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COMPOSITION AND EXTRACTION OF TUNGSTEN(VI) COMPLEXES WITH 2-HYDROXY-5-BROMTIPHENOL AND AMINOPHENOLS

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The interaction of tungsten(VI) with 2-hydroxy-5-bromothiophenol (HBTP) in the presence of aminophenols (APs)was studied as follows:(N,N-diethylaminomethylthio)-4-methoxyphenol (AP$_1$), 2-(N,N-dibuthylaminomethylthio) -4-methoxyphenol (AP$_2$), 2,6-bis-(N,N-dimethylaminomethyl)-4-methylphenol (AP$_3$), 2,6-bis-(N,N- dimethylaminomethyl)-4-chlorophenol (AP$_4$), 2-(N,N-dimethylaminomethyl)-4-methylphenol (AP$_5$), 2-(N,N-diethylaminomethyl)-4-bromophenol (AP$_6$) and 4-chloro-2-(N,N- dimethylaminomethyl)-6-thiophenylmethylphenol (AP$_7$).The obtained ternary complexes have a composition of 1:2:2 (W:HBTP:AP) or 1:2:1 (AP$_3$ and AP$_4$). The optimum conditions for their formation and subsequent solvent extraction were found: organic solvent (chloroform), pH (4.1-5.6), concentration of reagents and extraction time. Degrees of extraction made up 98.1-98.6%. The complexes have absorption maximum at 470-482 nm and molar absorptivity coefficients ranged between (2.6-3.9)×10^4. The Beer's law is observed in the range of concentrations of tungsten 0.2-16 μg/ml.

***Keywords**: tungsten (V), solvent extraction, 2-hydroxy-5-bromothiophenol, ternary complex, determination methods*

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INTRODUCTION

Alloys containing tungsten have valuable properties, including high temperature strength, corrosion resistance, hardness and wear resistance. The tungsten is used in argon-welding electrodes, light bulbs and X-ray tubes.

Methods for determining tungsten based on color reactions with organic reagents are numerous, but only some of them are used in analytical practice [1-3]. 2-hydroxy-5-chlorothiophenol [4,5], nitro catechol [6,7], 2,3-dihydroxynaphthalene [7] as well as various ion-binding reagents [8-9] are used for the determination of tungsten. It is known that the hydroxythiophenolate complexes of tungsten are not soluble in chloroform whereas the mixed ligand complexes with hydrophobic amines and aminophenols are well dissolved in various organic solvents. In that respect, a prospective reagent comprising a -OH and -SH group in its molecule is 2-hydroxy-5-bromothiophenol (HBTP, H$_2$R). The present work deals with the interaction of tungsten with HBTP in the presence of aminophenols (AP). The following APs were used: 2-(N,N-diethylaminomethylthio)-4-methoxyphenol (AP$_1$), 2-(N,N-dibuthylaminomethylthio) -4-methoxyphenol (AP$_2$), 2,6-bis-(N,N-dimethylaminomethyl)-4-methylphenol (AP$_3$), 2,6-bis-(N,N- dimethylaminomethyl)-4-chlorophenol (AP$_4$), 2-(N,N-dimethylaminomethyl)-4-methylphenol (AP$_5$), 2-(N,N-diethylaminomethyl)-4-bromophenol (AP$_6$) and 4-chloro-2-(N,N-dimethylaminomethyl)-6-thiophenylmethylphenol (AP$_7$). On the basis of the results obtained, new selective and sensitive procedures for the extraction and photometric determination of tungsten microscopes in steel were developed.

MATERIALS AND METHODS

Reagents. The W(VI) stock solution was prepared by dissolving $Na_2WO_4 \times 2H_2O$ in water. Its exact concentration is established gravimetrically - precipitation of tungsten in the form of acid and weighing of the obtained WO_3 [10]. Working solutions at a concentration of 0.1 mg / mL are obtained by diluting the stock solution. Chloroform solutions of HBTF and AF were used at a concentration of 0.01 mol/l. HBTP and AP were synthesized according to known methods: [11] and [12], respectively. Their purity was checked by paper chromatography and melting temperature determination. Chloroform is distilled before use.

