DEVELOPMENT OF A MODERN METHOD FOR THE DETERMINATION OF FERROUS ION (III)

Yulchiyeva S.T
Andijan State Medical Institute **(PhD)**
170127 Uzbekistan
Smanova Z.A
National University of Uzbekistan named after Mirzo Ulugbek, **prof**
700174 Uzbekistan
Xolboyev Yu.H
Andijan State Medical Institute **(DSc)**
170127 Uzbekistan
Khusanov U.Sh.
Andijan State Medical Institute **(PhD).**
170127 Uzbekistan
O'rmonov A.A.
Andijan State Medical Institute **(Ass)**
170127 Uzbekistan

Аннотация. Показана возможность использования 1-(4-антипирилазо)-2-нафтол -3,6- дисульфокислого натрия, иммобилизованного на полиакрилонитрильном волокне, в качестве реагента для определения железа(III). Оптимизированы условия иммобилизации и комплексообразования. Разработанная сорбционно-фотометрическая методика определения железа(III) применена к анализу природных вод (Sr<0,03), продолжительность которого не более 15 мин.

Ключевая слова: Спектрофотометр, железа, реагент, тяжелый металл, полимер, иммобилизация, анализ, сорбент.

Abstract. The possibility of using 1- (4-antipyrilazo) -2-naphthol -3,6-sodium disulfonic, immobilized on polyacrylonitrile fiber, as a reagent for the determination of iron (III) has been shown. The conditions for immobilization and complexation have been optimized. The developed sorption-photometric technique for the determination of iron (III) was applied to the analysis of natural waters (Sr <0.03), the duration of which was no more than 15 minutes.

Keywords: Spectrophotometer, iron, reagent, heavy metal, polymer, immobilization, analysis, sorbent.

Annotatsiya. Temir(III) ni aniqlash uchun reagent sifatida poliakrilonitril tolasida immobilizatsiya qilingan 1-(4-antipirilazo)-2-naftol-3,6 - natriy disulfoksiddan foydalanish imkoniyati ko'rsatilgan. Immobilizatsiya va komplekslanish sharoitlari optimallashtirilgan. Tabiiy suvlarni tahlil qilishda temirni(III) aniqlashning

ISSN 3030-3907

sorbsion-fotometrik usuli ishlab chiqilgan (Sr<0,03), uning davomiyligi 15 daqiqadan oshmaydi.

Kalit so'zlar: spektrofotometr, temir, reaktiv, og'ir metall, polimer, immobilizatsiya, tahlil, sorbent.

Introduction

The task of determining heavy and toxic metals in various environmental objects is one of the central problems of modern analytical chemistry and ecology, dictated by the toxic effect of these ecotoxicants on all living things, and, consequently, their negative impact in general and on the ecological balance in particular. Currently, many different methods for determining heavy and toxic metals are recommended, which have significant drawbacks, most of which, due to various circumstances, are inaccessible to modern analytical monitoring, due to insufficient equipment of chemical laboratories with appropriate equipment, a shortage of reagents, high cost and unavailability of materials for analysis [1].

In recent decades, physical (primarily spectral) methods of analysis have been developing most intensively, characterized by high metrological characteristics, productivity and occupying a worthy place in chemical analysis. However, many industry laboratories are focused primarily on the use of spectrophotometry, which is explained by the provision of appropriate equipment, cost-effectiveness, as well as simplicity and reliability. The spectrophotometric method of analysis is known to be one of the most widely used analytical techniques in practice [2]. However, the limitation of the spectrophotometric method in practical use for environmental studies is obvious due to its low sensitivity and very low maximum permissible concentration (MPC) for various elements in environmental objects. Therefore, the development of sensitive, reliable, simple and cost-effective methods for determining heavy and toxic metals is an urgent task of modern analytical chemistry. One of the existing methods that meets these requirements is spectrophotometry using immobilized organic reagents (OR) [3].

The task of determining heavy and toxic metals in various environmental objects is one of the central problems of modern analytical chemistry and ecology, dictated by the toxic effect of these ecotoxicants on all living things, and, consequently, their negative impact in general and on the ecological balance in particular. Currently, many different methods for determining heavy and toxic metals are recommended, which have significant drawbacks, most of which, due to various circumstances, are inaccessible to modern analytical monitoring, due to insufficient equipment of chemical laboratories with appropriate equipment, a shortage of reagents, high cost and unavailability of materials for analysis [1].

