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SPECTROPHOTOMETRIC RESEARCH INTO MULTI-LIGAND COMPLEXES FORMED BY ZIRCONIUM (IV) WITH STILBAZOLE AND CETYLPYRIDINIUM CHLORIDE

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Abstract: This study deals spectrophotometric analysis of the multi-ligand complex of zirconium (IV) with stilbazole and cetylpyridinium chloride. Dichloroethane was selected as the extragent and acetonitrile as the dispersant solution. Optimal conditions for the complex formation were identified. The results of the extraction process and complex formation were calculated statistically with Plackett Burman design and central composite design (via the Minitab 19 program). The values obtained showed that experimental results can be expressed as statistical results. Most important factors influencing absorption during complex formation were pH and ligand content. To obtain the maximum absorption, pH should be 4.5 and the ligand content should be 300 μL . The stoichiometric composition of the components in the complex was determined by various methods. The interval of subordination to Beer's law was 2-7.6 $\mu\text{g mL}^{-1}$, the molar light absorption coefficient was $2.6 \times 10^4 \text{ L Mol}^{-1}\text{cm}^{-1}$, $\lambda_{\text{max}} = 590\text{nm}$. A highly selective methodology was developed to determine the zirconium (IV) micronutrients in water samples.

Keywords: zirconium (IV), stilbazole, cetylpyridinium chloride, central composite design.

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Introduction

Zirconium is a metal widely used in various industries and medicine. Generally, zirconium and its chemical compounds are used in iron and steel, ceramics, paints, paper, textiles, fertilizers, nuclear power plants, machinery, aircraft, makeup, chemical and leather industries. It is used as an essential material in dentistry due to its biocompatibility with the human body and non-allergic reactions. Thus, zirconium caps are used as the cheekteeth are subject to strong mechanical impact during chewing. Zirconium caps are more like natural teeth in terms of color and refractive index.

Determination of zirconium content in environmental and industrial samples is significant due to the high toxicity of its compounds. There are many analytical methods in the literature to determine the zirconium amount. Examples of various spectro-analytical methods include the molecular fluorescence spectrophotometry [1], atomic absorption spectroscopy [2], laser ablation inductively

coupled plasma optical emission spectrometry [3], spectrophotometric method [4,5], solid phase extraction and spectrophotometric determination [6,7], plasma atomic emission spectrophotometric determination [8], inductively coupled plasma atomic emission spectroscopy [9], UV-Vis spectrophotometry [10,11] and flotation-spectrophotometric method [12]. Moreover, work was done to separate zirconium from mixtures with hafnium and for analytical determination [13-17].

The stilbazole with zirconium ions forms a blue-purple homogeneous ligand complex [18]. The homogeneous ligand complexes formed by the stilbazole with zirconium and mixed-ligand complexes with the participation of EDTA were studied [19].

To determine the zirconium, a variety of ligand complexes formed with stilbazole and cetylpyridinium chloride were used in the research. Stilbazole is one of the azo compounds of pyrocatechol. The sulphogroup in

the stilbazole affects the density of the π -electron system in the aromatic ring of pyrocatechol, and the hydrogen atom in the O-H group of pyrocatechols is motile and replaced by hydrogen cetylpyridinium chloride.

By interacting with surfactants, it is possible to change the physicochemical properties of organic reagents in aqueous

solutions [20-24]. As a result of the application of cetylpyridiniumchloride, it is possible to obscure many elements that interfere with the determination and to obtain a complex combination of low-density zirconium ions at high concentrations. This significantly increases the sensitivity and selectivity of the determination.

Experimental part

Apparatus, reagents and solutions. The optical density of color solutions was measured on a UV-VIS Evolution 60S spectrophotometer and spectrophotometer SF-46. The pH of all solutions was measured using the Cond./TDS/Temp universal pH meter.

