

*Вивчено взаємодію скандію (III) з еріохромціаніном R і поліелектролітом полісульфонілпиперидинілметиленгідроксидом (ПСПМГ). Визначено оптимальні умови комплексоутворення і характеристики трикомпонентної сполуки. Розроблена методика спектрофотометричного визначення скандію у вигляді комплексу з еріохромціаніном і ПСПМГ. Запропоновано тест-шкали для візуального визначення скандію у вигляді потрійних систем на пінополіуретані та бавовні в інтервалі концентрацій від  $1 \cdot 10^{-6}$  до  $1 \cdot 10^{-5}$  моль/л*

*Ключові слова: скандій, еріохромціанін, поліелектроліт, тест-методика, поверхнево-активна речовина, спектрофотометрична методика*

*Изучено взаимодействие скандия (III) с эриохромцианином R и полиэлектролитом полисульфонилпиперидинилметиленгидроксидом (ПСПМГ). Определены оптимальные условия комплексообразования и характеристики трехкомпонентного соединения. Разработана методика спектрофотометрического определения скандия в виде комплекса с эриохромцианином и ПСПМГ. Предложены тест-шкалы для визуального определения скандия в виде тройных систем на пенополиуретане и хлопке в интервале концентраций от  $1 \cdot 10^{-6}$  до  $1 \cdot 10^{-5}$  моль/л*

*Ключевые слова: скандий, эриохромцианин, полиэлектролит, тест-методика, поверхностно-активное вещество, спектрофотометрическая методика*

# POLYELECTROLYTE EFFECT ON SPECTROPHOTOMETRIC DETERMINATION AND INTERACTION OF SCANDIUM (III) WITH ERIOCHROMCYANINE

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## 1. Introduction

Modern analytical chemistry has a diverse arsenal of methods for scandium determination. Instrumental methods of atomic spectroscopy, such as emission spectrographic analysis [1, 2], flow-injection [3], neutron activation [4], mass spectrometric analysis [5] make it possible to accurately determine even small quantities of scandium in geological and biological environments. But various economic factors do not allow to provide all laboratories with this expensive equipment, therefore spectrophotometry is still widely used due to accuracy, simplicity and low cost. By performing physical-chemical research, analysis of various compounds and materials, containing scandium with a certain combination of other elements, spectrophotometric methods are widely used now. To improve these methods, new organic derivatives of scandium are used as analytical forms, which, together with inorganic anions and complex cations, can significantly increase the selectivity of the determination [6].

## 2. Literature review and problem statement

For the photometric method of quantitative determination of scandium in complex objects, numerous reagents with high sensitivity, but relatively low selectivity were pro-

posed. These are bis-(2,3,4-trioxy-phenylazo)-benzidine [6] eriochromcyanine R (ECR) [7], chromazurol S (CAS) [8], arsenazo III [9]. These organic reagents do not differ in their unique specificity and can not be used to detect scandium in the presence of many elements, without the prior separation of scandium from the interfering elements. Extraction using various extragens such as bis-(trifluoromethylsulfonyl)-imide [10], N-(2-hydroxy-5-nonylbenzyl)-hydroxyethylmethylamine [11], 1-alkylcarboxyl-3-methylimidazole-bis (trifluoromethylsulfonyl)-imide [12] or a system of isopropyl alcohol-water-sodium nitride-potassium rhodanide [13] is used to increase the sensitivity and selectivity of spectrophotometric determination of scandium. When choosing an extractant for scandium, along with characteristics such as completeness of extraction and selectivity, it is necessary to take into account the danger (toxicity and volatility of used reagents), efficiency. These requirements are most fully consistent with biphasic water systems based on water-soluble polymers such as polyethylene glycols (PEGs) that are used for separation, concentration and determination of liquid, radioactive elements. For extraction and further determination of scandium, extraction systems of polyethylene glycol-sodium (ammonium) nitrate-water are proposed [8]. Also, multi-ligand complexes are used to enhance the sensitivity and selectivity of scandium detection. In [14], it was proposed to determine scandium in the form of a multi-li-

gand complex with bis-(2,3,4-trioxy-phenylazo)-benzidine and diantiprylmethane (or homologues thereof).