ISSN 3030-3907

In recent decades, physical (primarily spectral) methods of analysis have been developing most intensively, characterized by high metrological characteristics, productivity and occupying a worthy place in chemical analysis. However, many industry laboratories are focused primarily on the use of spectrophotometry, which is explained by the provision of appropriate equipment, cost-effectiveness, as well as simplicity and reliability. The spectrophotometric method of analysis is known to be one of the most widely used analytical techniques in practice [2]. However, the limitation of the spectrophotometric method in practical use for environmental studies is obvious due to its low sensitivity and very low maximum permissible concentration (MPC) for various elements in environmental objects. Therefore, the development of sensitive, reliable, simple and cost-effective methods for determining heavy and toxic metals is an urgent task of modern analytical chemistry. One of the existing methods that meets these requirements is spectrophotometry using immobilized organic reagents (OR) [3].

Methodology of the study. The work was carried out in static and dynamic modes. In the static mode, 10.0 ml of 0.5 □ reagent solution (pH 6-7) were added to 50.0 ml flasks, the carrier disk was lowered into it, and mixed for 5-8 min. Holding the carrier with a glass rod, the reagent was poured out, the immobilized carrier was washed with distilled water and lowered into the analyzed solution. In the dynamic mode, the analyzed solution was passed through the immobilized disk at a rate of 10 ml/min, and then the study was started.

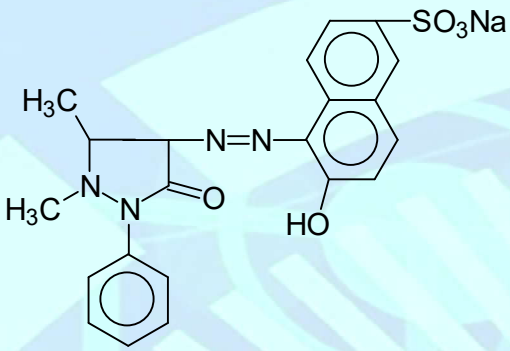
Results and discussion

Fibrous materials modified with various anion-exchange groups were tested as a carrier for AAHC immobilization. The best analytical effect was achieved by sorption on a fibrous carrier modified with hexamethylenediamine (HMDA) (sorbent CMA-1) with subsequent complexation on the solid phase, and it was found that AAHC is retained quite firmly on such a carrier, and therefore the CMA-1 - AAHC system was selected for further studies.

Based on IR spectroscopic and potentiometric studies, the following chemical structure of the polymer fiber was proposed when processing HMDA, and a fiber containing both weakly basic and strongly basic functional groups was obtained [10].

The AAHC reagent is a dark red powder, highly soluble in water and forming colored compounds with ions of many metals. In an acidic medium, it interacts with ions of cobalt, nickel, titanium, and at pH 3-7 also with ions of mercury and iron. The structural formula of the azo dye has been proven by IR spectroscopic studies

ISSN 3030-3907



The reagent was immobilized on the sorbents used from aqueous solutions, and to remove the unfixed reagent, the carriers were washed with distilled water and stored in Petri dishes. The amount of immobilized reagent in the wash waters was determined photometrically by its residual concentration. When studying the degree of reagent retention, in the pH range of 1-7, the immobilization efficiency was 80-90%.

The effect of concentration and immobilization time was studied in the range of 1 10-6 – 1 10-3 M AAHC and 3-30 min., respectively (Table 1).

Table 1

Spectrophotometric characteristics of AAHC fixed on the SMA-1 sorbent

Reagent	Carrier	Immobilization time, min	pH	$\lambda_{max.}$ Reagent , HM	λ_{max} kompleks , HM	, M	
						In solution	On the carrier
AAHC	CMA-1	6	6-7	520	630	$1{,}0\cdot10^{-4}$	$8{,}0\cdot10^{-5}$

Experiments have shown that the reaction of iron(III) with AANS on a solid sorbent is as contrasting as in solution (□□=110 nm). Comparison of the spectra of the immobilized and original organic reagents has shown that the functional-analytical groups responsible for complex formation in the immobilized and native reagents are similar, which indicates the preservation of the reagent structure in the immobilized state (Table 2).

Table 2

Characteristic frequencies and their assignment in the IR spectra of the reagent and complex (cm-1)

Compound	ν_{-SO3H}	$\nu_{-N=N-}$	ν_{-OH}	ν_{-CN}	ν_{-O-Fe}
Reagent	1200	1628	3550	2244	-
Immobilized reagent	1172	1629	3450	2244	-
Complex	1171	1600	3441	2204	598

ISSN 3030-3907

Comparison of the IR spectra of the complex with that of the organic reagent showed that the spectrum of the complex contains a band at 598 cm-1, which is absent from the IR spectrum of the reagent and is attributed according to [11] to vibrations responsible for the stretching vibrations of the -O-Fe bond.

When passing iron solutions of different volumes through disks of the immobilized reagent, results close in accuracy were obtained (Table 3).

