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PRODUCTION OF SORBTSION-SPECTROSCOPIC METHODS OF DETERMINATION OF RHENIUMION

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Abstract:
A new spectrophotometric method for the determination of rhenium ion has been developed and the limits of detection of rhenium ions contained in industrial wastewater and sludge are presented, calculations of standard deviation values and an immobilization technique are given. Optimal conditions are selected and the mechanism of reagent immobilization and complex formation is shown, which are studied by X-ray fluorescence and IR spectroscopy.

Keywords: Bismuthol - 2, sorption-spectroscopic method, immobilization, fibrous sorbent PPM-1, X-ray fluorescence and IR - spectroscopic analysis.

INTRODUCTION

The composition of rhenium (Re), selenium (Se) and a number of other impurity elements in rocks found in a shale deposit in the central part of the Volga in the southeast of Russian deposits has been studied. The metal ions found in the deposit are enriched in organic matter (15-37 wt.% in carbonaceous shale) and contain long-lived oil shale interlayers up to 0.7 m3 thick. The maximum concentrations of Re and Se in shales averaged 0.13 g/t (0.09-0.19) for Re and 10.39 g/t (6.54-12.4) for Se, respectively. Statistical analysis of the data obtained made it possible to establish that the combined Re and Se compounds are closely related to organic substances and to Cu, Cd, U, and Ag. [one].

This is based on the method for determining rhenium ions depending on the concentration of sorption solutions of ion exchangers and functional group reagents, as well as on the kinetics of this method and the regeneration properties of ion exchangers of ligand reagents, metal ions and acid concentrations in the solution, the duration of the number of sorption and desorption cycles were studied. The optimal conditions for sorption and desorption of perrhenate anions have been established. The eluent used in this case showed a good result, a solution of 2N H2SO4, 10% NH4OH, 1N. NaOH and 1N. H3PO4, It has been established that the sorbent containing polyethyleneamine groups is regenerated better than others and is suitable for reuse without significant losses during re-treatment in a series of adsorption-desorption processes. The results obtained show that this sorbent can be used as an adsorbent for efficient removal of rhenium ions from sulfate solutions. [2].

To detect rhenium from molybdenum and lead concentrates, a method of dissolving samples in acids and microwave atomic emission analysis of rhenium have been proposed. It is shown that the range of metal concentrations for detection is 0.05-0.5% by mass, and the standard repeatability range is 0.002-0.01%. After detection, the content of rhenium ions in the resulting solutions was determined using an atomic emission plasma spectrometer. The most accurate line at 227.5 nm for the rhenium ion was chosen experimentally for analysis. Sample weight 0.1-0.2 g, as a reagent used a solution of nitric acid (1:2) in the ratio. The temperature was maintained at 20°C for 15 and 30 min, and the optimal conditions for dissolving the sample in acid for 1 and 2 N were selected. 22°C, and according to the results, a temperature of 20°C was chosen for 30 min [3].

Studies have been carried out, kinetic and equilibrium isothermal models have been developed in a static technique to demonstrate the processes associated with the absorption of rhenium ions by the Purolite A-170 ion exchanger [4]. Adsorption isotherm models are subdivided in detail to implement the adsorption mechanism. The results are generally such that all models used agree satisfactorily with laboratory data, but the Freundlich and D-R isothermal models showed a selectivity factor (R2) of more than 0.99 pmm and can therefore be used to control

process equilibrium. In addition, the maximum wavelength (pmm) from the Langmuir isotherm under current conditions was calculated to be 166.67 mg/g. An analysis of the results showed that the second-order adsorption rate was determined correctly relative to the others [5].

MATERIALS AND METHODS:

1. To prepare a working solution of 0.01% bismuth-2 reagent, weigh 0.01 g of bismuth-2 reagent on an analytical balance, transfer it to a 100 ml volumetric flask and dilute with water to the mark. The prepared solution was diluted and used for further work. To prepare a standard solution of Re7+ ion with a concentration of 1 mg/ml, 0.732 g of ammonium perrinate salt was weighed out, placed in a 100-ml flask, and made up to the mark with distilled water. This solution was used in subsequent studies [7]. [151].

