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SPECTROPHOTOMETRIC RESEARCH INTO COMPLEXATION OF TUNGSTEN(VI) WITH *o*-HYDROXYTHIOPHENOL DERIVATIVES IN THE PRESENCE OF HYDROPHOBIC AMINES

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Abstract: Spectrophotometric methods were used to study the reaction of complexation of tungsten with derivatives of *o*-hydroxythiophenol (HTPDs) {2-hydroxy-5-chlorothiophenol (HCTP), 2-hydroxy-5-bromothiophenol (HBTP) and 2-hydroxy-5-iodothiophenol (HITP)} in the presence of aminophenols. Aminophenols used were 2(N,N-dimethylaminomethyl)-4-chlorophenol (AP1) and 2(N,N-dimethylaminomethyl)-4-bromophenol (AP2), 2(N,N-dimethylaminomethyl)-4-iodophenol (AP3). Optimal conditions for the formation and extraction of mixed ligand complexes (MLC) were found, and the ratios of the components in the complexes established. It revealed that MLC are formed in a weakly acidic environment (pH_{opt} 4.5-5.5). The maximum in the light absorption spectrum is observed at $\lambda=460-490$ nm. The molar coefficient of light absorption is equal to $\epsilon = (2.0-2.8) \times 10^4$. Optimal conditions for the formation and extraction of these compounds is the concentration of $(1.2-2.5) \times 10^{-3}$ mol / l GTP and $(2.0-2.8) \times 10^{-3}$ mol/l AP. The maximum optical density is reached within 5-8 minutes. Tungsten MLC extracts with HTP and AP obeys Beer's law at concentrations of 0.04–3.8 $\mu\text{g}/5$ ml. The proposed method was applied to determine Tungsten in steel and in soils.

Keywords: Tungsten, *o*-hydroxythiophenol derivative and hydrophobic amines, extraction-photometric method, detection limit, determination.

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Introduction

Spectrophotometric methods [1-5] are among the most precise instrumental methods of analysis for the determination of elements in trace amounts. These methods are remarkable for their versatility, sensitivity and precision. Owing to these methods it is possible to cover a very extensive range of concentrations for the trace analysis of the elements. There are various spectrophotometric methods which suffer from low sensitivity, non-selectivity and complexity in the procedures for the determination of tungsten in trace amounts using thiocyanate, vanadophosphoric acid as ligands [1,3]. Many organic reagents are used as analytical reagents for spectrophotometric determination of tungsten but most of these are not suitable for routine analysis of the metal ion due to one or more of the above drawbacks [1,3,6]. This

circumstance made it necessary to search for new better methods and accordingly, we have synthesized several very effective reagents - *o*-hydroxythiophenol derivative (HTPDs, L) {2-hydroxy-5-chlorothiophenol (HCTP), 2-hydroxy-5-bromothiophenol (HBTP), and 2-hydroxy-5-iodothiophenol (HITP)} has been used as a complexing agent for tungsten (VI) in the spectrophotometric determination of metal ion to meet the above requirements.

In the present paper, we report results from liquid-liquid extraction-spectrophotometric experiments on 9 different systems, each of which containing W(VI), a HTPDs and an *o*-aminophenols (AP) {As aminophenole 2(N, N-dimethylaminomethyl)-4-chlorophenol (AP1) and 2(N, N-dimethylaminomethyl)-4-bromophenol (AP2), 2

(N, N-dimethylaminomethyl) -4-yodophenol (AP3) were used}. We suggest new procedures for determining tungsten in soils and plants.

