EXTRACTION-SPECTROPHOTOMETRIC DETERMINATION OF Zn (II) ION WITH 1-(2-HYDROXY-1-NAPHTOYAZO)-2-NAPHTHOL-4-SULFOCID SOLUTION

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Abstract. *Recommended an extraction-spectrophotometric method for determining zinc ions with a solution of 1-(2-hydroxy- 1-naphthoiazo)-2-naphthol-4-sulfonic acid. Found optimal conditions: the volume ratio of the organic and aqueous phases is 1:9, the distribution coefficient is 0.95. The light absorption of the complex is 410 nm, the absorption of the reagent is 500 nm, the contrast is 90 nm, this proves the high sensitivity of the developed method. It has been shown that the determination of scandium ions is highly accurate with a lower detection limit of 1.14 μg/L and a correlation coefficient of 0.9999. The developed method for determining scandium ions is applied to the analysis of model mixtures and the relative standard deviation does not exceed 0.0015.*

Keywords: *extraction-spectrophotometry, distribution coefficient, 1-(2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfonic acid, zinc.*

Introduction

Zinc is used in the production of ligatures, high-power metal halide lamps, analytical standards, medical devices, household appliances and lasers. In nature, zinc is found in minerals and ores, and in the waste waters and waste of factory process of gold, silver and copper.

Therefore, development of scandium ion detection and extraction methods is one of the actual problems. In the literature, zinc ion were studied by extraction spectrophotometric methods with phosphorus organic compounds - dioxide methylenediphosphine [1], oxides (dialkylcar- bamoylmethyl) diarylphosphine (KMFO) [2], XAD-4 [3], eriochromcyanine [4], chlorocyanogenformazan [5], phosphorylated calixarenes [6], 2-amino-4-(m-tol- yazo)pyridine-3-ol (ATAP) [7], polyethylene glycol PEG-3000 (C3H7OH)–NaNO3 (NaNO3 + KSCN)–H2O [8], phosphoryl stored podands [9], 2-hydroxy-5-T-bu- tylphenol-4¢-metoxyazobenzene (HR) [10], antipyrine - sulfosalicylic acid [11] by forming a complex. Methods for the extraction-conductometric determination of zinc in kaolin with 2,7-dinitroso-1,8-dihy- droxynaphthalene-3,6-disulfonic acid with tributyl phosphate and trioctylphosphine oxide have also been developed [12,13]. In addition, selective and rapid determination procedure for Th, Zr and Zn in rock samples using adsorbent silica modified with arsenazo-III (SAR- III) was synthesised by a single step process in presence of cationic surfactant cetyltrimethylammonium bromide. [14]. The most effective extractants of zinc are organophosphate compounds, but their reextraction process is difficult.

Currently, the search for effective and economically convenient extraction agents for the extraction of this metal remains an actual problem.

The aim of this work is to develop a methodology for the extraction-spectrophotometric detection of zinc (II) ion with 1-(2-hydroxy-1-naphthoyazo)- 2-naphthol-4-sulfonic acid reagent.

For this, the effect of the nature of the solvent, the acidity of the solution, and the partition coefficient of Zn (II) between the aqueous and organic phases was studied.

According to the Gaussian method electron densities of reagent were 0.203-0.247 of hydroxyl groups in the ring and 0.070-0.042 of -N=N- group. And this gives an opportunity to form a chemical bond with metal ions.

Experimental part

Newly synthesized 1-(2-hydroxy-1-naphthoiazo)-2- naphthol-4-sulfonic acid was used as an extractant to separate the zinc (II) ion in the solution. Toluene, chloroform and benzene "ch.c." or "c.f.a." certain organic solvents were used.

Solutions containing Zn (II) and other metals (Ce, Y, Pr, Te, Eu, Tm, Tb, Yb) with a concentration of 10^{-5} mol/l were prepared by dissolving the appropriate salts.

Zn (II) ion concentrations in initial and equilibrium solutions were determined by spectrophotometry (EMC-30PC-UB spectrophotometer). The acidity of the solution was determined by potentiometric titration with KOH solution (pH meter / mV / TEMP meter P25 Ecomet (Korea)).

Results and discussions

Knowing the effect of the nature of the organic solvent on the extraction rate, the effect of different organic solvents was studied. The nature of the organic solvent has a strong influence on the distribution constants of the reagent and enhances complex formation. In this study, we used chloroform $CHCl_3$ (P=1.49), benzene C_6H_6 (toxic, P=0.88) and toluene $C_6H_5CH_3$ (P=0.87).

