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RESEARCH INTO REACTIONS OF CHROME (VI) COMPLEXATION WITH 2-HYDROXY-5-IODIOPHENOL IN THE PRESENCE OF HYDROPHOBIC AMINES

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Spectrophotometric methods were used to study the complexation of chromium (VI) with 2-hydroxy-5-iodothiophenol (HITP) in the presence of hydrophobic amines. Out of hydrophobic amines (Am), there were used *o*-xylidine and *m*-xylidine. Chloroform, dichloroethane and chlorobenzene proved to be the best extractants. Following a single extraction with chloroform, 97.4-98.8% of chromium was extracted as an ionic associate. It revealed that mixed ligand complexes are formed in a weakly acidic medium (pH 0.2-6.9). For the formation and extraction of MLC, a 26-30-fold excess of complexing agents is required. For example, optimal conditions for the formation and extraction of these compounds are provided at 1.0×10^{-3} M HITP and $(1.12-1.16) \times 10^{-3}$ M Am. Maximum in the absorption spectrum of the complexes is observed at $\lambda = 475-492$ nm. Molar absorption coefficients are $(3.1-3.3) \times 10^4$. The stoichiometric ratio of the reacting components is Cr:HITP:Am = 1:3:3. The Beer's law is in effect in the range of 0.5-20 $\mu\text{g/ml}$ chromium concentrations. Based on the evidence found, photometric methods for the determination of chromium in soils were developed.

Keywords: chromium, 2-hydroxy-5-iodothiophenol, xylidin, mixed-ligand complexes, extraction-photometric method

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Introduction

Chromium is one of the toxic metals. Chromium (VI) compounds which are among the most dangerous pollutants of natural objects, have the most carcinogenic effect [1-2]. In this regard, in modern practical chemical analysis there is a need for the definition of this element-pollutant. Special control require

drinking, natural and industrial water, the chromium content in which is strictly regulated. To assess the content of toxic components in various environmental objects, the methods based on the determination of elements with organic reagents are quite promising and actively developing [3].

Table 1. Characteristics of previously spectrophotometric methods for chromium determination

Reagent*	Linear range/ mg L^{-1}	λ_{max} , nm	Molar absorptivity/ L $\text{mol}^{-1}\text{cm}^{-1}$ $\epsilon \times 10^{-4}$	Relative standard deviation/ %	Ref.
Cr-EDTA	6.0-160	485	0.21	0.6	[4]
Cr-5-Br-DMPAP	0.02- 0.56	546	7.8	2	[4]
Cr-PAR	—	540	4.7	-	[4]
Cr-PAN	0.3-2.0	420	1.28	1.25	[4]
Cr-TAR	3.4-79	390	4.51	0.95	[5]
Cr- HBTP- Ap1	4.1-5.3	470	2.92	0.89	[6]
Cr- HBTP- Ap2	4.0-5.2	465	3.28	0.95	[6]

*Note: EDTA – ethylenediamine tetraacetic acid; TAR – 4-(2- Thiazolylazo)-resorcinol; PAR – 4-(2-pyridylazo)resorcinol; PAN – 1-(2-Pyridylazo)-2-naphthol; 5-Br-DMPAP – 2-(5-bromo-2-pyridylazo)-5-dimethylaminophenol; HBTP – 2-hydroxy-5-bromothiophenol; Ap1 – N, N-dimethylaminomethyl -4-methylphenol; Ap2 – N, N-dimethylaminomethyl -4-chlorophenol.

The paper deals with research into conditions of interaction of chromium (VI) with 2-hydroxy-5-iodothiophenol (HITP, H₂L). In the presence of hydrophobic amines, extraction of these compounds in the organic

phase in the form of a mixed-ligand complex (MLC) is observed. Out of hydrophobic amines (HAs): *o*-xylydine (*o*-xyl) and *m*-xylydine (*m*-xyl) are used.