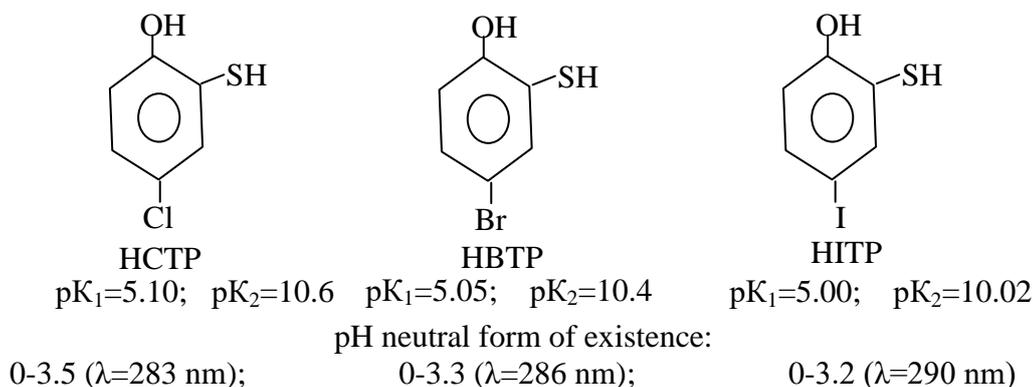
Experimental part

Reagents and solutions. A standard stock solution (100 mL) of tungsten (VI) containing 1 mg mL⁻¹ of the metal ion is prepared by dissolving an accurately weighed amount (0.179g) of sodium tungstate (A.R) in distilled water. The concentration of the tungsten solution was adjusted gravimetrically by sedimenting tungsten in the form of H₂WO₄ and weighing WO₃ [2, 7]. Lower concentrations at µg mL⁻¹ (10, 20, 50 and 100) level are prepared by suitable dilution there from.

Chloroform was purified by washing

with conc. H₂SO₄ and shaking with distilled water followed by washing with a 5% solution of NaOH. Chloroform is used for extraction of W(VI) – HTPDs-AP complex. HTPDs is prepared by reported methods [8] as follows.

Complexing agents may be a dibasic (HCTP, HBTP, HITP, H₂L) weak acid, and depending on pH of the medium may be in molecular and anionic forms. Some characteristics of the investigated reagents are presented below:



Synthesized compounds were characterized by physicochemical methods: IR [16] and NMR spectroscopy:

HCTP - IR (KBr): 3460 cm⁻¹ ν(OH), 2570 cm⁻¹ ν(SH), 1580 cm⁻¹ ν(C₆H₅); 1H NMR (300.18 MHz, C₆D₆): δ 5.70 (s, 1H- OH), δ 3.40(s, 1H - SH), δ 7.05 (s, 2H Ar-H), δ 6.25 (s, 2H Ar-H).

HBTP - IR (KBr): 3458 cm⁻¹ ν(OH), 2568 cm⁻¹ ν(SH), 1550 cm⁻¹ ν(C₆H₅); 1H NMR (300.18 MHz, C₆D₆): δ 5.70 (s, 1H- OH), δ 3.40(s, 1H - SH), δ 7.05 (s, 2H Ar-H), δ 6.25 (s, 2H Ar-H).

HITP- IR (KBr): 3456 cm⁻¹ ν(OH), 2578 cm⁻¹ ν(SH), 1570 cm⁻¹ ν(C₆H₅); 1H NMR (300.18 MHz, C₆D₆): δ 5.55 (s, 1H- OH), δ 3.32(s, 1H- 1SH), δ 6.80 (s, 1H Ar-H), δ 7.30 (s, 2H Ar-H).

Apparatus. An optimum acidity was created by means of 0.01 M HCl or an ammonium acetate buffer solution and

controlled using an I-120.2 potentiometer equipped with a glass electrode. The absorbance of the extracts was measured using a KFK-2 photocolormeter (USSR), a SF-26 spectrophotometer (USSR), equipped with 5 and 10 mm pathlength cells. Muffle furnace was used for dissolution of the samples.