The stilbazole (4,4'-bis-(pyrocatechol)-stilbene-2-2'-disulfuric acid) was dissolved in distilled water and 5×10^{-3} mol L⁻¹ solution was prepared, the structural formula of the stilbazole is given in Fig.1. A standard zirconium solution

with a concentration of 135 $\mu\text{g mL}^{-1}$ was prepared by dissolving "high purity" metallic zirconium in a HF + HClO₄ mixture. The hydrogen fluoride is then evaporated and separated, mixed with 4 M HClO₄ and 5 M H₂SO₄. 20 $\mu\text{g mL}^{-1}$ standard solution was obtained by diluting the feed solution of zirconium with distilled water. 0.1% cetylpyridinium chloride solution was used as a surfactant.

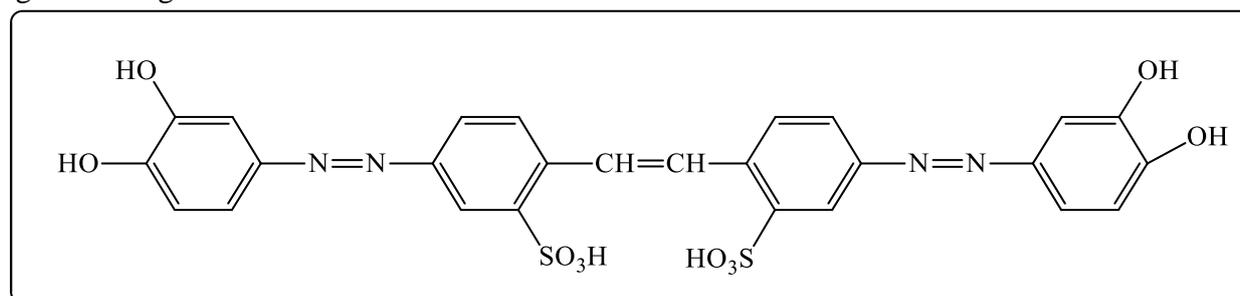


Fig. 1. Stilbazole (4,4'-bis-(pyrocatechol)-stilbene-2-2'-disulfuric acid) structure.

Method. 300 μL SB and 150 μL SP are added to a 20 μg zirconium sample and mixed, and then 300 μL of the dispersant solution is added and mixed. Also, 400 μL extragent solution is added to the mixture. The acidity of the system is then brought to pH = 4.5. Total volume is diluted with distilled water up to 10 mL and centrifuged at 3500 rpm for 3 minutes. The organic phase collects at the bottom of the vessel and the water phase at the top is separated with a pipette. The light absorption of the organic phase is measured.

The stilbazole donor heteroatoms are prone to protonation; therefore the multi-ligand complexes formed with cetylpyridinium chloride are formed in an acidic environment. The reaction of stilbazole with zirconium ions also occurs in acidic environments. This study

revealed that when the pH is lowered, stilbazole forms a precipitate and the complex it forms with zirconium breaks down, forming basic salts and metal hydroxides. When the pH rises above a certain value, the colors of the ionic forms of the stilbazole present in the solution overlap (at different values of alkalinity, the stilbazole turns yellow, red, raspberry, purple or blue), and this results in the reduction of sensitivity and the number of repeated analyses.

The optical density was measured in the range of pH =1-7 to study the optimal conditions for complex formation and the effect of pH on extraction (Fig. 2). As graph shows, the value of optical density decreases when pH<4 and pH> 5. The maximum value of light absorption occurs when pH = 4.5. Therefore, pH = 4.5 was optimally selected for the Zr (IV) ion.