Another promising direction of increasing the sensitivity and selectivity of spectrophotometric techniques is the use of surfactants, or polyelectrolytes (e. g., PSPMG) [15]. Data on complexation of scandium with ECR in the presence of cationic surfactants are presented: for example, cetylpyridinium chloride (CPC), cetyltrimethylammonium bromide (CTAMB), dodecyl trimethylammonium bromide (DTMAB), nonionic surfactant – triton X-100, or anionogenic sodium dodecyl sulfate (SDS). With the specified reagent, modified surfactant, scandium was detected in synthetic alloys, waters [7]. When introducing polyelectrolytes (PEs) or surfactants, an increase in molar absorption coefficients occurs and the contrast of reactions increases (shifts of  $\lambda_{\max}$  by about 50–150 nm). As a rule, there are two models of the mechanism of action of surfactants: solubilization and formation of triple complexes. In the presence of surfactants, hydrophobic molecules can become soluble in an aqueous medium [16].

### 3. The aim and objectives of the study

The aim of the work is to study the system Sc-ECR-polyelectrolyte to increase the sensitivity and selectivity of the photometric determination of scandium, and on this basis to develop the test systems based on the results.

To achieve this aim, the following objectives were solved:

- to investigate the triple complex of scandium with eriochromcyanine R and polyelectrolyte of polysulfonylpiperidinylmethylene hydroxide;
- to determine optimal conditions of complex formation and characteristics of a three-component compound;
- to develop the method of spectrophotometric determination of scandium in the form of a complex with ECR and PSPMG;
- to study the sorption of photometric reagents on scandium of with eriochromcyanine and chromazurol S, their adducts with PSPMG and CPC, on sorbents, and to develop a test system.

### 4. Materials and methods of research applied to the development of spectrophotometric and colorimetric methods for the determination of scandium

Organic reagents chromazurol S, eriochromcyanine R (Fig. 1) of the company Fluka, USA, surfactant cetylpyridinium chloride, polyelectrolyte polysulfonylpiperidinylmethylene hydroxide (average molecular mass of PSPMG – about 2500,  $(a:b=100:(40-90), n=10-12)$ ) were used.

A solution of scandium salt was prepared from scandium oxide by dissolving the accurately weighed amount in the concentrated hydrochloric acid under heating. The brands of all reagents are “chemically pure” or “clean for analysis”. The initial solutions were prepared according to the accurately weighed amounts of the substance, solutions of the given concentration – by dilution of the initial solutions.

The starting solution of polysulfonylpiperidinylmethylene hydroxide was prepared by dissolving the appropriate polymer sample in distilled water.

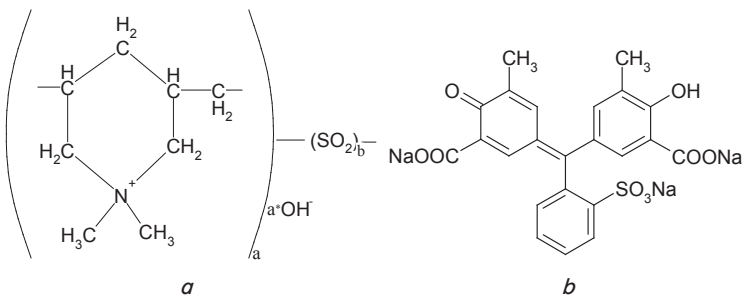


Fig. 1. Formulas of reagents: *a* – polysulfonylpiperidinylmethylene hydroxide; *b* – eriochromcyanine R

Sorbents: aluminum oxide, chromatographic paper, polyurethane foam (PUF) and fabrics (flax, cotton, satin) were investigated as a solid matrix for immobilization of the system.