Methods. Portions of a working tungsten solution, 0.1 to 0.8 mL with an increment of 0.1 mL, 1.0-2.0 mL of 0.01 M HCl, and 0.3-0.5 mL of a 0.01 M HTPDs solution were placed in calibrated test tubes with ground stoppers. The mixture was stirred thoroughly for 5 min to reduce tungsten, then 2.0-3.0 mL of a 0.01 M solution of AP was added. The volume of the organic phase was adjusted to 5 mL with chloroform, and the volume of the aqueous phase was brought to 20 mL with distilled water. Following the formation of the hydroxythiophenolate complex of tungsten, it was extracted, and the absorbance

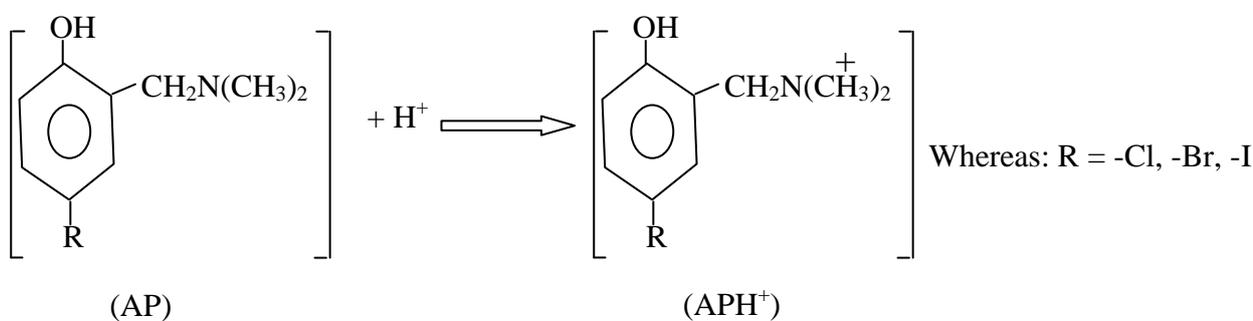
of extracts was measured by a KFK-2 photoelectrocolorimeter at 490 nm.

Results and Discussion

Research into the oxidation state of tungsten. In acidic medium, HTPDs exhibit reducing properties [9-11]. Hence, in the complex formation with HCTP, tungsten (VI) is reduced to tungsten (V) by the reagent itself. This fact was confirmed by ESR spectrometry [11].

Charge of the complex tungsten. The present study deals with the investigation of W (V) interaction with HTPDs, resulting in the formation of colored complexes insoluble in nonpolar solvents. To determine the sign of the

complex charge, ion exchange chromatography was used: AV-17 anion exchanger in chloroform absorbs a part of solution; the chromatographic column is colored in orange and tungsten is not detected in the filtrate. The anion complex is extracted in the presence of a hydrophobic amine, wherein the complex stability increases, and the color becomes more saturated. To neutralize the charge of the anion complex, aminophenol is used as a hydrophobic amine, which transforms into an aminophenol ion in acidic medium as follows:



The Choice of the Extractant. To extract complexes, we used CHCl_3 , $\text{C}_2\text{H}_4\text{Cl}_2$, CCl_4 , C_6H_6 , $\text{C}_6\text{H}_5\text{-CH}_3$, $\text{C}_6\text{H}_5\text{Cl}$, *iso*- $\text{C}_4\text{H}_9\text{OH}$, *iso*- $\text{C}_5\text{H}_{11}\text{OH}$, *n*- $\text{C}_4\text{H}_9\text{OH}$ and their mixes.

Extractability of complexes was assessed by coefficient of distribution (D) and extent of extraction (R%) [9]:

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

Chloroform, dichloroethane and chlorobenzene appeared to be the best extractants. Fast division of layers and the maximum value of molar coefficient of absorption were received at extraction of complexes by chloroform. After a single extraction with chloroform about 96.2-98.8% of tungsten was extracted as an ion associate. Further studies were carried out with chloroform. The concentration of tungsten in the organic phase was determined with dithiol by photometric measurements after back

extraction, while in the aqueous phase it was determined by the difference.