The reason is that they are insoluble in water and differ from the dissociation constants of the reagent and the stability of the complex. Received data are shown in Figure 1.

First, the light absorption maximum of the 0.1% alcohol solution of 1-(2-hydroxy-1-naphthoyazo)- 2-naphthol-4-sulfonic acid reagent was measured in a spectrophotometer. Then, 1.0 ml of 65 μg/ml zinc (II) solution, 2.0 ml solution of the 0.1% 1-(2-hydroxy- 1-naphthoiso)-2-naphthol-4-sulfonic acid reagent in al- cohol, 12.0 ml of distilled water and 2.0 ml of chloroform, toluene, benzene solution as an extractant were added to a 50 ml separatory funnel. The mixture was shaken for 2 min and allowed to stand. The organic layer was separated in a separatory funnel, and the optical density of the obtained complex was measured compared to the reference solution (λmax= 410, *l* = 1,0 sm). The measurement results are presented in Figure

1. The same procedures were repeated with solutions of the reagent 1-(2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfonic acid in toluene and in benzene.

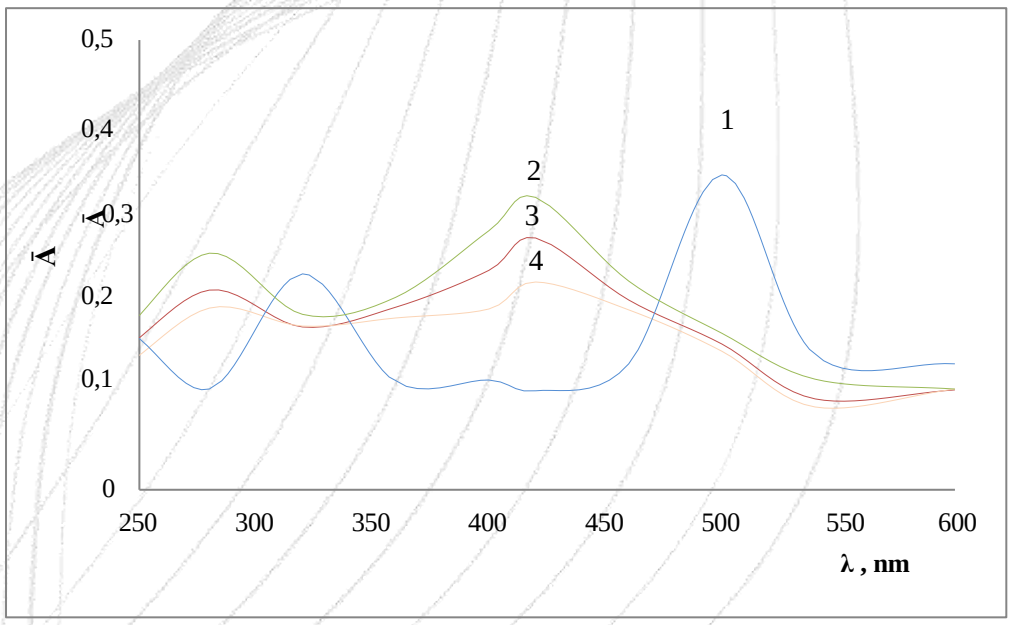

Figure 1. Light absorption spectra of 0.1 % alcohol solution of 1-(2-hydroxy-1-naphthazo)-2-naphthol-4-sulfonic acid reagent (1) and its mixtures in chloroform (2), toluene (3) and benzene (4)

According to the obtained results, the extraction rate of zinc was the highest in the mixture of 1-(2- hydroxy-1-naphthoyazo)-2-naphthol-4-sulfonic acid reagent in chloroform. For this reason, chloroform was chosen as the extractant for the 1-(2-hydroxy-1- naphthazo)-2-naphthol-4-sulfonic acid reagent for the extraction of zinc.

It can be seen from the above picture that 1-(2- hydroxy-1-naphthoiso)-2-naphthol-4-sulfonic acid extracted zinc (II) ion well in chloroform, which is due to its good solubility and high density in chloroform [15].

The influence of the volume ratio of the aqueous and organic phases on the level of extraction of metals in the extraction with a standard solution of zinc in a solution of 1-(2-hydroxy-1-naphthazo)-2-naphthol- 4-sulfonic acid in chloroform was studied. The degree of separation of zinc was determined in the following proportions of volumes of organic and aqueous phases: 1:1; 1:2; 1:5; 1:8; 1:9; 1:10; 1:11; 1:15 a.m.; 1:20;. It was found that changing the volume ratio of organic and aqueous phases from 1:5 to 1:10 does not lead to a decrease in the level of extraction of elements.