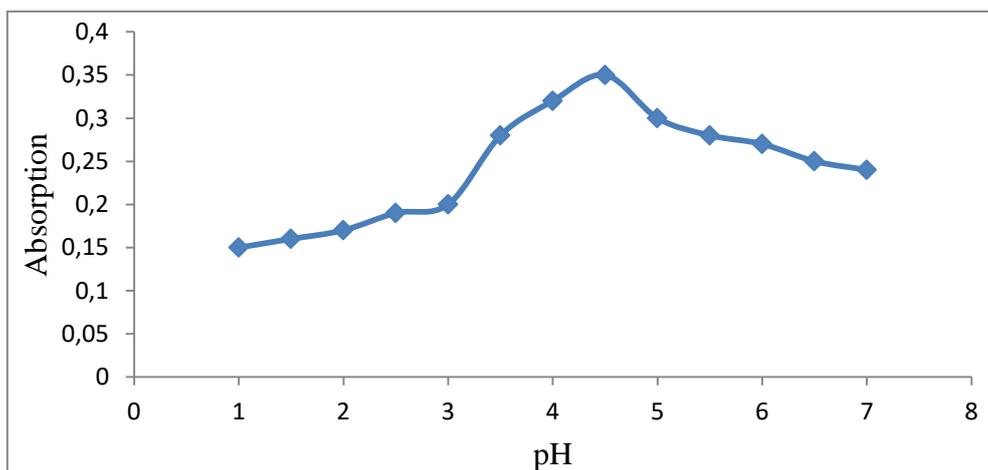


Fig. 2. pH effect on light absorption

The stilbazole content effect on the light absorption. A series of solutions in the range of 100-500 μL was prepared from the $5 \times 10^{-3} \text{ mol L}^{-1}$ solution of the stilbazole. Absorption was measured at different volumes of the stilbazole while maintaining other experimental conditions. Stilbazole interacts with cetylpyridinium chloride after

deprotonation. Deprotonation weakens as acidity increases, and after a certain value of acidity, deprotonation does not occur. As the amount of stilbazole increases, the optical density of the color solution of the complex increases. After a certain limit, the value of optical density does not change (Fig. 3).

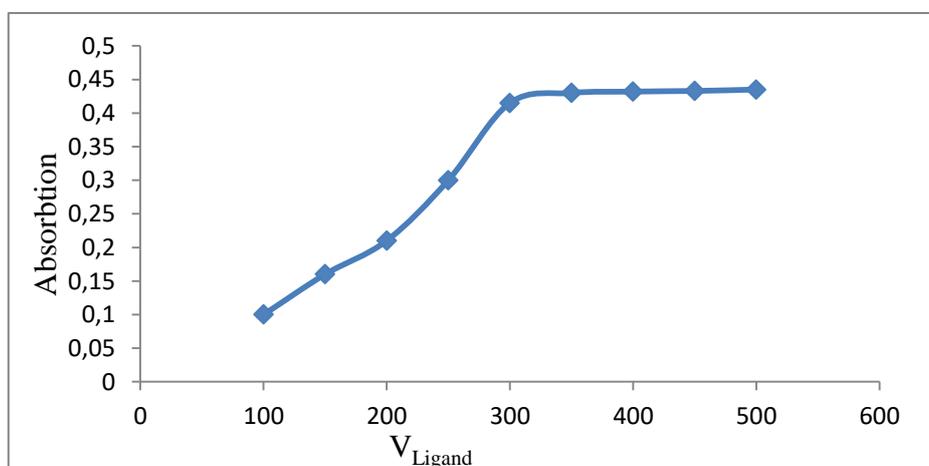
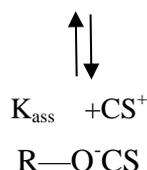


Fig. 3. Effect of stilbazole content on light absorption.