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

Procedure. In the graduated tubes equipped with ground stoppers, there are a W (VI) solution (0.1 to 0.8 mL, 0.1 mL interval), a solution of HBTP (2.4 ml) and AP solution (2.2-2.3 mL). The necessary acidity is created by adding solutions of NaOH or HCl at a concentration of 0.1 mol/l. The volume of the organic phase is brought to 5 ml with chloroform and the aqueous phase to 25 mL (with water). Suffice it to shake for extraction (15 min) and after complete phase separation, the organic layer is separated and its absorption measured in 0.5 cm (490 nm) (KFK-2) wavelength cuvettes.

Procedure for dissolving steels. A weighted amount of steel (depending on the concentration of tungsten in it) is dissolved in HCl (1:1). A few drops of conc. HNO_3. 60 ml of hot water and 5 ml of HCl (1:1) were added. It is refluxed for 5 minutes. The resulting yellowish precipitate of tungstic acids was removed by filtration through a blue band filter. The residue was thoroughly washed with 100 ml. water and dissolved in a solution of 0.5 mol/l NaOH. After cooling, the solution is transferred to a 50 ml volumetric flask. Water is added to the brand.

Experiments related to the reduction of tungsten. It is known that W (V) forms a dithiol colored compound, and W (VI) is reduced to W (V) by the reagent itself [3]. To fortify the conclusion, we conducted two series of experiments: 1) interaction between HBTP and W (V) obtained by reduction with $SnCl_2$ and KI in the hydrochloric acid medium; 2) interaction between HBTP and W (V) in the absence of additional reducers. The products of both reactions were characterized by a maximum at $\lambda = 470$ nm.

RESULTS AND DISCUSSION

In a weak acid and neutral medium (pH 2.0-6.8) the W (V) forms a water-soluble orange compound with HBTP. As the reagent concentration and the pH rises, the intensity of the coloration increases as well, and with the addition of various AF and organic solvents under certain conditions, well-extractable triple complexes are obtained.

Choice of solvent. We experimented with various organic solvents - chloroform, carbon tetrachloride, benzene, toluene, xylene, isobutanol, isopentanol, dichloroethane and chlorobenzene. The extractability of the complexes was assessed by the distribution coefficient and the extraction rate. The chloroform, dichloroethane and chlorobenzene solvents were most suitable. All further studies were performed with chloroform.

The content of tungsten in the organic phase was determined photometrically by toluene-3,4-dithiol after reextraction, and the tungsten content in the aqueous phase by difference [5].

Table 1. Characteristics on the ternary complex with different AP

AP	Composition (W: HBTP:AP)	pH_{opt}	λ_{max}, nm	$\Delta\lambda$, nm	$\varepsilon 10^{-4}$,	lg β	lg $K_{ек}$	Adherence to Beer's law, µg/mL
AP_1	1:2:2	4.2-5.2	470	200	3.7	6.3	9.14	0.2-13.0
AP_2	1:2:2	4.7-5.4	480	210	3.9	6.9	10.1	0.2-16.0
AP_3	1:2:1	4.3-5.3	482	212	2.8	5.3	8.20	0.2-13.0
AP_4	1:2:1	4.5-5.5	475	205	2.7	5.6	7.35	0.2-13.0
AP_5	1:2:2	4.5-5.4	473	203	2.6	5.5	7.12	0.2-15.0
AP_6	1:2:2	4.1-5.4	479	209	3.0	6.2	8.92	0.2-14.0
AP_7	1:2:2	4.2-5.6	477	207	3.3	6.5	9.01	0.2-16.0

We have found that the essence of the AF practically does not affect the extent of the tungsten extraction. As for single extraction in chloroform, it is in the range of 98.1-98.6%.

Effect of pH on the aqueous phase. Tungsten interacts with HBTP at pH> 1.5. For the formation and extraction of the various triple complexes, the optimal pH values are taken in the range of 4.1-5.6 (Table 1). At pH above, the 7-8 extraction is not practically carried out due to the decrease in the protonation rate of aminophenols.