Table 3

Results of determination of iron from different volumes (n=3; P=0.95; CFe=50 µg)

Sample volume, ml	50,0	100,0	200,0	500,0	800,0	1000
Iron found, mg	49,8	49,6	49,7	49,6	49,1	48,7
Concentration factor	99,6	99,2	99,4	99,2	98,2	97,4

It was found that the volume of the analyzed solution in certain quantities does not have a significant effect on the degree and amount of metal extraction and the coefficient of its concentration.

When comparing the results of the analysis obtained in the solution and on the immobilized sorbent, it was shown that with immobilization, the pH of complex formation shifts to a more acidic region and the detection limit decreases by an order of magnitude (Table 4).

To assess the selectivity of reactions, numerous experiments were carried out to determine the effect of foreign ions on the results of determining iron ions. The maximum permissible quantities of foreign elements were found, i.e. those concentrations at which the error in determination does not exceed ±2%. According to the data obtained, the maximum permissible ratios of the foreign element to the ion being determined were found, which were taken as the selectivity factor (Table 4).

Table 4

Comparative characteristics of some analytical parameters of the immobilized reagent AAHC obtained in solution and on a carrier

System	λ_{max}, HR, нм	λ_{max}, MeR, нм	pH	Detection limit	Concentration of interfering cations, мкг
Solution	476	616	4-5	0,02	Pb (10), Cd(20), Co(40), Al(80), Zn(50),Cu(2), Ni(50) и др.
Carrier	520	620	3-4	0,008	Ca, Mg (1000), Cd(500), Co, Ni(350), Al(300), Cr(250), Hg

ISSN 3030-3907

					(70), Pb(50) Cu(20), Zn(100)

As can be seen from Table 4, IMOR allows determining iron in the presence of copper (1:20), lead, mercury (1:50) and other cations.

The results of the study allowed us to propose the SMA-1 -AANS system for use as a sensitive layer of a sensor for iron ions, which was used to determine in samples of wastewater from industrial zones.

Determination of iron (III) using immobilized AANS on fibrous materials allows for its selective determination without elution, which significantly increases the rapidity of analysis with a decrease in the detection limit by an order of magnitude.

LIST OF REFERENCES

1. Upor E., Mohai M., Novak D. Photometric Methods For Determination Of Traces Of Inorganic Compounds. Moscow: Mir.1985. 359 P.

2. Unified Methods For Monitoring Background Pollution Of The Environment. Moscow: Gidrometeoizdat.1986.182 P.

3. Brykina G.D., Marchak T.V., Krysina L.S., Belyavskaya. Sorption-Photometric Determination Of Iron (Iii) Using A Modified Anion Exchanger Av-17 // Zhurn. Analit. Chemistry. 1983.V.38.№8.P.1463-1469.

4. Madusmanova N.K., Smanova Z.A., Zhuraev I.I. Properties Of The New Analytical Reagent 2-Hydroxy-3-Nitrosonaphthaldehyde// Journal Of Analytical Chemistry, 2020, 75(1), Pp. 135–138.

5. Morosanova E.I., Velikorodny A.A., Nikulin I.V., Puganova E.A., Zolotov Yu.A. Xerogels Modified With 1-(2-Pyridylazo)-2-Naphthol And Xylenol Orange. Indicator Tubes For The Determination Of Copper(Ii) And Iron(Iii) In Solutions // Zhurn. Anal. Chemistry. 2000. Vol. 55. No. 5, Pp. 539–545.

6. Myasoedova G.V., Nikashina V.A., Molochnikova N.P. Properties Of New Types Of Fibrous Sorbents With Amidoxime And Hydrazine Groups // J. Analytical Chemistry. 2000. Vol. 55. No. 6. Pp. 611-615.

7. Dedkova V.P., Shvoeva O.P., Savvin S.B. Test Method For Determination Of Iron(Ii), Cadmium, Lead From One Sample On Fibrous Sorbent Panv-Av-17 // J. Analytical Chemistry, 2006. Vol. 61. No. 8. Pp. 880-885.

8. Korostelev P.P. Preparation Of Solutions For Chemical-Analytical Works. Moscow: Chemistry. 1962. Pp. 213-214.

9. Lurye Yu.Yu. Handbook Of Analytical Chemistry. Moscow: Chemistry. 1979.480 P.

10. Madusmanova N.K., Smanova Z.A., Zhuraev I.I. Properties Of The New Analytical Reagent 2-Hydroxy-3-Nitrosonaphthaldehyde// Journal Of Analytical Chemistry, 2020, 75(1), Pp. 135–138.

ISSN 3030-3907

11. Bellamy L. New Data On The Ir Spectra Of Complex Molecules. Moscow: Mir 1971.-318 P.

ISSN 3030-3907