Effect of cetylpyridinium chloride on light absorption. A series of solutions in 50-300 μL volume range was prepared from a 0.1% solution of cetylpyridinium chloride, and the optical density of the color complex occurred by maintaining other experimental conditions was measured. The best results were obtained when the volume of cetylpyridinium chloride was 150

μL . Cetylpyridinium chloride facilitates the dissolution of the reagent and the occurred complex. A new region is observed in the light absorption spectrum of the reagent showing that cetylpyridinium chloride forms an association with the reagent. The association mechanism of the reagent with the cationic surfactant (CS) can be shown as follows:





When the amount of cetylpyridinium chloride is less, the above processes are relatively weak, and as the concentration of cetylpyridinium chloride increases, the formation of associations increases. However, as the concentration of cetylpyridinium chloride increases, its solubility weakens, and flotation occurs. Thus, when the amount of cetylpyridinium chloride in the system is at the optimum thickness and the acidity of the environment is optimal, the reaction in the system becomes quite sensitive and selective. A coloured complex is formed immediately after the addition of metal ions, reagent and cetylpyridinium chloride to the

reaction system in the appropriate sequence. The colour complex remains unbroken during the day and the optical density does not change.

Effect of extragent and dispersing solutions. To study the effect of the volume of the extragent solution on the absorption, a series of solutions prepared at 200-650 μL of dichloroethane were used, and 100-500 μL of acetonitrile made it possible to study the effect of the volume of the dispersant solution on the absorption. Maximum light absorption was observed at 400 μL of dichloroethane (Fig. 4a) and 300 μL of acetonitrile (Fig. 4b).

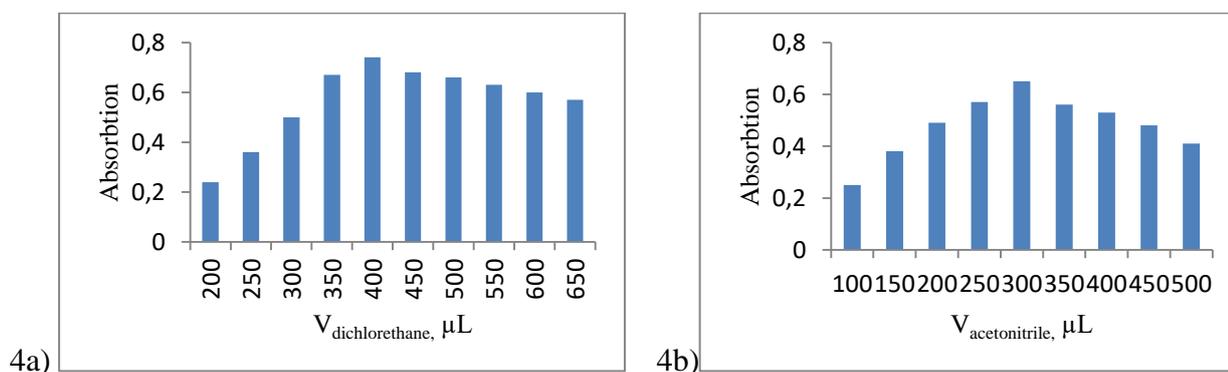


Fig. 4. Effect of extragent solution on light absorption (4a) and dispersant solution on light absorption (4b).

Influence of other parameters on absorption.

The effect of centrifugation time and speed on absorption was studied. By maintaining other experimental conditions, optical density of the complex solution was measured at intervals of 2-6 minutes at 2000-4500 rpm. Maximum absorption was observed when the coloured solution was centrifuged at 3500 rpm in 3 minutes. Besides, the extraction recovery is high in the range of 10-40 mL of the volume of the water phase, in the temperature range of 18-36 °C, and the colour intensity of the complex remains constant.

Effect of foreign ions. Along with increasing the selectivity of the reaction by changing the amount of acidity, it is also possible to shield foreign ions. Oxalate, citric, tartaric acid, EDTA, H₂O₂ break down zirconium complexes.

If the reagents and then glycerine are added to the zirconium solution, the colour intensity of the zirconium complex is reduced by half. Sulphate ions do not decolorize zirconium complexes. This allows the determination of zirconium in sulfuric acid. When heated, the zirconium complex decolorizes in the presence of slightly excess sulfuric acid. A comparison of several factors shows that increasing the selectivity of the zirconium reaction by increasing the acidity of the solution has a better effect than increasing the selectivity using coagulants.