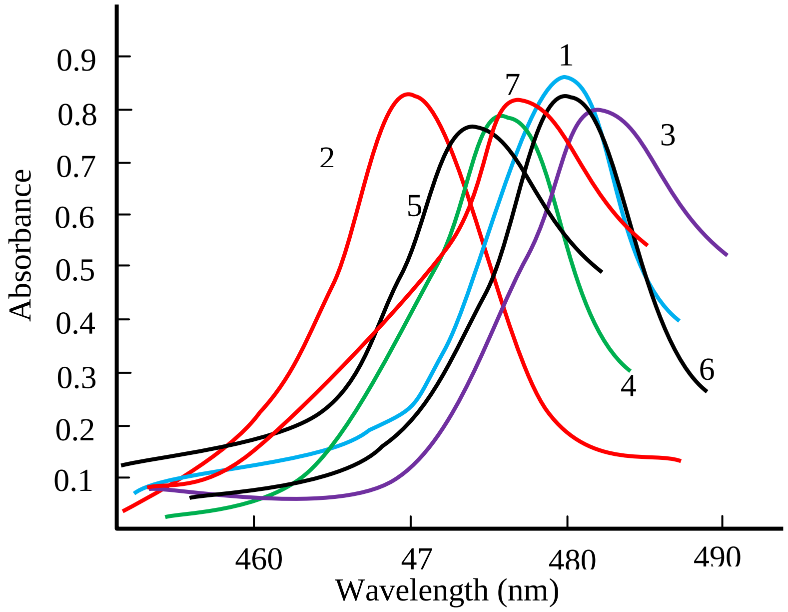

Fig. 1. The absorption spectra of the spectra of W(V)-HBTP-AP under optimal extraction conditions are as follows:
1-W(V)-HBTP-AP_1; 2-W(V)-HBTP-AP_2; 3-W(V)-HBTP-AP_3; 4-W(V)-HBTP-AP_4; 5-W(V)-HBTP-AP_5; 6- W(V)-HBTP-AP_6; 7-W(V)-HBTP-AP_7
$C_{W(VI)}$= 1.09×10^{-5} M; C_{HBTP}=(9.2-9.6)×10^{-4} M; C_{AP}= (8.8-9.3)×10^{-4} M; SF-26; λ=490 nм; ℓ=1.0 cm.

Electronic Spectra. Chloroform extracts of the various complexes have absorption maximum in the range of 470-482 nm. The molar absorbances ε are in the range of (2.7 - 3.9)× 104. They are higher than those reported in [4, 5, 8, 9, 13,16]. Table 1 shows that the complexes with AP1 and AP2 involved have the best characteristics. Their spectra are presented in Fig. 1.

Effect of reagent concentration. For the formation and extraction of triple complexes, the concentration of HBTP in the aqueous phase should be in the range (9.2-9.6)×10^{-4} mol/l, and that of AP - (8.8-9.3)×10^{-4} mol/l. When using chloroform solutions of HBTP, AP$_1$ and AP$_2$ (at a concentration of 0.01 mol/l), the optimal volumes are 2.4, 2.2 and 2.3 ml respectively. It is not recommended to use larger amounts of aminophenols.

The law of Bern is observed in a wide range of concentrations (Table 1) which allows the use of ternary complexes for the determination of tungsten. The detection limits of the AP$_1$ and AP$_2$ complexes are 0.06 g/ml and 0.05 g/ml respectively.

Resistance to dyeing and extraction time. Unlike the double complex of W (V) with HBTP, the ternary complexes are resistant to both aqueous and organic solvents. We found that after extraction in chloroform, the staining was stabilized for about a month. To achieve an extraction equilibrium, shake it for approximately for 15 minutes.

