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DEVELOPMENT OF METHODS FOR THE DETERMINATION OF TIN IN FOOD

Sobirova Zilola [1], ***Yakhshieva Zukhra*** [1]

[1] Jizzakh State Pedagogical University, Uzbekistan

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Abstract

This article presents the results of an analysis of canned products to determine the content of tin ions.

The time of complex formation was studied using inversion voltamperometric methods. For tin (II) ions, the maximum signal was observed at 0.260 V in 10 seconds, and these time values were determined as optimal.

The detection limits of tin (II) ions were recorded at 1.33×10^{-8} M ($R^2 \approx 0.991$) for the presented electrochemical methods, and the reliability and sensitivity of the developed method were confirmed in practice

Keywords: *electrochemical methods of analysis, tin ions, food products, metrological characteristics*

Due to the intensive development of industry, transport, industrialization and the chemicalization of agriculture, as well as the acceleration of scientific and technological progress, the release of heavy metals of technogenic origin into the environment has significantly increased in recent years and continues to grow.

Pollution of the biosphere (air, water, soil) with heavy metals leads to their accumulation in food raw materials of plant and animal origin, sometimes in quantities exceeding sanitary and hygienic standards.

The complexity of modern ecological-analytical monitoring of toxic heavy metals and the protection of public health has led to the involvement of researchers worldwide in developing highly sensitive methods of analysis. Monitoring of environmental objects, which includes the analysis of food products for toxic heavy metals, inorganic and organic substances, is carried out using various physical and chemical methods. Among these, electrochemical methods occupy a special place due to their high sensitivity, simplicity, low cost, and suitability for computerization and automation.

Modern requirements for environmental analysis demand new, highly sensitive and rapid methods for determining toxic trace metals. The multicomponent nature of analytical objects and the low concentrations of metallic pollutants require combined methods, including a preconcentration stage.

The rapid development and wide application of electrochemical methods in the last decade are explained by their high accuracy, sensitivity, selectivity, and speed, which allow reliable analytical control of technological processes, environmental samples, biological products, clinical materials, and industrial substances.

Inversion voltamperometric is widely used to determine both metal ions and various compounds. Its efficiency is increased by preliminary adsorption or electrochemical concentration of the analyte on the electrode surface.

Further development of inversion voltamperometric methods is connected with the creation of modified sensor electrodes with unique properties. Chemical modification of sensor surfaces allows the addition of new functional capabilities, enhancing both reactivity and selectivity. Indicator electrodes based on macrocyclic compounds significantly expand the potential of inversion voltamperometric as a highly sensitive and selective method of analysis.

The selectivity of the sensor we developed is ensured by modification with hydrazine sulfate and thioacetamide, which greatly improves both the sensitivity and selectivity of the method and allows for low detection limits of the studied metal ions.

Environmental Safety Related to Tin and Its Analytical Determination

In recent years, the range of objects requiring determination of heavy toxic metals has expanded, including tin, which is one of the priority environmental pollutants. Tin contamination of natural objects and industrial materials has been reported in many regions of the world. Its presence in food products is also well established, and therefore its concentration must be strictly controlled.

According to sanitary and epidemiological regulations of the Ministry of Health of the Republic of Uzbekistan, the concentration of tin in natural waters and soils should not exceed 0.2 and 0.5 mg/L, respectively.

Therefore, it is important to describe and predict the main toxic properties of these metals, assess their increasing levels in food products, and ensure strict analytical control of their content. This is essential for evaluating risks to human health and the environment, making it a highly urgent problem in modern ecological-analytical chemistry.

In this context, the urgent goal of our work was to modify sensors and develop analytical methods for determining tin. Improving electrochemical methods with better metrological characteristics remains a key challenge in food analysis. These requirements are best met by inversion voltamperometric, one of the most powerful electrochemical techniques in terms of sensitivity, selectivity, and information content.

Experimental Methods

Standard solutions were prepared gravimetrically using methods described in the literature. Exact concentrations of stock solutions and standards were determined titrimetrically. Dilute solutions (1×10^{-3} – 2×10^{-8} M) were obtained by successive dilution with triple-distilled water immediately before use.