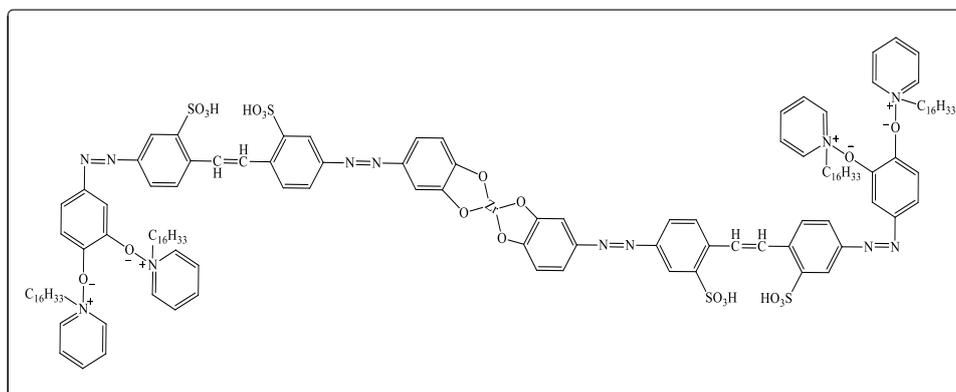
The acid-base and spectral properties of the multi-ligand complexes formed by stilbazole in the presence of surfactants are shown in Table 1.

Table 1. Acid-base and spectral properties of zirconium-SB-SP complex.

Parameters	amounts
λ_{\max} , (nm)	590
Molar light absorption ratio ($\text{L Mol}^{-1}\text{cm}^{-1}$)	2.6×10^4
Limit of detection (μgmL^{-1})	0.1
Limit of quantification (μgmL^{-1})	0.34
Linear Range (μgmL^{-1})	2-7.6
pH	4.5
$\lg\beta_k$	9.96
Zr: SB: SP	1:2:4

Probable structure of the multi-ligand complexes formed by Zr (IV) with stilbazole and cetylpyridinium chloride (Fig. 5). The stoichiometric ratio of the components in the complex was determined by equilibrium

displacement, isomolar series method and straight-line methods of Asmus [25]. The molar ratio of the components in the complex is Zr: SB: SP = 1: 2: 4.

**Fig. 5.** Probable structure of a multi-ligand complex formed by Zr (IV) stilbazole and cetylpyridinium chloride.

Analytical performance

Samples of zirconium of known concentration were added to 1000 mL water and analyzed by the proposed method. Limit of detection (LOD) and limit of quantification (LOQ) of zirconium were determined in each water sample. The best analytical results were

obtained in the experiment with tap water. Determination coefficient and sensitivity were studied by adding different amounts of zirconium samples to distilled water. Sensitivity was studied inter-day and intra-day. Results obtained are given in Table 2.

Table 2. Analytical features of the proposed method

Zirconium ($\mu\text{g L}^{-1}$)	Accuracy (%)			
	Inter-day		Intra-day	
	Consistency, ($\mu\text{g L}^{-1}$)	Relative error, %	Consistency, ($\mu\text{g L}^{-1}$)	Relative error, %
2.0	2.02	1	1.99	0.5
3.0	2.98	0.7	3.01	0.33
4.0	3.97	0.75	4.02	0.5

5.0	4.99	0.2	4.98	0.4
6.0	6.02	0.33	5.97	0.5
7.0	7.04	0.6	7.02	0.3

Optimization of statistical calculations and results. Statistical calculations were performed in two stages. First, the factors affecting absorption were analyzed to be meaningful/ meaningless by applying the Plackett Burman design. The low and high

values of these six factors, including the ligand content, the volume of the surfactant, the volume of the extragent and dispersing solutions, the pH and the rate of centrifugation are given in Table 3.

Table 3. Values of independent variables included in the statistical calculation.