Table 1 graphically shows the dependence of the degree of separation of scandium with a solution of 1- (2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfonic acid in chloroform on the ratio Vo:Vs.

Table 1.

Dependence of the degree of separation (R%) in the extraction of zinc (II) ion on the volume ratio of aqueous and organic phases

Organic and aqueous phase ratio Vo:Vs	1:1	1:2	1:5	1:8	1:9	1:10	1:11	1:15	1:20
Zn^{2+}, R%, degree of separation	90	91	92	93	93	93	92	92	92

Based on the obtained results, it was found that the extraction rate of zinc extraction was the highest when the ratio of aqueous and organic phases was 9:1. For this reason, it was determined that the ratio of aqueous and organic phases was 9:1 for the extraction of zinc.

In order to study the effect of environmental acidity, HCl, H_2SO_4 and HNO_3 acids were selected, and 1.0 ml of 65 μg/ml zinc (II) solution, 2.0 ml solution of the 0.1% 1-(2-hydroxy-1-naphthoiso)-2-naphthol-4-sulfonic acid reagent in alcohol, 3.0 ml of a 0.1 M nitric acid solution, 12 ml of distilled water and 2.0 ml of a chloroform solution were added to a 50 ml separatory funnel. The mixture was shaken in the separatory funnel for 2 minutes and then allowed to stand.

The organic layer was separated in a separatory funnel, and the optical density of the obtained complex was measured compared to the reference solution (λmax= 410, *l* = 1,0 sm).

The measurement results are given in table 2. Based on this methodology, the work was performed several times with 0.1 M solutions of sulfuric and hydrochloric acids. Based on the obtained results, it was determined that nitric acid is the most optimal for extracting scandium.

In the extraction of metals, the values of the degree of separation are different in different acids and reach a maximum level in certain acids.

Table 2.

Effect of aqueous phase acidity on scandium ion degree of separation (R%).

Ion	Acid	Degree of separation, R %
Zn^{2+}	hydrochloric acid	89
Zn^{2+}	sulfuric acid	91
Zn^{2+}	nitric acid	94

Based on the obtained results, the level of extraction of zinc in nitric acid environment had the highest value. For this reason, it was found that the use of nitric acid is effective in forming a complex of zinc (II) ion with 1-(2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfonic acid.

In order to study the effect of the amount of selected acid on the level of extraction, 1.0 ml of 65 μg/ml zinc (II) solution, 2.0 ml solution of the 0.1% 1-(2-hydroxy-1-naphthoiso)-2-naphthol-4-sulfonic acid reagent in alcohol, 3.0 ml of different concentrations solutions of the nitric acid solution at 0.025 M; 0.05 M; 0.1 M; 0.125 M; 0.15 M, 12 ml of distilled water and 2.0 ml of chloroform solution were added to a 50 ml separatory funnel, and then optical densities were measured. The measurement results are shown in Table 3.

Table 3.

Effect of nitric acid concentration on degree of separation in scandium extraction

HNO_3 , C_M	0.025	0.05	0.1	0.125	0.15
Degree of separation R, %	88	92	95	94	93

As can be seen from the obtained results, the degree of separation (R%) of scandium with 0.1 M nitric acid solution has reached the maximum value.

In order to study the dependence of the complex compound on the amount of added reagent, 1.0 ml of 65 μg/ml zinc (II) solution, different volumes of 0.1% 1-(2-hydroxy-1-naphthoiso)-2-naphthol-4-sul-fonic acid reagent in alcohol (0.5; 1.0; 1.5; 2.0; 2.50; 3.0 ml), 3, 0 ml of 0.1 M nitric acid solution, 12 ml of distilled water and 2.0 ml of chloroform solution as an extractant were added to a 50 ml separatory funnel and the optical densities were studied and is given in Table 4.

Table 4.

Dependence of the optical density on the amount of reagent (λ max =410 nm , *l* = 1.0 cm, extragen= chloroform, C_M = 0. 1 HNO_3)

1-(2-Hydroxy-1-naphthoiso)-2-naphthol-4-sulfonic acid V, ml	0.5	1	1.5	2	2.5	3
Ā	0.145	0.211	0.274	0.325	0.316	0.308

Conclusion

The obtained results show that the developed extraction-spectrophotometric method is recommended for the detection and separation of scandium (III) ion from the composition of technological solutions with 1-(2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfoacid reagent in the presence of HNO_3 acid.

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