Molar relations, elemental analysis and complexation equations. The molar interaction was established by the Asmus method and the equilibrium displacement method [14] (Fig. 2). The results obtained make it possible to conclude that when using AP$_1$, AP$_2$, AP$_5$, AP$_6$ and AP$_7$ the molar ratios are 1:2:2 (W: HBTP: AP). When using the bis-aminophenols AP$_3$ and AP$_4$, which may form cations of the H_2AP^{2+} type, the molar ratios are 1:2:1 (W: HBTP:AP).

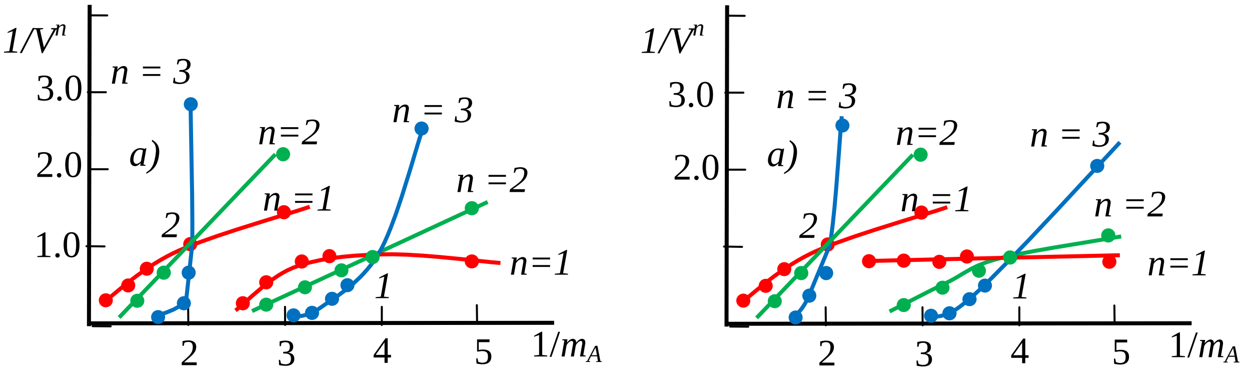

Fig. 2. Determination of the complexes composition by the Asmus method. 1 -W; 2 - AP. a) W(V)-R-AP$_1$; for R and for AP$_1$ tgα ≈2; b) W(V)-R-AP$_3$; for R tgα ≈2; for AP$_3$ tgα ≈1;. $C_{W(VI)}$= 1.09×10^{-5} M; SF-26; λ=490 нм; ℓ=1.0 см.

When adjusted for the molar ratios obtained and the fact that W(VI) is reduced to W (V) by HBTP (H_2R) (and W(V) at pH 3-7 exist mainly in the form of [WO(OH) and $[WO(OH)]^{2+}$ [1]), we assume that the complex formation proceeds according to the following equations:

$$[WO(OH)]^{2+} + 2H_2R \rightleftarrows [WO(OH)R_2]^{2-} + 4H^+ \qquad (1)$$
$$[WO(OH)R_2]^{2-} + 2HAP_1^+ \rightleftarrows (HAP_1^+)_2[WO(OH)R_2] \qquad (2')$$
$$[WO(OH)R_2]^{2-} + H_2AP_3^{2+} \rightleftarrows (H_2AP_3^{2+})[WO(OH)R_2] \qquad (2'')$$

Equation 2’ represents the association of $[WO(OH)R_2]^{2-}$ with the HAP + (AP$_1$, AP$_2$,

To confirm the relationship between the components in the triple complexes with AP$_1$ and AP$_2$, they were isolated in solid state and assayed by elemental analysis:

AP$_5$, AP$_6$ and AP$_7$) cations, and (AP$_3$ and AP$_4$) equation 2”.

$(HAP_1)_2[WO(OH)R_2]$ - {Calculated,%: 19.19 W, 16.68 Br, 6.67 S, 2.92 N; Found,%: 19.43 W, 16.37 Br, 6.63 S, 2.76 N} and $(HAP_2)_2[WO(OH)R_2]$ – {Calculated,%: 14.72

W, 12.80 Br, 5.12 S, 2.24 N; Found,%: 14.62 W, 12.74 Br, 5.09 S, 2.11 N}.