Independent variables	Symbol	Lowest value	Highest value
pH	pH	1	7
Volume of extragent solution, (μL)	VES	200	650
Volume of ligand, (μL)	VL	100	500
Volume of dispersing solution, (μL)	VDS	100	500
Volume of surfactant solution	VSS	50	300
Centrifuge speed, rpm	SC	2000	4500

When studying the factors impact on the outcome with Plackett Burman's design, no interactions between factors are considered [26]. When performing statistical calculations, first low and high levels are determined for each factor, a design table is prepared for the coded

levels, and then experiments are performed by writing the real values of the factors instead of the coded values [27]. In all experiments, the amount of Zr (IV) was kept invariable at 20 μg . The results are shown in a pareto graph (Fig. 6).

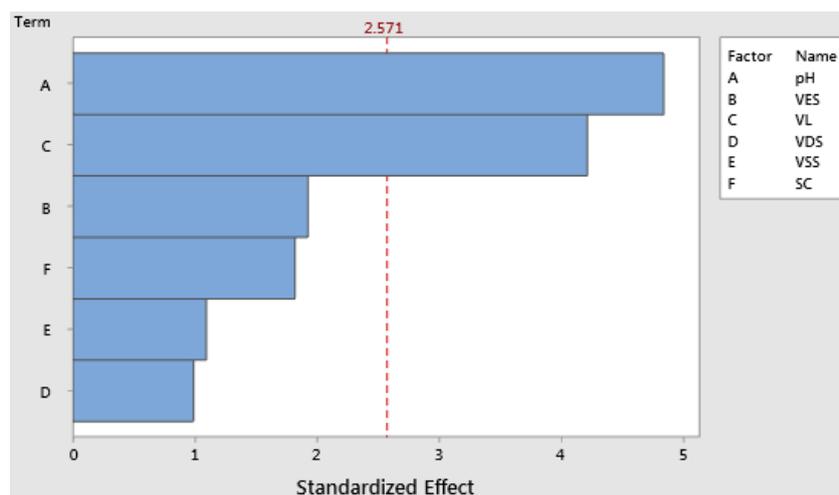


Fig. 6. Pareto chart of factors impacting absorption.

As shown in the chart, the factors that most affecting the extraction recovery are the pH and ligand volume. The effect of other experimental factors is statistically insignificant.

Optimal values of pH and ligand volume are not found with this statistical calculation.

In the second stage, a central composite design was applied to find out the optimal

values of the factors whose effect on extraction recovery and Zr (IV) absorption was statistically significant. Five levels of each factor were chosen for statistical calculation: low level (-1), medium level (0), high level (+), and two-star points $\pm \alpha$ ($\alpha = 1.414$). As shown in Fig. 7, the value of absorption increases as the pH and ligand content increase. However, after a certain

pH value, the absorption decreases. When the ligand volume is increased by a certain amount, the absorption increases and then comes down. Statistically, the optimal values for pH and ligand volume were pH = 4.5 and $V_{\text{ligand}} = 300$ μL . These values coincide with the experimentally calculated values for pH and ligand volume.

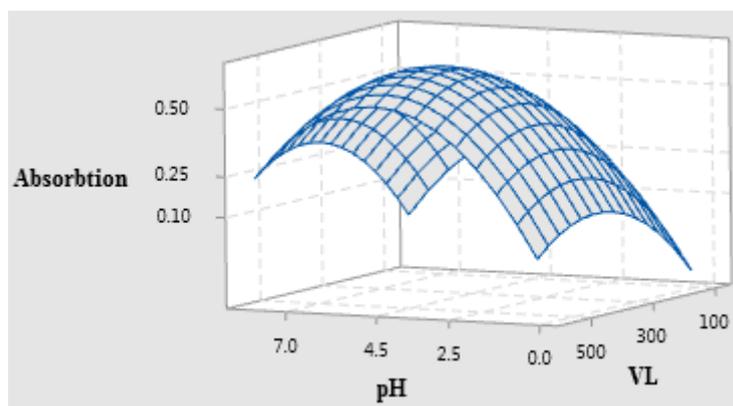


Fig. 7. Impact of pH and volume of the ligand content to the absorption in the extraction process