Materials and methods

Reagents. A solution of Chromium (VI) ($1 \mu\text{g mL}^{-1}$) was prepared through dissolving (0.1935) gm of K₂Cr₂O₇ p. f. a. (pure for analysis) in 1000 ml of water. Concentration of Chromium solution was established gravimetrically [7]. Working solution with concentration of 0,1 mg/ml was prepared by dilution of stock with deionized water. HITP was synthesized according to the method [8]. The purity was checked by paper chromatography and melting temperature determination. Chloroform was distilled before use. Chloroform solutions of HITF (0.01 mol/l) and Am (0.02 mol/l) were used. To create optimal acidity, 0.1 mol/l solutions of HCl, NaOH, or ammonium acetate {CH₃COOH CH₃COONH₄ (pH 4-12)} were used.

Apparatus. The absorption of the extracts was read using a photocolorimeter KFK-2 and a spectrophotometer SP-26. For pH determination, an I-120.2 ionometer was used with a glass electrode.

Procedure. Portions of stock solutions of chromium (III) 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 HBTP, and a 2.5 mL portion of a 0.01M solution of Am were placed into calibrated test tubes with ground-glass stoppers (volume of the organic-phase was 5 mL). A required value of pH was adjusted by adding 1M HCl. The volume of the aqueous phase was increased up to 20 mL using distilled water. In 15 minutes after 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 490 nm

($l=0.5\text{cm}$).

Determination of Chromium (III) in soils. The proposed procedures for the determination of Chromium were applied in light-chestnut soil from the Caspian zone. A 0.5-g weight was finely ground in an agate mortar and calcined in a muffle furnace for 3 h. after cooling, the sample was treated and dissolved in an graphite cup with a mixture of 16 mL of conc. HF, 5mL of conc. HNO₃, and 15 mL of conc. HCl at 50-60⁰C to remove excess hydrogen fluoride, a 8 mL portion of conc. HNO₃ was added triply to the solution being every time evaporated to 5-6mL. After that, the solution was transferred to a 100 mL volumetric flask and its volume brought to the mark with distilled water. Chromium was determined in aliquot portions of the solution using the proposed procedures.

Chromium oxidation studies. It is known that hydroxyhalogenthiofenol has reducing properties in an acidic environment [6, 8]. Previous studies with Cr(VI)-H₂L-HAm and Cr(III)-H₂L-HAm showed that only Cr(III) forms stable complexes with this reagent. To determine the degree of oxidation of chromium in the presence of HITP, we conducted two series of experiments. In the first series, we used Cr(VI), and in the second series -- Cr(III) obtained by adding an additional reducing agent (SnCl₂ or KI). A comparison of the spectra obtained showed that $\lambda_{\text{max}} [\text{Cr(VI)-H}_2\text{L-HAm}] = \lambda_{\text{max}} [\text{Cr(III)-H}_2\text{L-HAm}]$. This fact can be considered as an indication [6, 8] that Cr(VI) is reduced to Cr(III) by the reagent itself during complex formation.

Results and discussion

Choice of solvent. For the extraction of complexes we used CHCl₃, CCl₄, C₆H₆, C₆H₅CH₃, C₆H₄(CH₃)₂, C₂H₄Cl₂, *iso*-butanol

and *iso*-pentanol. Extractability of complexes was estimated at coefficient of distribution (D) and extent of extraction (R%) [9]:

$$D = \frac{[W]_{org}}{[W]_{aq}}; \quad R = \frac{100 \times D}{D + \frac{V_{aq}}{V_{org}}}$$

The extractivity of the complexes was estimated by means of distribution and recovery coefficients. Chloroform, dichloroethane and chlorobenzene appeared to be the best extractants. Further investigations were carried out with chloroform. After a single extraction with chloroform, 97.4-98.8% of chromium was extracted as an ion associate. The chromium content in the organic phase was determined photometrically from diphenylcarbazide [1] after desorption in the aqueous phase by difference.

Influence of the pH of the aqueous phase. Study into dependence of a complex formation from pH showed that the exit of

complexes of chromium is maximum at pH 2.8 – 4.7. Extraction of Cr (III) rose as the acidity of the initial solution increased; further rise in acidity led to gradual decrease of recovery which was obviously associated with decrease in the concentration of the ionized form of HITP. Probably, it is present in the solution in the non-dissociated state. At $\text{pH} \geq 7$, the complexes were hardly extracted probably due to decrease in the degree of Am protonation. The effect of pH on the intensity of the color reaction is shown in Fig. 1 and Table 2. Existence of one maximum of absorbance in the specified limits pH reaffirms the formation of one complex connection.