Influence of the pH of the Aqueous Phase. Studying of dependence of a complex formation from pH showed that, the exit of complexes of tungsten is maximum at pH 4.5-5.5. Extraction of W (V) enhanced with the increase in the acidity of the initial solution but 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 o-hydroxythiophenol

derivatives. Probably, it is present in the solution in the non-dissociated state. At $\text{pH} \geq 7.1$ the complexes were hardly extracted, perhaps, owing to the decrease in the degree of AP protonation. The effect of pH on the intensity of the colour reaction is shown in Fig.1 (Table 1). It could be seen that W(V)-HCTP-Am species are extracted in a great extent at pH values in the range 2.5-7.1 (with AP1), 2.3-6.9 (with AP2) or 2.1-6.8 (with AP3). W-HBTP-Am complexes are extracted at low pH: 2.2-6.9

(with AP1), 2.0-6.8 (with AP2) or 1.7-6.7 (with AP2). W- HITP-Am complexes are extracted at lower pH: 2.0-6.6 (with AP1), 1.8-6.4 (c AP2) or 1.5-6.2 (with AP3). Existence of one maximum peak of absorbance in the specified limits of pH reaffirms the assumption of formation of one complex connection. The nature of acids (HCl, H_2SO_4) does not practically influence a complex formation of Molybdenum with o-hydroxythiophenol derivatives and AP.

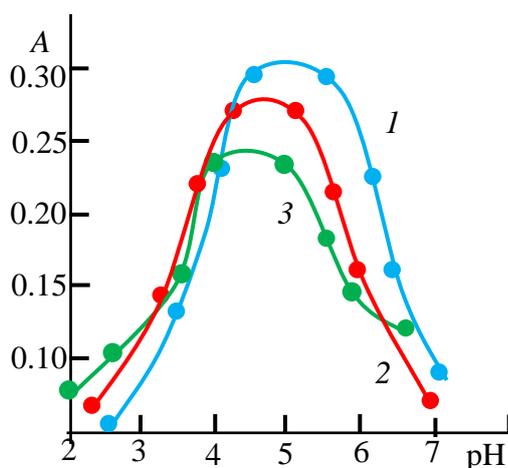


Fig. 1 Absorbance of W(V)-HTPDs-AP complexes in chloroform pH of the aqueous phase. 1 – W(V)-HCTP-AP1; 2 – W(V)-HBTP-AP1; 3 – W(V)-HITP-AP1. $C_W = 1.09 \times 10^{-5} \text{ mol L}^{-1}$, HTPDs = $(2.2-2.8) \times 10^{-3} \text{ mol L}^{-1}$, $C_{AP} = (2.0-2.5) \times 10^{-3} \text{ mol L}^{-1}$, $\lambda = 490 \text{ nm}$, SF-26, $\ell = 1.0 \text{ cm}$

Electronic Absorption Spectra. The absorption maxima (λ_{max}) of the ternary W(V)-HTPDs-AP complexes is identified within the range of 461-490 nm (Table 1). All color

reactions proved to be rather contrast since the initial solutions are colorless (λ_{max} (HTPDs) = 283-290 nm). Thus, bathochromic shift makes 171-207 nm.

Table 1. Optical characteristics, precision and accuracy of the spectrophotometric determination of W(V) with HTPDs and AP

Analytical characteristics	HCTP			HBTP			HITP		
	AP1	AP2	AP3	AP1	AP2	AP3	AP1	AP2	AP3
pH^1	2.5-7.1	2.3-6.9	2.1-6.8	2.2-6.9	2.0-6.8	1.7-6.7	2.0-6.6	1.8-6.4	1.5-6.2
pH^2	4.5-5.5	4.1-5.3	4.0-5.1	4.3-5.2	4.1-5.0	3.9-4.8	4.0-5.0	3.7-4.8	3.5-4.6
$R^3 / \%$	96.2	98.8	96.7	97.4	98.1	98.5	97.2	98.0	96.5
$\lambda_{\text{max}} / \text{nm}$	490	485	483	480	476	472	470	465	460
$\Delta\lambda_{\text{max}} / \text{nm}$	207	202	200	194	190	186	180	175	171
$\text{Ma}^4 / \text{L mol}^{-1} \text{cm}^{-1}$	2.8	2.7	2.6	2.4	2.3	2.3	2.2	2.1	2.0
Cp^5 / γ	1.01	0.95	1.03	1.05	1.06	1.07	1.09	0.99	1.02