2. When preparing a 1.0·10-1 M hydrochloric acid solution was prepared by diluting concentrated hydrochloric acid.

3. To the universal buffer mixture of various pH (1-12) buffer solutions was added 0.04 M (H3BO3, H3PO4, CH3COOH) 0.2 M NaOH solution.

4. For the preparation of fibers, 0.2 g of fibers synthesized at the Department of Polymer Chemistry was extracted. The fibers were chlorinated by immersion in 0.1M hydrochloric acid. Rinse with distilled water until neutral. Stored in a wet Petri dish.

Immobilization procedure: 10 ml of 0.1% bismuth-2 reagent was placed in 50.0 ml beakers, 0.2000 g of fiber was added and stirred for 5-8 minutes with a glass rod. Then the fiber was washed with distilled water and the amount of the reagent deposited on the fiber was measured, and the results showed that the immobilization of the bismuth-2 reagent in the fiber is determined by the following formula [8].

P-NH2+ HC1 →P-+NH3C1-

P-+NH3C1- + S – R →P-+NH3 – S – R – N ¨Me

Here, P-NH2 polymer carrier

Reagent P–NH2–S–R–bismuthol–2

THE DISCUSSION OF THE RESULTS

1-Table

Optimal support and complexation with the bismuthol-2 reagent

Волокно	А до иммобилизации (висмут-2)	А после иммобилизации (висмут - 2)	Δ А
ППА-1 [H^+] **ППМ-1**	0,35 **0,700**	0,12 **0,290**	0,23 **0,410**

As can be seen from Table 1, PPM-1 is the best immobilized fiber. therefore, this fiber has been used in later work.

Influence of the amount of reagent on sorption

10 ml of the reagent 2.5·10-4 bismuthol-2 and 0.2000 g of PPM-1 fibers were added to beakers with a capacity of 50 ml, and a reagent solution of different concentrations and a metal solution of the same concentration were sorbed for 30 min. The degree of sorption was determined by studying the optical densities before and after the reaction [9–11]. The results are presented in table 2

2-Table

Dependence of immobilization on reagent concentration

(l=1, PPM-1, 25±50C, λ=364nm, pH=4-5, tmin=30, λ=600nm)

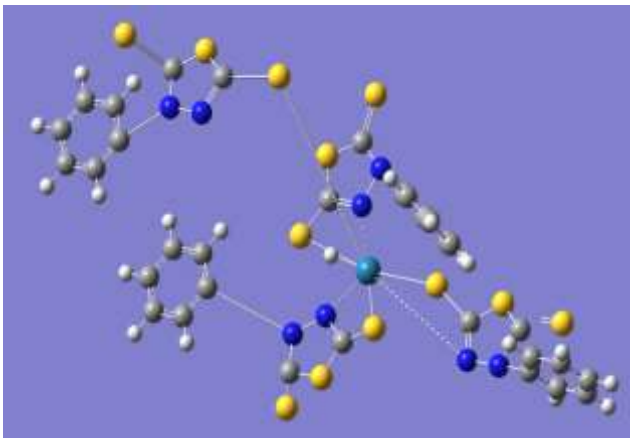
Концентрация реагента, М	А до иммобилизации	А после иммобилизации	ΔА
$1,0•10^{-4}$	0,37	0,30	0,07
$1,0•10^{-3}$	0,59	0,37	0,22
$2,0•10^{-4}$	**0,60**	**0,30**	**0,26**
$1,0•10^{-2}$	0,61	0,35	0,15
$1,0•10^{-1}$	0,8	0,65	