The pH of the solutions was controlled using a universal ionizer EV-74 (Belarus) using the argentine chloride comparison electrode EVL-1M3.1 and the glass indicating electrode ESL-43-07. Spectra of solutions were recorded on a spectrophotometer Specord M40 (Germany).

Spectrophotometry and potentiometry methods were used for research.

### 5. Investigation of the triple complex Sc-ECR-PSPMG for spectrophotometric and colorimetric determination of scandium (III)

#### 5.1. Results of triple complex research

It is known that the ECR and scandium form a complex with a maximum absorption of about 535 nm [1]. The introduction of PSPMG into the dual Sc-ECR system results in an increase in the absorption intensity ( $A_{Sc-ECR-PSPMG}/A_{Sc-ECR}=4$ ) and a significant bathochromic shift of the absorption maximum ( $\Delta\lambda=60$  nm) of the triple system, which is in the 595 nm region (Fig. 2).

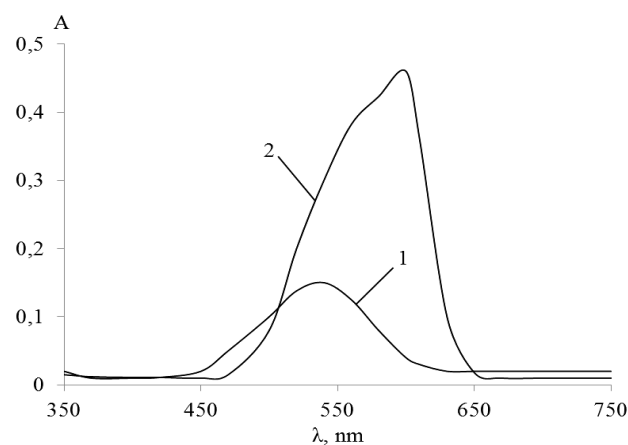


Fig. 2. Absorption spectra of the scandium complex with eriochromcyanine R in the absence (1) and the presence of polysulfonylpiperidinylmethylene hydroxide (2); pH 5.5.  $C(\text{Sc})=4 \cdot 10^{-6}$  M,  $C(\text{ECR})=12 \cdot 10^{-6}$  M,  $C(\text{PSPMG})=8 \cdot 10^{-6}$  M,  $l=1$  cm

For the system Sc-ECR-PSPMG, the order of the merging of the components is important, since the initial solution of salt Sc is strongly acidic and cation  $\text{Sc}^{3+}$  is highly subject to hydrolysis at dilution [1]. The effect of the order of merging of reagents on the interaction in the system was investigated spectrophotometrically. The maximum effect is

observed for the solution obtained by the merging in the sequence: 1 – scandium, 2 – ECR, 3 – PSPMG, 4 – buffer solution. In determining the dependence of complex formation in the system Sc-ECR-PSPMG on the pH value, solutions were used:  $C_{Sc}=1\cdot 10^{-5}$  M,  $C_{ECR}=4\cdot 10^{-5}$  M,  $C_{PSPMG}=4\cdot 10^{-5}$  M. The influence of the pH value on the complex formation was determined spectrophotometrically in the pH range from 2.0 to 9.0. The triple compound is formed in the pH range of 4.5 – 8.0. The maximum and constant value of the optical density is observed in the pH range of 5.5–6.5 (Fig. 3).

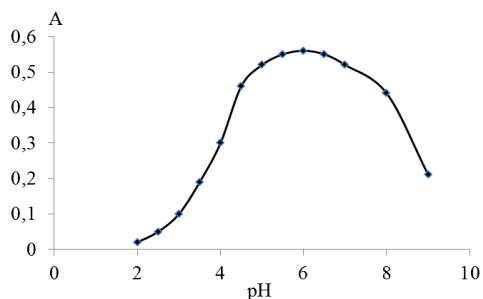


Fig. 3. The dependence of the optical density of the system Sc-ECR-PSPMG on the pH.  $C_{Sc}=1\cdot 10^{-5}$ ,  $C_{ECR}=4\cdot 10^{-5}$  M,  $C_{PSPMG}=4\cdot 10^{-5}$ .  $\lambda=595$  nm,  $l=1$  cm

The Sc:ECR and Sc:PSPMG ratio in the three-component complex was determined by spectrophotometric methods – isomolar series, equilibrium shift [17]. According to the results of the method of isomolar series, scandium and ECR are part of the triple complex in the ratio of 1:1 (Fig. 4, a) and 1:4 (Fig. 4, b). For analytical purposes, the latter is recommended. The Sc:PSPMG ratio, determined by the method of equilibrium shift, was 1:1.

