EPRA International Journal of

RESEARCH & DEVELOPMENT
(IJRD)

AMPEROMETRIC DETERMINATION OF TUNGSTEN AND ANTIMONY WITH A SOLUTION OF NAPHTHOL DERIVATIVES

Yakhshieva Zuhra Ziyatovna

Doctor of chemical sciences, Professor, Department of Teaching Chemistry Methodology, Jizzakh State Pedagogical Institute named after Abdulla Kadiriy. Uzbekistan

Bakakhonov Anvar Akobirovich

Doctoral candidate at the Department of Teaching Chemistry Methodology, Jizzakh State Pedagogical Institute named after Abdulla Kadiriy. Uzbekistan

Kalonov Rustam Mamadierovich

Teacher of the Department of Teaching Chemistry Methodology, Jizzakh State Pedagogical Institute named after Abdulla Kadiriy. Uzbekistan.

Muyassarova Kumush Ikrom kizi

3-year student of the Department of Teaching Chemistry Methodology, Jizzakh State Pedagogical Institute named after Abdulla Kadiriy. Uzbekistan

ABSTRACT

The paper covers the conditions and the possibility of amperometric determination of tungsten and antimony in a wide range of their concentrations and, with the appropriate selection of the conditions for determination, it becomes sensitive and highly selective.

KEYWORDS: *amperometric titration, organic reagents, tungsten ions, antimony ions, selectivity.*

DISCUSSION

The rapid development and widespread application of amperometric titration methods is due to its high precision, sensitivity, selectivity and accuracy, which are reflected in technological processes, environmental objects, biological products, sanitary ware and many industrial weather conditions allows to successfully and quickly solve the tasks of analytical control (monitoring) of clinical materials. This method is widely used to determine both metal ions and various organic compounds. At present, complex multicomponent mixtures can be analyzed. The simultaneous determination of 30-40 or more elements in the sample, and at low concentrations, up to 10-5-10-8%, is achieved due to the electrochemical concentration of the component detected on the electrode surface.

The growing industrial needs for tungsten and lead, which are distinguished by their hardness, weight, and refractoriness, make the problem of analyzing environmental objects with a micro content of these metals urgent. Tungsten is less common in nature. The mass fraction of metal in the Earth's crust is $1.3 \cdot 10^{-4}\%$. In addition, it is used in the manufacture of machinery and equipment for the metalworking, construction and mining industries, in the manufacture of lighting fixtures and fixtures, in the transportation and electronics industries, in the chemical industry and other fields. A tool made of tungsten steel can withstand the intensity of the most

EPRA International Journal of Research and Development (IJRD)

intense processes in metalworking. The determination of trace amounts of antimony in environmentally important objects is associated, on the one hand, with the ever-increasing use of this metal in the semiconductor industry in the production of diodes, infrared detectors, etc., and as a component of lead alloys, increasing their hardness and mechanical strength, and , on the other hand, with its high toxicity. This in turn requires the development of a new methodology for the determination of tungsten and antimony with the best metrological characteristics and analytical parameters. Along with the expressiveness of determination and the most important requirement for the analysis of materials of various nature is accuracy, correctness and reproducibility.

This means, with all clarity, that the presented article is devoted to the development of a scientific and methodological framework for the targeted determination of microconcentrations of tungsten ions in environmental objects and food products in the field.

EXPERIMENTAL PART
Used devices and solutions.

Voltammograms were taken on polarimeters PPT-1 and PU-1 with self-records: LCD 4-003 and PDP 4-002, with a three-electrode cell. To optimize the titration conditions of W (VI), the pH of the medium was determined using a universal EV-74 ionomer and a pH / mV / TEMP Meter P 25 EcoMet pH meter. The initial solution (1 mg / ml) of W (VI) and Sb (III) was prepared by dissolving weighed portions of 2 g of their "chemically pure" salts. A sample of the sample (1.0-5.0 g) was weighed on an analytical balance, after which it was placed in a special heat-resistant glass, 4.0-5.0 ml conc. HCl and the same 5% solution of H_2O_2 were heated at a temperature no higher than 80–90 ° C for 5–10 min. The resulting solutions were diluted with bidi stilled water in a volumetric flask (250 ml). After that, an aliquot of the sample is taken and the background (buffer) is added and the total volume is adjusted to 10.0 ml and the voltammogram is taken. Measurements were performed at room temperature. Solutions with a lower concentration were prepared daily by appropriate dilution of the stock solutions with bidistillate and stored for no more than a week.