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SİRKONIUMUN (IV) STİLBAZA VƏ SETİLPİRİDİNXLORİD İLƏ ƏMƏLƏ GƏTİRDİYİ MÜXTƏLİFLİQANDLI KOMPLEKSİN SPEKTROFOTOMETRİK TƏDQIQI

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Xülasə: Bu işdə sirkoniumun (IV) stilbaza və setilpiridin xlorid ilə müxtəlifliqandlı kompleksi spektrofotometrik üsulla tədqiq edilmişdir. Ekstragent kimi dixloretan, dispersləşdirici məhlul kimi isə asetonitril seçilmişdir. Kompleksmələgəlmənin optimal şəraiti müəyyən edilmişdir. Ekstraksiya prosesinin və kompleksmələgəlmənin nəticələri Plackett Burman dizaynı və mərkəzi kompozit dizaynı (Minitab 19 proqramı vasitəsilə) ilə statistik olaraq hesablanıb, alınan qiymətlər təcrübi nəticələrin statistik nəticələr kimi ifadə edilə biləcəyini göstərilib. Kompleksmələgəlmə zamanı absorpsiyaya ən çox təsir edən faktorların pH və liqandın miqdarı olduğu müəyyən edilmişdir. Maksimum absorpsiya əldə etmək üçün $\text{pH}=4.5$, liqandın həcmi isə $300 \mu\text{L}$ olmalıdır. Kompleksdə komponentlərin stexiometrik tərkibi müxtəlif metodlarla təyin edilmişdir. Ber qanununa tabeçilik intervalı $2-7.6 \mu\text{g mL}^{-1}$, molyar işıqudma əmsali $2.6 \times 10^4 \text{ L Mol}^{-1}\text{cm}^{-1}$, $\lambda_{\text{max}}=590 \text{ nm}$ olmuşdur. Su nümunələrində sirkoniumun (IV) mikromiqdarının təyini üçün yüksək seçiciliyə malik metodika işlənmişdir.

Açar sözlər: sirkonium (IV), stilbaza, setilpiridin xlorid, mərkəzi kompozit dizaynı.

СПЕКТРОФОТОМЕТРИЧЕСКОЕ ИССЛЕДОВАНИЕ РАЗНОЛИГАНДНЫХ КОМПЛЕКСОВ ЦИРКОНИЯ (IV) СО СТИЛБАЗО И ЦЕТИЛПИРИДИНИЕМ ХЛОРИДОМ

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Данная работа посвящена исследованию разнолигандного комплекса циркония (IV) со стилбазо и цетилпиридиния хлоридом спектрофотометрическим методом. В качестве экстрагирующего агента был выбран дихлорэтан, а в качестве диспергирующего – ацетонитрил. Определены оптимальные условия комплексообразования. Результаты процесса экстрагирования и комплексообразования были статистически рассчитаны с помощью планов Плакетта-Бермана и центрального композиционного плана (посредством программы Minitab 19). Полученные оценки показали, что практические результаты могут быть выражены в виде статистических результатов. Было установлено, что факторами, оказывающими наибольшее влияние на абсорбцию при комплексообразовании, являются pH и количество лиганда. Для достижения максимальной абсорбции pH должен быть равен 4.5, а количество лиганда составлять 300 мкл. Стехиометрический состав компонентов в комплексе определялся различными методами. Интервалы, подчиняющиеся закону Бера, составляли $2-7.6 \text{ мкг мл}^{-1}$, молярный коэффициент светопоглощения – $2.6 \times 10^4 \text{ лмоль}^{-1}\text{см}^{-1}$, а λ_{max} был равен 590 нм. Разработана методика с высокой селективностью для определения микроколичеств циркония (IV) в пробах воды.

Ключевые слова: цирконий (IV), стилбазо, цетилпиридиния хлорид, центральный композиционный план.