ₄ ⁻	0.1	0.5	20.73	94.6		15 ^f	500	20.01	100.2
NH ₄ ⁺	20	500	20.02	100.3	Nb(V)	0.5	2.5	19.25	85.0
Na ⁺	30	1500	20.13	100.4		10 ^f	500	20.04	100.8
K ⁺	30	1500	20.11	102.2	Ti(IV)	2.5	125	20.17	103.4
Ca ²⁺	15	500	20.01	100.2		5 ⁱ	250	19.88	97.7
Ba ²⁺	5	250	20.15	103.1	Ni ²⁺	2.5	125	19.91	98.2
Sr ²⁺	20	100	20.11	102.2	Cr(VI)	2.5	125	19.91	98.2
Mg ²⁺	30	1500	20.18	103.5	Cr(III)	1.5	75	19.80	96.0
Mo(VI)	5	250	19.85	97.0	W(VI)	5	250	19.88	97.7

^a in the presence of 2.5 mg oxalate;

^b in the presence of 0.75 mg $\text{SC}(\text{NH}_2)_2$; 10 min extraction time;

^c in the presence of 2.5 mg oxalate;

^d in the presence of 3.0 mg citrate;

^e in the presence of 0.75 mg EDTA; 15 min extraction time;

^f in the presence of 3.0 mg NaF;

ⁱ in the presence of 3.0 mg Ascorbic acid.

Effect of Cobalt (II) Concentration. 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 Co(II) may be determined in the range 0.5-100 $\mu\text{g}/\text{ml}$ (table 1). Table 3 summarizes the calibration characteristics obtained with HTPD+An. With the increase of the basic amine (pKa) complexes improved analytical parameters (pKa(An)=4.58, pKa(mAn) = 4.85).

Table 3. Analytical characteristics of some ternary complexes of Co with o-hydroxythiophenol derivatives in the Presence of aniline

Compound	LOD*: $\text{ng} \cdot \text{mL}^{-1}$	LOQ*: $\text{ng} \cdot \text{mL}^{-1}$	SS*: $\mu\text{g} \cdot \text{cm}^{-2}$	Beer's law range ($\mu\text{g} \cdot \text{mL}^{-1}$)	The equation of calibration curves
Co-HTP-An	15	53	2.30	0.05-2.8	0.045+0.110x
Co-HXTP-An	14	46	2.22	0.05-2.8	0.056+0.107x
Co- HTSA -An	13	42	2.17	0.05-2.8	0.069+0.103x
Co- HTBA -An	13	42	1.95	0.05-3.0	0.045+0.131x
Co- HNTP -An	12	39	1.87	0.05-3.2	0.054+0.133x

Note*: LOD- Limit of detection; LOQ - Limit of quantification; SS-Sandell's sensitivity.

The proposed method compares favourably with the existing ones (Table 4) and offers the advantages of better simplicity, rapidity, sensitivity and selectivity [2-6].

Table 4. Comparative characteristics of the procedures for determining of cobalt

Reagent [Ref.]	pH (solvent)	λ , nm	$\epsilon \cdot 10^{-4}$	Beer's law range ($\mu\text{g} \times 5\text{cm}^{-3}$)
Nitroso-R-salt [2]	weakly acidic medium	415	3.5	0.7-29
1-nitroso-2-naphtol [2,4]	≥ 3	415	2.9	0.8-56
2,3,5-triphenyl-2H-tetrazolium chloride [3]	5.2-5.8	525	4.26	0.2 - 75
2-nitroso-1-naphtol [2]	≥ 4	365	3.7	0.1-60
HTSA -An	2.3-3.8(CHCl_3)	540	3.12	0.6-95
HTBA -An	2.4-3.5 (CHCl_3)	556	3.49	0.5-80
HNTP -An	1.9-3.1 (CHCl_3)	558	3.72	0.6-80

4. Analytical Applications

The proposed method under the already established optimum conditions was applied for the determination of Co(II) in various objects. The results presented in Table 5 and Table 6 indicate the successful applicability of the proposed method to real sample analysis.

Determination of Cobalt (II) in Steel. A weighed sample of 0.2 g was dissolved in 20 ml of H₂SO₄ (1: 1) was oxidized with a few drops of concentrated nitric acid and evaporated twice to vapor SO₃. The precipitated salt was dissolved in 20 ml of 15% tartaric acid under heating, the solution was cooled, adjusted with water to 100 ml in a volumetric flask, stirred and filtered. An aliquot of 5 ml was put into a separatory funnel, was added 1 ml of 10% hydroxylamine solution, 1 ml of 3% ascorbic acid and was determined cobalt using the proposed procedures.

Determination of Co (II) in Sewage Water and Bottom Sediments. 11 taken for analysis of waste water is evaporated to obtain a precipitate, do not boil. The precipitate was dissolved in 5 ml of HNO₃, was transferred to a 50 ml flask and diluted to the mark with water.

Table 5. Determination of Cobalt in steel (n=6, P=0.95)

Compound	Steel	X	S _x	ε	S _r	μ
Co-HTP-An	M 441 (0.012 %Co)	0.0124	3.12×10 ⁻⁴	3.28×10 ⁻⁴	0.025	(1.24±0.0328) ×10 ⁻²
Co-HXTP-An		0.0129	3.27 ×10 ⁻⁴	3.31×10 ⁻⁴	0.022	(1.29±0.0331) ×10 ⁻²
Co- HTSA -An		0.0118	3.16 ×10 ⁻⁴	3.45×10 ⁻⁴	0.017	(1.18±0.0345) ×10 ⁻²
Co- HTSA -An	№156 (0.56 % Co)	0.520	0.026	0.021	0.037	0.520±0.0210
Co- HTBA -An		0.569	0.020	0.027	0.033	0.569±0.027
Co- HNTP -An		0.552	0.029	0.021	0.049	0.552±0.021

Table 6. Determination results of cobalt (II) in the Sewage water and Bottom sediments (n = 6, P = 0.95)

Compound	Analysis object	Added, μg	Found, μg	Found in the sample, μg / kg	S _r
				$\bar{X} = \pm \frac{t_p S}{\sqrt{n}}$	
Sewage water					
Co- HTBA -An	Sample 1	2.0	2.45	0.45±0.05	0.06
Co- HNTP -An	Sample 2	5.0	6.14	1.14±0.11	0.07
Bottom sediments					
Co- HTBA -An	Sample 1	5	6.26	1.26±0.05	0.05
Co- HNTP -An	Sample 2	5	6.92	1.92±0.04	0.08

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