A universal Britton-Robinson buffer mixture with the required pH values was prepared by mixing 0.04 M solutions of H_3PO_4, CH_3COOH and H_3BO_3 in 0.2 M NaOH [1-4]. A 0.1 M solution of methylanabazine naphthol was prepared by dissolving (1.829 g) a weighed portion of a freshly recrystallized and well-dried preparation in 96% C_2H_5OH.

The experiments showed that 0.005-0.1 M methylanabazine naphthol solutions prepared with alcohol practically do not change their titer (provided that they are stored in the dark and in the cold) for 10-15 days. All used reagents had qualifications "special parts", "chemical parts" and "analytical grade", therefore, they were not subjected to additional purification.

RESULTS AND DISCUSSION

To obtain a voltage-dependent curve of the current, the test solution must be electrically conductive.

Precise determination of the trace amount of lead tungsten reduced in the negative potential is complicated by the presence of a large number of elements and, accordingly, prevents the detection of these ions in minerals of different ores and minerals. It is very common with them in nature to interfere with their precise quantitative determination in various alloys containing other metals.

The amperometric titration method we have developed can be very well applied in solving such problems, since this method can be used to determine up to five metals simultaneously, in one aliquot of the analyzed sample, if their reduction potentials differ from each other by 0.2 V.

Cations that are undetectable against the background of HCl should not impede the selective determination of tungsten and antimony, thereby preliminary, it will be possible to predict and eliminate the influence of some extraneous interfering cations.

Having selected the optimal conditions for obtaining the best analytical signal W (VI) and Sb (III), a priori it is impossible to unequivocally state the correctness and reproducibility of the obtained experimental data without determining them in individual solutions. In order to evaluate the accuracy of the developed amperometric determination methods for W (VI) and Sb (III) as an example in their individual solutions, it is quite possible, since the relative standard deviation (Sr) in all cases does not exceed 0.33, which indicates the absence of systematic errors. Thus, it can be assumed that the methods of amperometric titration that we developed are quite suitable for the accurate determination of the trace amounts of tungsten and antimony in model binary, ternary, and more complex mixtures.

It is known that the amperometric detection method is one of the most powerful analytical and informative methods among other well-known analytical methods, as it allows to determine the oxidation state of an element in solution, which is the most important indicator. Because this parameter often determines the level of toxicity and determines the adsorption properties of the microamphrases in the object under analysis.

For tungsten and antimony, assessing the degree of the oxidized form in solution is a special

case of assessing the lability ratio, since one of the forms of the studied ions may turn out to be electron-active in the selected potential region of the indicator electrode, and the other non-electroactive. Under certain conditions of the study, some ions are electron-active, while others are passive. The total content of the element is determined after appropriate chemical transformations and processing of the sample, and the content of the element in an inert degree of oxidation or reduction is found by the difference between the estimates of the total content and the labile form of the tungsten ion and antimony. Thus, the problem of determining the concentrations of tungsten and antimony ions is relevant and therefore, under the chosen optimal conditions, we experimentally established the electrooxidation potential at which an analytical signal ($E = -0.45$ V) for tungsten (IV) and ($E = -0$, 65 V) for antimony (III) against a background of 0.2 M HCl, 2.0 ml in volume.

CONCLUSION

In accordance with the intended purpose and the problems raised, we studied the effect of background electrolytes and buffer mixtures of different nature and concentration on the form of the electrooxidation curves of tungsten and reagent. The correlation established between the value of the analytical signal and the half-wave potential, the nature of the depolarizer and the range of the current of the electroactive substance, the temperature of the test solution, the nature and amount of the background electrolyte and the buffer mixture, the boundaries of the determined concentrations of tungsten (VI) and antimony (III) are established, measurement errors are revealed their useful analytical signal. The developed methods for the determination of tungsten (VI) and antimony (III) are quite suitable for the analysis of waters, soils, standard samples, ores, minerals, tails, concentrates, and other objects of different nature, since the correlation coefficients that we established, the lower boundaries of the determined contents of the studied ions and their detection limits (sensitivity) are at the MPC level and lower, which is very necessary in the analysis of various natural objects and industrial materials.

The obtained experimental results are processed by the rules and procedures of mathematical statistics known in the world literature.

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