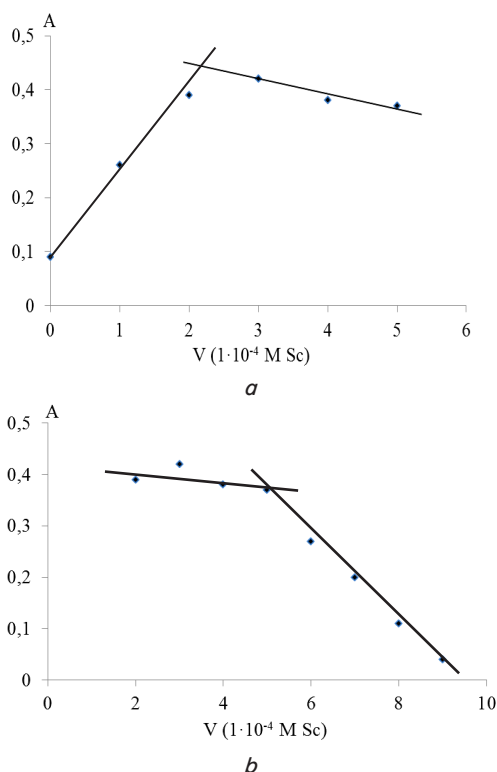


Fig. 4. Determination of the Sc:ECR ratio in the three-component complex by the method of isomolar series.  $C_{PSPMG}=1.01\cdot 10^{-4}$  M=const;  $C_{Sc}+C_{ECR}=4.0\cdot 10^{-5}$  M: a – the ratio of Sc:ECR is 1:1; b – the ratio of Sc: ECR is 1:4

Table 1 presents the comparative characteristics of some ternary complexes of scandium and ECR with surfactants.

Table 1

Chemical-analytical properties of mixed complexes of scandium with eriochromcyanine R and surfactants

P	Ratio Sc:R:P	pH	$\lambda_{ScRP}$ , nm	$\Delta\lambda$ , nm ( $\lambda_{ScRP}-\lambda_{ScR}$ )	$\epsilon\cdot 10^{-5}$	Literature
CTAMB	1:3:1	6.5	610	75	5.6	11
CPC	1:4:1	4.1	595	60	1.3	10
PSPMG	1:4:1	5.5–6.5	595	60	1.8	–
	1:1:1	–	560	25	–	

The calculated molar absorption coefficient of the triple complex Sc-ECR-PSPMG composition (1:4:1) is  $1.8\cdot 10^5$ . The results of the experiment are the basis of the spectrophotometric method for determining the scandium, and used to develop a test system for scandium.

*Construction of calibration curve.* An alternate amount of scandium solution was injected to 25 ml flasks, 6.25 ml of  $1\cdot 10^{-4}$  M ECC solution, 2.5 ml of  $5\cdot 10^{-4}$  M of PSPMG solution, 2 ml of acetate buffer solution (pH 5.5) was diluted to volume with distilled water. The optical density of the obtained solutions was measured in a cell with  $l=1$  cm at a wavelength of 595 nm (Fig. 5). The linearity range of the calibration curve is  $(0.1-3)\cdot 10^{-6}$  mol/L Sc. The correlation coefficient was 0.998. The method is tested on model solutions using the addition method. The obtained data on the determination of scandium are presented in Table 2.