Ec ⁶ /lgK _e	6.45	6.87	7.24	8.13	8.61	8.72	9.43	9.66	9.89
Sc ⁷ /lgβ	7.19	7.36	7.68	8.73	8.97	9.05	10.57		11.25
Wr ⁸ /μg cm ⁻³	0.5-95	0.5-90	0.5-95	0.5-90	0.5-85	0.5-80	0.5-85	0.5-80	0.5-80

Note: 1- pH range complexation; 2- The pH range of maximum extraction, 3- Degree of extraction; 4- Coefficient polymerization; 5- Molar absorptivity; 6- Equilibrium constant; 7- Stability constant; 8- Working range

Close values of maxima of light absorption make it possible to draw a conclusion that the formed complexes were ionic associates. Contrast of reactions was high i.e. initial reagents are colorless while complexes are intensively painted. Molar coefficients of absorption make up $(2.0-2.8) \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$.

Reagent Concentration and Incubation Time Influence. The effect of the concentration of HTPDs on the completeness of complexation was studied at the optimum pH and at a constant concentration of tungsten and AP. The optimum amount of AP for the maximum binding of the anionic hydroxyl thiophenolate complex of tungsten [W(V)-HTPDs] into an ionic associate was established by varying the quantity of AP added. For the formation of mixed-ligand complex (MLC) W(V)-HTPDs-AP, the concentration of $(1.2 - 2.5) \times 10^{-3} \text{ M}$ of HTPDs and $(2.0-2.8) \times 10^{-3} \text{ M}$ of AP in the solution is required.

MLC of W(V)-HTPDs-AP were stable

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

were given in Table 1.

The IR spectra of the complexes W-HCTP-AP1, in the field of $780-810 \text{ cm}^{-1}$ is indicative of an intensive strip of absorption caused by valent vibration of group $[O=W-Cl]^{2+}$ [14]. The disappearance of a distinct strip at 2580 cm^{-1} which is observed within the range HCTP shows that sulfhydryl groups participate in the complex formation. Observed reduction of intensity of a strip of absorption in area of $3200-3600 \text{ cm}^{-1}$, with a maximum at 3455 cm^{-1} , emergence of a wide strip in area $3050-3150 \text{ cm}^{-1}$ shows that the hydroxyl group takes part in

in aqueous and organic solvents and did not decompose for three days, and after extraction, for more than a month. The maximum absorbance is attained within 5-8 minutes.

Stoichiometry of the Complexes and the Mechanism of Complexation. The ratio of components in the complex corresponds to W(V) : HTPDs : AP = 1 : 2 : 2; it was determined by the methods of straight line, equilibrium shift, and the relative yield [12].

Also, additional experiments by the Akhmedly's method [13] showed that the complex exists in monomeric form in the organic phase (obtained coefficient of polymerization γ was equal to 0.95-1.09).

The stability constant was determined by crossed lines method. The stability constant of W(V)-HTPDs-AP complexes was calculated and found to be $\lg \beta = 7.19-11.25$ at room temperature. The sizes of equilibrium constant K_{ex} calculated on a formula

the formation of coordination communication in the ionized state. Detection of strips of absorption at 2385 cm^{-1} indicates availability of the protonated AP1 [15].