Equilibrium and extraction constants. On the basis of equation (2’), the following equilibrium constants can be written:

$$\beta = \frac{\{(HAP_1)_2[WO(OH)R_2]\}_o}{\{[WO(OH)R_2]^{2-}\}_{aq}\{(HAP_1)^2\}_{aq}}$$

The distribution coefficient (D) is equal to

$$D = \frac{\{(HAP_1)_2[WO(OH)R_2]\}_o}{\{[WO(OH)R_2]^{2-}\}_{aq}}$$

therefore

$$\beta = \frac{D}{[HAF_1{}^+]^2}$$

When logging, we get

$$\lg \beta = \lg D - 2\lg\,[HAP_1{}^+] \qquad (3’)$$

and in the case of AP_3 and AP_4 (equation 2)

$$\lg \beta = \lg D - \lg\,[H_2AP_3{}^{2+}] \qquad (3”)$$

We calculated the extraction constants according to the equations

$$\lg K_{ек} = \lg D - 2\lg\,[HBTP^{2-}] - 2\lg[HAP_1{}^+] \qquad (4’)$$

$$\lg K_{ек} = \lg D - 2\lg\,[HBTP^{2-}] - \lg[H_2AP_3{}^{2+}] \qquad (4”)$$

The results of the calculations are presented in Table 1.

To determine the degree of aggregation of the complexes in the organic phase, we calculated the degree of polymerization by equation set out in [15]. Calculations show that all complexes are in monomeric form (γ = 0.9-1.2).

Table 2. Comparative characterization of tungsten determination procedures

Reagent(s)	pH	Solvent	λ, nm	$\varepsilon \cdot 10^{-4}$,	Linear interval, μg/ml W	[Ref.]
Toluene-3,4-dithiol	1.5-2.0	chloroform	640	1.92	-	[2]
8-Mercaptoquinoline	0.5-3.0	isobutanol	412	0.367	≤4	[1]
8-Hydroxyquinoline	4.4	chloroform	363	0.64	-	[1]
4-nitrocatehol	1.2-3.6	chloroform	415	2.8	0.9-8.8	[13]
3-hydroxy-2- (2'-thienyl) -4-oxo-4H- 1 -benzopyran	0.2 M HCl	dichloromethane	415	6.45	0-2.8	[16]
HBTP + AP_1	2.0-5.8	chloroform	470	3.7	0.2-13.0	This work
HBTP + AP_2	2.2-5.9	chloroform	480	3.9	0.2-16.0	

Table 3. Results of tungsten determination in steel 339 (W - 0.183%, *n* = 5, *P* = 95%)

Method		$\bar{X}$, %	RSD, %
Our Methods*	HBTP + AP_1	0.180±0.011	5
	HBTP + AP_2	0.182±0.007	3
Toluene-3,4-dithiol [2]		0.185±0.009	4

* - *In the presence of 2 mL thioglycolic acid at a concentration of 0,01 mol/L*

Effect of foreign ions and masking reagents. To assess the applicability of ternary complexes for the determination of tungsten in real objects, the influence of side ions was studied. It should be noted that a large amount of alkaline, alkaline- and rare-earth ions F^-, Cl^-, Br^-, SO_3^{2-}, SO_4^{2-} and $C_2O_4^{2-}$ does not affect the definition. The inhibitory effect of Fe (III) was removed by means of thioglycolic acid (0.01 mol/l, 2 ml); Ti(IV) - ascorbic acid Cu(II) by means of thiourea Mo(VI) with oxalate. When using EDTA, the definition does not interfere with Ti (IV), V (IV), Nb (V), Ta (V), Mo (VI) and Fe (III).

Comparative characteristics of the procedures for determining W. Table 2 includes data that compare the analytical characteristics of the procedures developed by us with those of the already known procedures [1, 2, 13, 16].

Table 3 shows the results of the real object analysis - steel 339 (W content - 0.183%). It can be concluded that the proposed procedures based on the use of HBTP and aminophenols AP_1 AP_2 are not inferior to those described in the literature. They allow reasonable, selective, accurate, cheap and reliable determination of tungsten in real objects.