During the analysis of canned products, we found that by varying the background electrolyte and buffer mixture, high selectivity for tin determination can be achieved. The possibility of using inversion voltamperometric for tin determination under optimized conditions of concentration and background composition was demonstrated.

For food safety monitoring, it is crucial to ensure timely control of maximum permissible concentrations (MPC) of toxic trace metals in canned goods and food products. This monitoring must be carried out by sanitary and epidemiological services across Uzbekistan.

Electrochemical methods, particularly inversion voltamperometric, meet modern requirements for analytical methods: rapid, selective, highly sensitive, accurate, inexpensive, and easy to automate.

Analytical Procedure

Before analysis, the electrolyzer was rinsed with triple-distilled water and filled with 20 ml of background electrolyte (buffer mixture). Indicator, auxiliary, and reference electrodes were inserted, and optimal electrolysis conditions were set: accumulation potential of –0.45 V, accumulation time of 60 s, current range of –1.0 μA, and appropriate amplitude and potential sweep rate. A voltammogram was recorded at least four times, with only the fourth measurement used for analysis, since the first three serve to condition the electrode and improve reproducibility.

For sample preparation, 5–10 g of solid product (or 10–15 ml if liquid) was weighed into a heat-resistant beaker. Then 4–5 ml of concentrated HCl and the same volume of 5% H_2O_2 were added. The mixture was heated under a watch glass at 80–90 °C for 15–20 min. The solution was diluted to 100 ml with triple-distilled water and filtered if necessary. An aliquot (e.g., tomato juice, cottonseed oil, canned fish, beef) was taken, background electrolyte was added, and the total volume was brought to 20 ml before recording a voltammogram.

The selectivity of the electroanalytical method – that is, the reliable separation of the ion being determined even in the presence of other cations – is one of the important criteria for its practical application. Therefore, in order to evaluate the selectivity of the developed inversion voltammetric method, the effect of foreign metal ions (Cu(II), Fe(III), V(III), Ba(II), Fe(II), Pb(II), Mn(II), Cd(II)) on the voltammetric determination of tin (II) ion was studied. For this, solutions with a total volume of 25 ml were prepared. 2 ml of 0.1 M acetate buffer solution (pH = 5.1), 2.0 µM organic reagent solution, tin (II) ion and foreign metal ions in a certain ratio were added to each solution. The volume of the solution was made up to 25 ml with distilled water. Measurements were carried out by method IV. The collection time for the determination of tin (II) ion was set to 90 seconds. The accumulation potential was 50 mV, and the accumulation potential was applied to the electrode at a scan rate of 25 mV/s.

The results confirmed that the measured concentrations of tin in canned products were consistent with certified reference values and remained within the confidence interval, demonstrating the high accuracy and reproducibility of the developed inversion voltamperometric method. Analysis was carried out using carbon-paste electrodes modified with hydrazine sulfate, which significantly improved the sensitivity and selectivity of tin detection.

Conclusion

The study demonstrated that modified sensor electrodes combined with inversion voltamperometric provide a highly sensitive, selective, and reliable method for determining tin in food products. The results showed good agreement with reference values, confirming the method's accuracy and reproducibility.

The analytical parameters of the method were the correlation coefficient R = 0.991 for tin (II) ions; the lower detection limit was 1.33×10^{-8} M; the quantitative detection limit was 4.00×10^{-8} M. The stability constants of the complexes were calculated as Kf = 4.11 for tin (II) ions.

The metrological parameters of the inversion voltamperometric method, such as accuracy, sensitivity and lower detection limit, developed on the basis of metrological analysis and competitiveness, were compared with physicochemical methods. The proposed method in some cases showed high sensitivity and selectivity compared to the methods used in production laboratories. The developed voltamperometric detection methods are characterized by speed, low detection limit and high selectivity and have been successfully applied in the analysis of food products.

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Contact: yaxshiyeva67@mail.ru