In a highly acidic medium, there are various cationic forms of tungsten (V) in the solution with dominating WO_3^+ ion [10]. The number of protons replaced by tungsten in a HTPDs molecule appeared to be two. With due regard for the identified component ratio in the complex and an ionic state of tungsten, it was assumed that upon complexation, the following reactions proceed:

Al(III)	210		30.0	4
Ni(II)	85	EDTA	30.2	5
Co(II)	68	EDTA	29.6	4
Ti(IV)	20	Ascorbic acid	29.5	6
Mn(II)	70		29.8	5
U(VI)	60	CH ₃ COO ⁻	30.1	3
Mo(VI)	30		30.3	4
Cr(III)	50		29.7	4
V(IV)	90		30.4	6
Ta(V)	50		30.5	4
Nb(V)	50		30.6	5
Pt(II)	75	Na ₂ C ₄ H ₄ O ₆	30.4	4
Pd(II)	78		29.5	5

To conclude, the analytical parameters pertaining to the proposed method are given in Table 1.

Effect of tungsten (V) 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. Beer's law is obeyed over the concentration range 0.04 – 3.8 $\mu\text{g W(V) mL}^{-1}$ (Fig. 2). Table 3 summarizes the calibration characteristics obtained with HTPDs and AP.

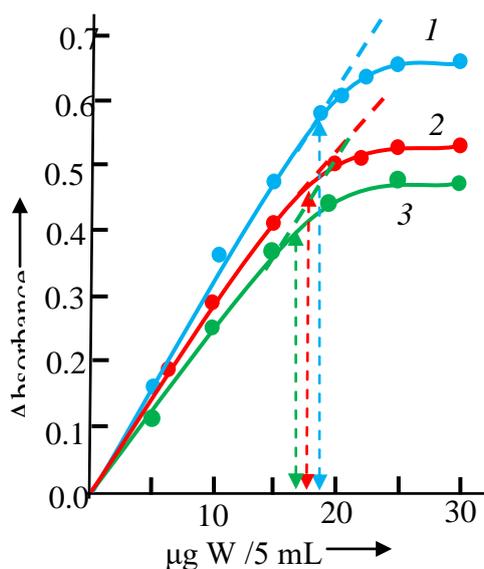


Fig.2. Beer's law range of W (V)- HTPDs-AP complex in chloroform at 470-490 nm
1–W(V)-HCTP-AP1; 2–W(V)-HBTP-AP1; 3–W(V)-HITP-AP1.

Table 3. Analytical characteristics of some ternary complexes of W with HTPDs and AP

Compound	SS* ($\mu\text{g cm}^{-2}$)	Beer's law range ($\mu\text{g} \cdot \text{mL}^{-1}$)	LOD*, ng mL^{-1}	LOQ*, ng mL^{-1}	Equation of calibration curves	Cc*
W-HCTP-AP1	0.0033	0.05-3.8	11	36	$0.032+0.25x$	0.9987
W-HCTP-AP2	0.0034	0.04-3.6	12	40	$0.027+0.24x$	0.9974
W-HCTP-AP3	0.0035	0.05-3.8	11	36	$0.045+0.22x$	0.9931

W-HBTP-AP1	0.0038	0.04-3.6	12	40	0.039+0.21x	0.9981
W-HBTP-AP2	0.0040	0.04-3.4	15	50	0.015+0.20x	0.9891
W-HBTP-AP3	0.0040	0.05-3.3	14	45	0.014+0.20x	0.9956
W-HITP-AP1	0.0044	0.04-3.4	10	33	0.031+0.18x	0.9989
W-HITP-AP2	0.0046	0.05-3.2	12	40	0.024+0.16x	0.9975
W-HITP-AP3	0.0050	0.05-3.2	9	29	0.023+0.15x	0.9991

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

The proposed method is comparable favorably with the existing ones (Tab. 4) and offers the advantages of better simplicity, rapidity, sensitivity and selectivity [1-3,6].