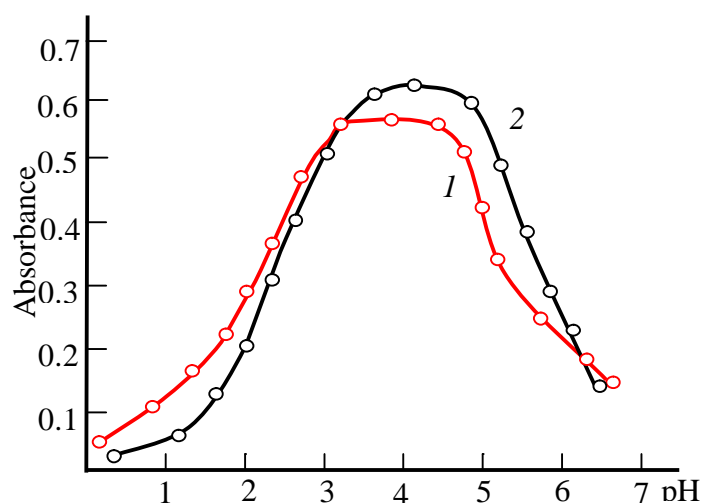


Fig. 1. Absorbance of mixed-ligand complexes as a function of the pH of the aqueous phase: 1- Cr(III)-HITP-*o*-xyl; 2- Cr(III)-HITP-*m*-xyl. $C_{Cr} = 3.84 \times 10^{-5}$ M, $C_{HITP} = 1.0 \times 10^{-3}$ M, $C_{Am} = (1.12-1.16) \times 10^{-3}$ M, KFK-2, $\lambda = 490$ nm, $\ell = 0.5$ cm.

Effect of reagent concentration. For the formation and extraction of MLC, a 26-30-fold excess of complexing reagents is required; for example, the optimal conditions for formation and extraction of these compounds are provided by 1.0×10^{-3} M HITP and $(1.12-1.16) \times 10^{-3}$ M Am. Note that a larger excess of hydrophob amin interferes with the determination. However, it was found that excess in the reagent solution does not alter absorbance of the color reaction.

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 metal ion concentration indicates that Cr(III) may be determined in the range 0.5-20.0 $\mu\text{g/ml}$.

Resistance to dyeing and extraction time. Unlike single-ligand complexes, mixed-ligand complexes of Cr (III) with HITP and Am were stable in aqueous and organic

solvents and did not decompose for two days duration of the phase contact was 15 min. or over a month after extraction. The required

Table 2. Optimum conditions of education and analytical characteristic of chromium MLC with HITP and Am.

Compound	Optimum pH interval	λ_{\max} / nm	$\varepsilon \times 10^{-4}$ / $L \text{ mol}^{-1} \cdot \text{cm}^{-1}$	Working range / $\mu\text{g/ml}$
Cr-HITP- o-Xyl	3.1-4.5	475	3.3	0.5-20
Cr-HITP- m-Xyl	3.5-4.7	492	3.1	0.5-20

Electronic absorption spectra.

Neither metal ion nor reagent has appreciable absorbance at specified wavelengths. Hence, further studies were carried out at 475-492 nm. The reagent has minimum absorbance in terms of complex maximum absorbance. Thus, further absorbance measurements were made at 490 nm. The molar absorptivity of the complex was calculated by means of Komar method [10] to be $\varepsilon = (3.1-3.3) \times 10^4 L \text{ mol}^{-1} \text{ cm}^{-1}$.

Stoichiometry of the complexes and the mechanism of complexation.

It was found, using the Nazarenko method, that Cr(III) in the complexes was present in the three charging cations Cr^{3+} . The number of protons substituted by chromium in one HITP molecule turned out to be one [12].

The stoichiometric coefficients of the reaction were determined by the Asmus method, the relative yield method and the equilibrium shift method [10] (Fig. 2).