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VOLFRAMIN (VI) 2-HİDROKSİ-5-BROMTİOFENOL VƏ AMİNOFENOLLARLA KOMPLEKSLƏRİNİN TƏRKİBİ VƏ EKSTRAKSİYASI

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Volframın (VI) 2-hidroksi-5-bromtiofenol(HBTF) və aminofenollarla - 2-(N, N-dietilaminometiltio) -4-metoksifenol (AF$_1$), 2- (N,N-dibutilaminometil)-4-metoksifenol (AF$_2$), 2,6-bis- (N,N-dimetilaminometil)-4-metilfenol (AF$_3$), 2,6-bis- (N,N-dimetilaminometil)-4-xlorofenol (AF$_4$), 2-(N,N-dimetilaminometil) -4-metilfenol (AF$_5$), 2- (N,N-dietilaminometil) -4-bromofenol (AF$_6$) və 4-xlor -2- (N,N-dimetilaminometil) -6-tiofenilmetilfenol (AF$_7$) qarşılıqlı təsiri tədqiq edilmişdir. Alınan müxtəlifliqandlı kompleksləri 1:2:2 (W:HBTF:AF) və ya 1:2:1 (AF$_3$ və AF$_4$) tərkibinə malikdir. Onların əmələgəlməsi və ekstraksiyası üçün optimal şərait - üzvi həlledici - xloroform, pH$_{op}$ (4.1-5.6), reagentlərin qatılığı və ekstraksiya müddəti müəyyən edilmişdir. Ekstraksiya dərəcəsi 98.1-98.6% intervalında dəyişir. Komplekslər 470-482 nm-də maksimum işıqudmaya malikdir. Molyar udma əmsalı (2.6-3.9)$\times 10^4$ təşkil edir. Ber qanununa tabe olma 0.2-16 μg/ml W qatılığında müşahidə olunur.

Açar sözlər: *volfram (V), həlledici ilə ekstraksiya, 2-hidroksi-5-bromotiofenol, üçlü kompleks, təyin metodikası*

СОСТАВ И ЭКСТРАКЦИЯ КОМПЛЕКСОВ ВОЛЬФРАМА (VI) С 2-ГИДРОКСИ-5-БРОМТИФЕНОЛОМ И АМИНОФЕНОЛАМИ

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Изучено взаимодействие вольфрама (VI) с 2-гидрокси-5-бромтиофенолом (ГБТФ) в присутствии аминофенолов (АФ): 2- (N, N-диэтиламинометилтио) -4-метоксифенола (АФ$_1$), 2-(N,N-дибутиламинометил)-4-метоксифенола (АФ$_2$), 2,6-бис-(N,N-диметиламинометил) 4-метилфенола (АФ$_3$), 2,6-бис- (N, N-диметиламинометил) -4-хлорфенола (АФ$_4$), 2-(N,N-диметиламинометил)-4-метилфенола (АФ$_5$), 2-(N,N-диэтиламинометил)-4-бромфенола (АФ$_6$) и 4-хлор-2-(N,N-диметиламинометил)-6-тиофенилметилфенола (АФ$_7$). Полученные тройные комплексы имеют состав 1:2:2 (W:ГБТФ:АФ) или 1:2:1 (АФ$_3$ и АФ$_4$). Найдены оптимальные условия их образования и последующей экстракции: органический растворитель - хлороформ, pH$_{оп}$ (4.1-5.6), концентрация реагентов и время экстракции. Степень экстракции составляет 98.1-98.6%. Комплексы имеют максимумы поглощения при 470-482 нм. Молярные коэффициенты поглощения составляют $(2.6\text{-}3.9)\times10^4$. Закон Бера наблюдается в диапазонах концентрации вольфрама 0.2 0.2-16.0 мкг/мл.

Ключевые слова: *вольфрам (VI), экстракция растворителем, 2-гидрокси-5-бромтиофенол, тройной комплекс, методика определения*