Complexation of the rhenium ion with the reagent bismuthol-2

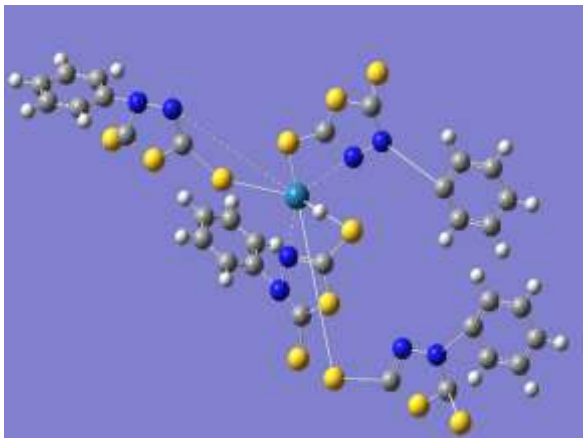
In the semi-empirical PM7 method and the Gaussianwieew method of the MOPAC 2016 program, the anion and rhenium oxide were placed close to the heterocyclic state, and when optimizing the system in the 1st case, the perrhenate anion and the potassium cation were removed from the reagent molecule. In the case of rhenium oxide, the rhenium atom approaches the nitrogen and sulfur atoms, forming a coordination bond.

1-Fig. Determination in the presence of rhenium (IV) oxide in the case of proximity to nitrogen atoms

2-Fig. Determination of the perrhenate anion in a reagent when it is close to the aromatic ring.



3-Fig. Determination of rhenium (IV) in the valence state by Gaussianwieew methods in the case of proximity to nitrogen and sulfur atoms.



4-Fig. Determination of rhenium (VII) in the valence state according to the Gaussianwieew method, close to the nitrogen, sulfur and aromatic rings

The dependence of the optical density of a complex compound on the number of elements (according to the Bouguer-Lambert-Beer law). Under the optimal conditions determined by the above experiment, 1.0 ml of a bismuth-2-reagent solution of various amounts (from 2.0 μg/ml to 50 μg/ml) of Re (VII) solution is added to 25 ml of 2.0 ml of the buffer solution (pH = 4-5 universal) was diluted with distilled water to the mark of 5.0 ml. The optical density (lmax = 600 nm, l = 1.0 cm) of the obtained complex compound was measured relative to a specific solution. The results obtained are presented in table 3

3-Table

Dependence of optical density on the amount of Re (VII)
(l=1, PPM-1, t=25±50C, λR=364nm, pH=4-5, tmin=30, λ=600nm, n=5, P=0.9)

№	C_{Re7+} 10 мкг/мл	Re^{7+} мкг/мл	Ā
1	0,1	1,0	0,015
2	0,5	5,0	0,028
3	1,0	10	0,044
4	1,5	15	0,067
5	2,0	20	0,082
6	2,5	25	0,100
7	3,0	30	0,115
8	3,5	35	0,133
9	4,0	40	0,148
10	4,5	45	0,162

The results show that compliance with the Bouguer-Lambert-Beer law was observed in the Re7+ concentration range from 3 μg to 3.5 μg. At higher concentrations, there was a deviation from the linear relationship.

Study of rhenium solutions in real objects

An aliquot of 5 ml of technical water was added to the analyzed mixture in 5 ml beakers, and 10 ml of bismuth-2 reagents and 5 ml of universal buffer solution were added. Detection was carried out by the "Additions" method. The results are presented in Table 4 below.

4-Table

Determination of rhenium ion in industrial water

Состав воды	A	Введено Мг	Найдено Мг	S	S_r
Re (7,100) Mo (0,105) Co (0,413)	0,54	10,0	10,15	0,012	0,004

The relative standard deviation did not exceed 0.0004.
This means that the water does not contain many rhenium ions.

CONCLUSIONS: A new sorption-spectroscopic method for the determination of rhenium ions was proposed, the organic reagent bismuth-2 was studied, the possibility of immobilization on sorbents of various nature. Optimal conditions for the immobilization of PPA-1, the mechanism of immobilization of bismuth-2 carriers, and optimal conditions for complexation of the studied reagent with rhenium ions in solution and in the immobilized state are proposed. An improvement in the sorption-spectroscopic properties of the reactions of complex formation of the studied metal with immobilized organic reagents by the immobilization method has been established. The sensitivity to the detection of rhenium by immobilized bismuthol-2 was studied.

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