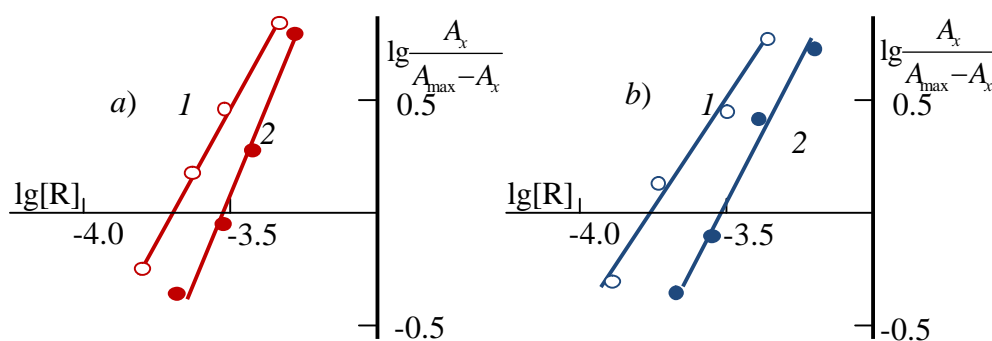
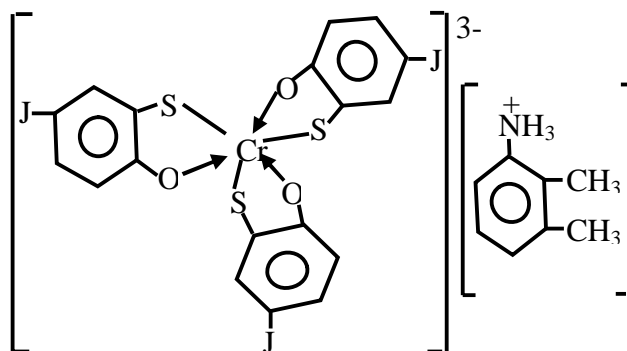


Fig. 2. Determination of the ratio of components by the equilibrium shift method for (a) Cr(III)- HITP-o-xyl and (b) Cr(III)-HITP-m-xyl. 1. Cr:HITP; 2. Cr : Am.

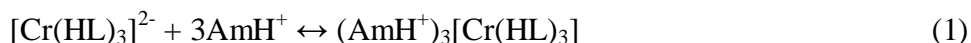
The formation of MLC can be offered as follows. When chromium ion interacts with three molecules of o-hydroxythiophenols, they form three-charged anionic complexes which

are extracted with three molecules of protonated Am. The structure of the extracted complexes is shown below:

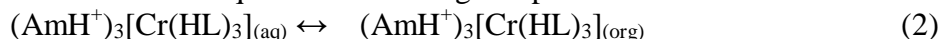


Several equilibrium processes should be taken into account for the system of $[\text{Cr}(\text{HL})_3]^{2-}$ - *o*-XylH⁺ - H₂O-CH₃Cl. Formation of ion-

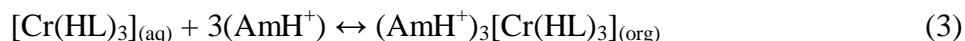
association complex in the aqueous phase is as follows:



Distribution of the complex between the aqueous and the organic phases:



Extraction from water into chloroform:



Based on the equation as follows:

$$\beta = \frac{\{[\text{Cr}(\text{HL})_3](\text{AmH})_3\}}{[\text{Cr}(\text{HL})_3^{2-}][\text{AmH}^+]^3} \quad (4)$$

$$K = \frac{\{[\text{Cr}(\text{HL})_3](\text{AmH})_3\}_{\text{org}}}{\{[\text{Cr}(\text{HL})_3](\text{AmH})_3\}_{\text{aq}}} \quad (5)$$

$$K_{\text{ex}} = K_D + \beta = \frac{\{[\text{Cr}(\text{HL})_3](\text{AmH})_3\}_{\text{org}}}{\{[\text{Cr}(\text{HL})_3^{2-}]\}_{\text{aq}}\{(\text{AmH}^+)\}_{\text{aq}}} \quad (6)$$

Note that constants of the association β were determined by means of independent methods: Mobile equilibrium method [12], Holme-Langmyhr method [13], Komar-Tolomachev Method [10] and Harvey-Manning method [14].

Constants of the distribution K_D were determined by comparison of the absorbance

values obtained due to a single extraction under optimum conditions (A_1) and triple extraction (A_3): $K_D = A_1/(A_3 - A_1)$. Extraction constants were calculated by the equation $K_{\text{ex}} = \beta + K_D$ [10]. All calculations were carried out at a probability of 95%. Obtained values are shown in Table 3.