Table 4. Comparative characteristics of tungsten determination methods

Reagent		pH (solvent)	λ , nm	$\epsilon \times 10^{-4}$	Beer's law range ($\mu\text{g} \cdot \text{ml}^{-1}$)	Sandell's Sensitivity ($\mu\text{g cm}^{-1}$)
Toluene-3,4-dithiol[1-3]		1.5-2.0 (CHCl_3)	640	1.92	Not known	0.0060
8- Mercaptoquinoline [1,2,6]		0.5-3.0 <i>iso</i> - $\text{C}_4\text{H}_9\text{OH}$ CHCl_3 (1:1)	412	0.367	≤ 4	Not known
8 – Hydroxyquinoline [2,6]		4.4 (CHCl_3)	363	0.64	Not known	
Proposed method	HCTP-AP1	4.5-5.5 (CHCl_3)	490	2.8	0.05-3.8	0.0033
	HBTP-AP1	4.3-5.2 (CHCl_3)	480	2.4	0.04-3.6	0.0038
	HITP-AP1	4.0-5.0 (CHCl_3)	470	2.2	0.04-3.4	0.0044

Analytical Applications

Determination of tungsten in steels. 0.1 g weighed sample of steel [composition steel EU-45 (0.24% of C, 0.60% of Mn, 0.03% of Si, 3.30% of Cr, 0.50% of Ni, 0.50% of W, 0.50% of Mo, 0.30% of V, and the rest of Fe)] was dissolved under heating in 4 mL of freshly prepared mixture of HCl and HNO_3 (3: 1) in the presence of a few drops of HF. After the dissolution, 0.5 mL of HCOOH is added, and the mixture is heated until the decomposition of HNO_3 occurs. After cooling the mixture is transferred to a 50 mL volumetric flask and diluted up to the mark with water. An aliquot portion of the resulting solution is transferred to a separatory funnel; 3.0 mL of 0.01 M HCl and 0.3-0.5 mL of a 0.01 M HTPDs solution were added, and, after thorough mixing, 2.0-3.0 mL of a 0.01 M AP solution was added. The volume of the organic phase was adjusted to 5 mL with

chloroform, and the volume of the aqueous phase was brought to 20 mL with distilled water. The mixture was shaken for 5-8 min. After layering of the phases, the absorbance of extracts is measured using a KFK-2 photocolormeter at 490 nm in cuvettes of 0.5 cm thick. The tungsten concentration is found from the calibration curve. The results are shown in Table 5.

Determination of tungsten in soils. The proposed procedures for the determination of tungsten were applied to reveal it in the light-chestnut soil from the Caspian zone. A 0.5 g weight was finely ground in an agate mortar and calcined in muffle furnace for 3 h. After cooling, the sample was treated and dissolved in a graphite cup in a mixture of 16 mL of HF (conc.), 5 mL of HNO_3 (conc.), and 15 mL of HCl (conc.) at 50-60°C to remove excess

hydrogen fluoride. A further 8 mL portion of HNO₃ (conc.) was added triply to the solution that had each time been evaporated to 5-6 mL. After that, the solution was transferred into a 100 mL volumetric flask and its volume was brought to the mark with distilled water. Thus,

tungsten was identified in aliquot portions of the solution using the proposed procedures.

The accuracy of the results was checked by three independent methods. The results are shown in Table 5.

Table 5. Tungsten content in steel and soil samples determined by different methods ($n=5$, $P=95\%$)

Method		Tungsten content in steel (EU-45: 0.50% W)		Tungsten content in soil	
		\bar{X} , %	RSD,%	$\times 10^4$, %	RSD,%
Toluene-3,4-dithiol [6]		0.48	3.4	4.65	3.6
8- Mercaptoquinoline [2]		0.52	3.2	4.53	3.9
8 – Hydroxyquinoline [2]		0.51	3.6	4.92	4.5
Proposed method	HCTP-AP1	0.47	3.5	4.85	3.2
	HBTP-AP1	0.51	3.5	4.49	5.1
	HITP-AP1	0.48	3.4	4.71	4.6

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VOLFRAMIN (VI) O-HİDROKSİTİOFENOL TÖRƏMƏLƏRİ VƏ HİDROFOB AMİNLƏRLƏ KOMPLEKS ƏMƏLƏGƏLMƏSİNİN SPEKTROFOTOMETRİK TƏDQIQI