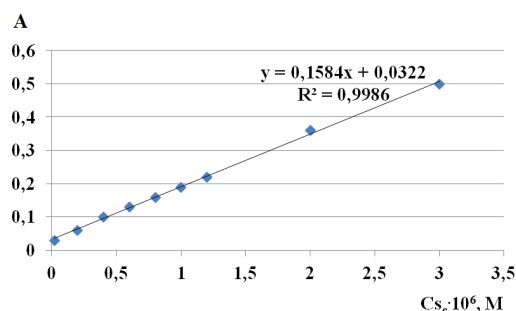


Fig. 5. Calibration curve for scandium content determination.  $C_{ECR}=2.5\cdot 10^{-5}$  mol/L,  $S_{PSPMG}=5\cdot 10^{-5}$  mol/L. pH 5.5;  $\lambda=595$  nm.  $l=1$  cm

Table 2

Results of spectrophotometric determination of scandium content in model solutions

Sc content, mol/L	Found $C\pm\Delta$ ,	$S_r$
$1.5\cdot 10^{-6}$	$(1.56\pm 0.05)\cdot 10^{-6}$	0.03

A twofold and larger excess of aluminum ions prevents the determination of scandium in the form of Sc-ECR-PSPMG in alloys, therefore, we recommend separating the scandium by precipitation with NaOH.

### 5. 2. Sorption of orgreagents, their adducts with PSPMG, CPH and triple complexes

On the basis of the spectrophotometric determination of Sc (III) ECR with PSPMG and CPH, test systems for scandium were developed. For comparison, the data of spec-

trophotometric studies of the Sc(III)-CAS-PSPMG and Sc(III)-CAS-CPC systems, which are given in [24], were used. The results of these studies were used to develop a colorimetric method for scandium.

To develop test systems for scandium, the reagents were sorbed in the form of associates with polyelectrolyte (PSPMG) and surfactant CPC.

As a solid matrix for the immobilization of the system, 6 sorbents were investigated and the optimum parameters of the scandium sorption on solid-state carriers were experimentally determined.

*Preparation of a solid matrix.* The fabric and the paper were cut into squares of 1 cm<sup>2</sup>, (polyurethane foam into cubes) and soaked for 1 hour in 0.1 M hydrochloric acid, and then washed with distilled water to neutralize the reaction.

*Method of investigation of adsorption of the orgreagent on carriers.* Sorption on the carriers was studied in a static mode at room temperature. To this end, a solution, investigated with a particular pH value, concentration, the prepared sorbents were introduced in the conical flasks and mixed with an electromechanical shaker for 15 minutes until the establishment of the sorption equilibrium. The colored sorbent with the adsorbed reagent were removed and dried, and the equilibrium concentration after sorption was determined by the photometric method. Adsorption was calculated by the formulas, using the method of comparison with the standard.

*Investigation of the sorption of the three-component system of scandium – ECR (CAS) – PSPMG (CPC) on PUF and cotton.* It was previously found that ECR (CAS) and their complexes with scandium in the absence and presence of a modifying polyelectrolyte PSPMG and surfactant CPC are better sorbed on PUF and cotton, the color is persistent and there is a contrast of transitions from the reagent to the complexes. Therefore, PUF and cotton are proposed for the development of the test system. Next, a series of solutions of the triple complex with a constant content of ECR (HAS) 2.0·10<sup>-5</sup> mol/L (3.0·10<sup>-5</sup> mol/L) and PSPMG (CPC) 2.0·10<sup>-5</sup> mol/L (4.0·10<sup>-5</sup> mol/L), and variable scandium content were prepared. The optimum pH value of 5.5 was determined by acetate buffer solution. Absorption spectra before sorption were taken.

20 ml of the test solution were taken, placed in a conical flask of 100 ml, and the prepared cotton (PUF) was added. Sorption was performed for 15 minutes, with intensive mixing on an electromechanical shaker. After this, the colored sorbent with the adsorbed reagent were removed and dried. Absorption spectra of the solutions after sorption were taken.