Table 3. Values of the extraction constants (K_{ex}), distribution constants (K_D), association constants (β) and recoveries (R%) for the Cr(III)- HTPD -Am-water-chloroform systems

Extraction system	lg β	lg K_D	lg K_{ex}	R%
Cr(III)-H ₂ L- <i>o</i> -xyl -H ₂ O-CHCl ₃	9.57±0.2 ^a	1.86±0.01	11.43±0.33 ^e	98.7
	9.82±0.4 ^b		11.69±0.23 ^f	
	9.34±0.7 ^c			
	9.39±0.3 ^d			
Cr(III)-H ₂ L- <i>m</i> -xyl -H ₂ O-CHCl ₃	9.28±0.3 ^b	1.42±0.08	10.56±0.38 ^e	97.4
	9.32±0.5 ^c			

Note: ^aCalculated by the Holme-Langmyhr method [13]; ^bCalculated by the Harvey-Manning method [14]; ^cCalculated by the Komar-Tolmachev method [10]; ^dCalculated by the mobile equilibrium method [12]; ^eCalculated by the formula $K_{\text{ex}} = K_D + \beta$ where b is determined by the Holme-Langmyhr method [29]; ^fCalculated by the formula $K_{\text{ex}} = K_D + \beta$ where b is determined by the Harvey-Manning method [14].

Calculation of extent of polymerization of complexes was carried out on the equation. The made calculations showed that MLC in an organic phase won't be polymerized and are in a monomeric form ($\gamma = 1.02-1.18$).

Influence of interfering ions. To evaluate the complex applicability for photometric determination of chromium, 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 NO₃⁻, ClO₄⁻, SO₄²⁻ and CH₃COO⁻ do not interfere determination of chromium with HTPD and Am. Interference of most cations is masked by the addition of complexone III. Tartrate mask the milligram quantities of Ta, Ti, W and Mo. Zr fluorides should mask, and copper-thiourea.

Analytical characteristics. Equations of obtained straight lines and some important characteristics over application of ternary complexes for extractive-spectrophotometric determination of Cr(III) are listed in Table 4.

Table 4. Analytical characteristics

Characteristic	Extraction system	
	Co-HIPT- o-xyl	Co-HIPT- m-xyl
Apparent molar absorptivity (ϵ), $L \text{ mol}^{-1} \text{ cm}^{-1}$	3.3×10^4	3.1×10^4
Adherence to Beer's law, $\mu\text{g mL}^{-1}$	0.5-20	0.5-20
Equation of the straight line	$0.1237x+0.062$	$0.1254x+0.038$
Coefficient of correlation	0.9995	0.9992
Sandell's sensitivity, ng cm^{-2}	0.00852	0.00748
Limit of detection, $\mu\text{g mL}^{-1}$	13	15
Limit of quantification, $\mu\text{g mL}^{-1}$	40	42

Table 5 demonstrates the data which allow a comparison of the analytical parameters of the procedures for the determination of chromium with the earlier known procedures [4].

Table 5. Comparative characteristics of procedures for determining chromium

Reagent	pH (solvent)	λ , nm	$\epsilon \cdot 10^{-4}$	Beer's law range, μg	[Ref.]
5-Br-DMPAP	0.1–10 M HCl (CHCl_3)	546	7.8	0.02 – 0.56	4
PAR	4-5	540	4.7	3.2-13.0	4
PAN	0.2–0.8 M HCl (aseton)	420	1.28	0.3 – 2.0	4
<i>Proposed method</i>					
Cr-HITP- o-Xyl	3.1-4.5(CHCl_3)	475	3.3	0.5-20	
Cr-HITP- m-Xyl	3.5-4.7(CHCl_3)	492	3.1	0.5-20	

Analytical applications. The proposed method in the already established optimal conditions was applied to determine Cr (III) in soils. Also, procedures for determining chromium in soils were verified by diphenylcarbazide methods. The results of the analysis are listed in Table 6 to indicate successful applicability of the proposed method for real sample analysis.