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Xülasə: *Volframın (VI) o-hidroksitiofenol törəmələri (HTF) {2-hidroksi-5-xlorotiofenol (HXTF), 2-hidroksi-5-bromotiofenol (HBTF) və 2-hidroksi-5-iyodotiofenol (HITP)} və aminofenolların iştirakı ilə kompleks əmələgəlmə reaksiyası spektrofotometrik tədqiq edilmişdir. Aminofenol kimi 2(N,N-dimetilaminometil)-4-xlorfenol (AF1) və 2(N,N-dimetilaminometil)-4-bromfenol (AF2), 2(N,N-dimetilaminometil)-4-yodfenol (AF3) istifadə edilmişdir. Müxtəlifliqandlı komplekslərin (MLK) əmələ gəlməsi və ekstraksiyası üçün optimal şərait, komplekslərin tərkibində komponentlərin molyar nisbəti müəyyən edilmişdir. MLK zəif turşu mühitdə əmələ gəlir (pH_{opt} 4.5-5.5). Işıqudma spektrində maksimum $\lambda=460-490$ nm dalğa uzunluğunda müşahidə edilir. Molyar işıqudma əmsali $\varepsilon = (2.0-2.8) \times 10^4$ -ə bərabərdir. MLK-lərin əmələ gəlməsi və ekstraksiyası üçün $(1.2-2.5) \times 10^{-3}$ mol/l HTF və $(2.0-2.8) \times 10^{-3}$ mol/l qatılıqda AF tələb olunur. Maksimum optik sıxlığa 5-8 dəqiqə ərzində çatılır. Volframın 0.04-3.8 $\mu\text{g}/5$ ml qatılıq intervalı Ber qanununa tabe olur. Təklif olunan üsul poladda və torpaqda volframu təyininə tətbiq edilmişdir.*

Açar sözlər: *volfram, o-hidroksitiofenol törəməsi və hidrofob aminlər, ekstraksiyalı-fotometrik üsul, aşkarlama həddi, təyini.*

СПЕКТРОФОТОМЕТРИЧЕСКОЕ ИССЛЕДОВАНИЕ КОМПЛЕКСОБРАЗОВАНИЯ ВОЛЬФРАМА(VI) С ПРОИЗВОДНЫМИ o-ГИДРОКСИТИОФЕНОЛА В ПРИСУТСТВИИ ГИДРОФОБНЫХ АМИНОВ

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Аннотация: *Спектрофотометрическими методами изучена реакция комплексообразования вольфрама с производными o-гидрокситиофенола (ГТФ) {2-гидрокси-5-хлортиофенол (ГХТФ), 2-гидрокси-5-бромтиофенол (ГБТФ) и 2-гидрокси-5-йодтиофенол (ГИТФ)} в присутствии аминофенолов. Из аминофенолов использованы 2(N,N-диметиламинометил)-4-хлорфенол (АФ1) и 2(N,N-диметиламинометил)-4-бромфенол (АФ2), 2(N,N-диметиламинометил)-4-йодофенол (АФ3). Найденны оптимальные условия образования и экстракции разнолигандных комплексов (РЛК) и установлены соотношения компонентов в комплексах. Установлено, что РЛК образуются в слабокислой среде (pH_{opt} 4.5-5.5). Максимум в спектре светопоглощения наблюдается при $\lambda=460-490$*

нм. Молярный коэффициент светопоглощения равен $\epsilon = (2.0-2.8) \times 10^4$. Оптимальным условием образования и экстракции этих соединений является концентрация $(1.2-2.5) \times 10^{-3}$ моль/л ГТФ и $(2.0-2.8) \times 10^{-3}$ моль/л АФ. Максимальная оптическая плотность достигается в течение 5-8 мин. Экстракты РЛК вольфрама с ГТФ и АФ подчиняются закону Бера при концентрациях 0.04–3.8 мкг/5мл. Предлагаемый метод был применен для определения вольфрама в стали и почвах.

Ключевые слова: вольфрам, производное о-гидрокситиофенола и гидрофобные амины, экстракционно-фотометрический метод, предел обнаружения, определение.