Based on the results obtained, graphs of sorption dependence on the equilibrium concentration Sc were constructed. As an example, an isotherm of adsorption of scandium on PUF (Fig. 6) and sorption parameters of  $A_{gran}$  and  $K_{ads}$  are given (Table 3).

Isotherms of adsorption (Fig. 6, a) can be attributed to the class L, which indicates the high affinity of the adsorbate to the adsorbent. They are linearized in the coordinates of the linear form of the Langmuir equation. Using the linearized isotherms,  $A_{gran}$  and  $K_{ads}$  of scandium in the form of triple complexes were graphically determined for the studied systems on PUF and cotton (Table 3).

*Modification of solid-state carriers.* PUF (cotton) was initially placed in a solution of CAS (ECR) with a concentration of 2·10<sup>-4</sup> mol/L at pH 5.5 and sorption was performed for 40 minutes on an electromechanical shaker and left to dry. Then, the solid-phase carrier with the sorbed orgreagent

was placed in a solution of PSPMG (CPC) of 1·10<sup>-3</sup> mol/L at pH 5.5, stirred for 40 minutes on an electromechanical shaker.

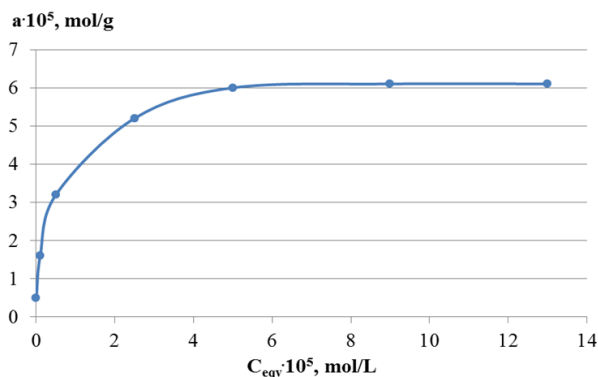


Fig. 6. Isotherm of adsorption of scandium in the form of a triple complex of Sc-ECR-CPC on polyurethane foam; pH=5.0

Table 3

Graphically found characteristics of scandium adsorption in the form of a triple complex of Sc-ECC-CPH on polyurethane foam

$A_{gran} \times 10^5, \text{ mol/g}$	$K_{ads} \times 10^{-3}$
6.67	1.50

*Test scales for determining scandium on solid-state carriers: cotton and PUF* were obtained at a pH of 5.5 in a static mode at room temperature. To this end, 20 ml of the standard solution of scandium with a concentration of (0.1–10)·10<sup>-6</sup> mol/L were placed in a conical flask at 100, pre-modified cotton (PUF) was added, hold for 15 minutes, with vigorous stirring on an electromechanical shaker. After this, the colored sorbent with the adsorbed reagent were removed and dried.

*Spectrophotometric determination of scandium with CAS in the presence of PSPMG* was carried out according to [24]. In a 25 ml flask, a variable amount of scandium solution was added, 3.75 ml of 2·10<sup>-4</sup> M CAS solution, 2.5 ml of 5·10<sup>-4</sup> M PSPMG, 2 ml of acetate buffer solution (pH 5.9) were diluted to volume with distilled water, controlling the pH value. The optical density of the solutions obtained at a wavelength of 610 nm was measured.

*Spectrophotometric determination of scandium with ECR in the presence of CPC.* A series of solutions of the three-component system of eriochromcyanine R with scandium in the presence of CPC were prepared. The aliquots of 6.25 ml of 1·10<sup>-4</sup> mol/L of ECR and 1.25 1·10<sup>-3</sup> mol/L of CPC were taken, introduced in each 25 ml flask and the following scandium aliquots were added: 0; 2.50 ml; 6.25 ml; 12.50 ml (1·10<sup>-5</sup> mol/L initial scandium solution); 1.88 ml; 2.50 ml; 6.25 ml (1·10<sup>-4</sup> mol/L scandium solution). In all solutions, the pH of 5.0 was determined using an acetate buffer solution. The prepared solutions were diluted to volume with distilled water in 25 ml flasks, mixed and the optical density was recorded in a dish with  $l=1$  cm at  $\lambda_{max}=595$  nm. The interval of the linearity of the calibration curve is (1–10)·10<sup>-6</sup> mol/L Sc.