Table 6. Correctness and reproducibility of determination of chromium in soil ($n=5$, $P=0.95$)

Method	$\bar{X} \pm \frac{t_p \cdot S}{\sqrt{n}}$, % $\times 10^{-4}$	SD $\times 10^{-4}$	RSD, %	$\bar{X} \pm \frac{t_p \cdot S}{\sqrt{n}}$
<i>Standard method</i>				
Diphenylcarbazide	2.89	0.156	6	(2.89 \pm 0.027)
<i>Proposed method</i>				
HITP-o-xyl	2.80	0.123	7	(2.80 \pm 0.013)
HITP-m-xyl	2.84	0.141	5	(2.84 \pm 0.032)

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XROMUN 2-HİDROKSİ-5-YODTİOFENOL VƏ HİDROFOB AMİN İŞTİRAKINDA KOMLEKSƏMƏLƏGƏLMƏ REAKSİYASININ TƏDQIQI

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Xromun 2-hidroksi-5-yodtiofenol (HYTF) və hidrofob aminlərlə (Am) kompleks əmələgəlmə reaksiyaları spektrofotometrik tədqiq edilmişdir. Hidrofob aminlərdən o-ksilidin və m-ksilidin istifadə edilmişdir. Ən yaxşı ekstragent kimi xloroform, dixloretan və xlorbenzol müəyyən edilmişdir. Xloroformla birdəfəlik ekstraksiyada xromun 97.4-98.8%-i ion assosiat şəklində ekstraksiya olunur. Müəyyən edilmişdir ki, ion assosiatlar zəif turş mühitdə əmələ gəlir (pH 0.2-

6.9). İon assosiatların əmələgəlməsi və ekstraksiyası üçün kompleksəmələgətirici reagentlərin 26-30 dəfə artığı tələb olunur. Bu birləşmələrin əmələgəlməsi və ekstraksiyası üçün 1.0×10^{-3} M HİTP u $(1.12-1.16) \times 10^{-3}$ M Am tələb olunur. Udma spektrində maksimum $\lambda = 475-492$ нм- də müşahidə olunur. Molyar işıqdurma əmsalı $(3.1-3.3) \times 10^4$ təşkil edir. Qarşılıqlı təsirdə olan komponentlərin stexiometrik nisbəti Cr: HİTP: Am = 1:3:3 kimidir. Ber qanununa tabe olma xromun 0.5-20 mkq /ml qatılığında müşahidə olunur. Alınan nəticələr əsasında torpaqda xromun təyini üçün fotometrik metodlar işlənilib hazırlanmışdır.

Açar sözlər: xrom, 2-hidroksi-5-yoditiofenol, ksilidin, qarışıq-ligand komplekslər, ekstraksiya-fotometrik metod

ИЗУЧЕНИЕ РЕАКЦИИ КОМПЛЕКСООБРАЗОВАНИЯ ХРОМА (VI) С 2-ГИДРОКСИ-5-ИОДТИОФЕНОЛОМ В ПРИСУТСТВИИ ГИДРОФОБНЫХ АМИНОВ

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Спектрофотометрическими методами изучена реакция комплексообразования хрома (VI) с 2-гидрокси-5-йодтиофенолом (HИTP) в присутствии гидрофобных аминов. Из гидрофобных аминов использованы (Am): α -оксилидин и м-ксилидин. Хлороформ, дихлорэтан и хлорбензол были лучшими экстрагентами. После однократной экстракции хлороформом 97.4-98.8% хрома экстрагировали в виде ионного ассоциата (ИА). Установлено, что ИА образуются в слабокислой среде (рН 0.2-6.9). Для образования и экстракции ИА требуется 26-30-кратный избыток комплексообразующих агентов; например, оптимальные условия для образования и извлечения этих соединений обеспечиваются 1.0×10^{-3} М HИTP и $(1.12-1.16) \times 10^{-3}$ М Am. Максимум в спектре поглощения комплексов наблюдается при $\lambda = 475-492$ нм. Молярные коэффициенты поглощения составляют $(3.1-3.3) \times 10^4$. Стехиометрическое соотношение реагирующих компонентов составляет Cr: HИTP: Am = 1:2:2. В диапазоне концентраций хрома 0.5–20 мкг/мл наблюдается закон Бера. На основании полученных данных разработаны фотометрические методы определения хрома в почвах.

Ключевые слова: хром, 2-гидрокси-5-йодтиофенол, ксилidin, смешано-лигандные комплексы, экстракционно-фотометрический метод