## 6. Discussion of the development results of test systems for scandium

To develop a colorimetric method with improved metrological characteristics, the properties of the analytical

reaction were varied, changing the sequence of modification and immobilization. Modification of the sorbent with subsequent immobilization with organic reagent (OR) on the sorbent; immobilization of OR on the sorbent with subsequent modification by polyelectrolyte; immobilization of the CAS (ECR)-Sc complex on the modified sorbent; immobilization of triple complexes on the Sc-OR-modifier was studied for optimization of the process.

The scandium adsorption isotherms were constructed,  $A_{gran}$  and  $K_{ads}$  sorption parameters were calculated.

On the basis of the obtained results, a test system was proposed for the visual determination of scandium in the form of triple complexes in the range of concentrations from  $1 \cdot 10^{-6}$  to  $1 \cdot 10^{-5}$  mol/L: Sc-ECR-CPC, Sc-CAS-PSPMG on PUF; Sc-ECR-CPC, Sc-CAS-PSPMG on cotton.

For the quantitative determination of scandium, the test scales were scanned, the parameters of the color functions were calculated, the values of the R, G, and B channels were found. The dependence graph of the intensity of the channel on the concentration was constructed, and according to the data of the approximation, the graph was chosen in which the approximation value would be closer to 1. The equations of calibration curves are given in Table 4.

Table 4

The equations of the calibration curves of the optimal channels

No.	Triple system	Equations of calibration curves	Correlation coefficient
1	Sc-CAS-PSPMG on PUF	$y = -1.0713x + 10.91$	$R^2 = 0.9692$
2	Sc-HAS-PSPMG on cotton	$y = 1.2565x - 1.7125$	$R^2 = 0.9578$
3	Sc-ECR-CPC on PUF	$y = -2.5916x + 13.614$	$R^2 = 0.9939$
4	Sc-ECR-CPC on cotton	$y = 1.7576x + 13.34$	$R^2 = 0.9035$

The best results were observed when determining scandium in the form of the triple complex of Sc-ECR-CPC on

PUF with a color change from pink to blue. Determination of scandium on PUF and cotton, modified alternately with PSPMG and CAS with a change in color from light brown to blue also yielded good results.

## 7. Conclusions

1. The effect of cationic water-soluble polyelectrolyte polysulfonylpiperidinylmethylene on the interaction of scandium (III) with an organic reagent eriochromcyanine was studied. It was shown that polyelectrolyte improves the chemical and analytical characteristics of this reagent: when adding PSPMG into the dual system of Sc-ECR, the absorption intensity is increased fourfold.

2. For the formation of the triple complex Sc-ECR-PSPMG, the optimal pH range (5.5–6.5) and the order of merging of the components (1 – scandium, 2 – ECR, 3 – PSPMG, 4 – buffer solutions) were determined, the composition of the Sc-ECR-PSPMG complex (1:4:1) was spectrophotometrically determined and the molar absorption coefficient ( $1.8 \cdot 10^5$ ) was calculated.

3. The method of spectrophotometric determination of scandium in the form of a complex with eriochromcyanine and PSPMG was developed, the calibration curve, the linearity of which is  $(0.1-3) \cdot 10^{-6}$  mol/L Sc was constructed, the correlation coefficient is 0.998. The method is tested on model solutions using the addition method.

4. For the development of the colorimetric method for scandium, sorption on PUF and tissue of orgreagents (ECR and HAZ), their adducts with PSPMG and CPH and triple complexes was investigated. Test scales for the visual determination of scandium in the form of triple systems Sc-ECR-CPH, Sc-HAZ-PSPMG were proposed, calibration curves were constructed in the range of concentrations from  $1 \cdot 10^{-6}$  to  $1 \cdot 10^{-5}$  mol/L. The colorimetric method of scandium identification in solutions is proposed.

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