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ANALYTICAL APPLICATION OF DEVELOPED INVERSION VOLTAMPEROMETRIC METHODS IN FOOD PRODUCT ANALYSIS

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Abstract

This article presents the results of food product analysis, specifically focusing on meat products, which are essential to human life. Selective, rapid, and accurate inversion voltammetric methods have been developed for the determination of zinc in meat. The obtained results are presented and compared, showing that at a confidence level of $t_{0.95}$, no statistically significant discrepancies were found in comparison with control and certified values of the determined metal. In all cases, the relative standard deviation (Sr) did not exceed 0.33, and the total time required for the simultaneous determination of all components in the analyzed sample – without extraction-based separation or the use of additional procedures – was no more than 30–40 minutes. To obtain more complete information on the metrological characteristics of the proposed methods, the lower detection limit for zinc(II) was established at 0.001 µg in 10.0 mL of the analyzed solution

Keywords: *voltammetry, inversion voltammetry, food products, zinc ions*

The determination of heavy toxic elements is of particular importance and necessity, as the Republic of Uzbekistan, due to its geographical location, is situated in a very hot climatic region. As a result, the population tends to consume food products in quantities that significantly exceed the recommended intake levels, leading to the accumulation of substantial amounts of toxic metals in the human body. Therefore, strict analytical control (monitoring) of the content of these elements in food products is a pressing and highly relevant task for modern electroanalytical chemistry and environmental science.

As is well known, the inversion voltammetric method is used in the analysis of macro- and micro-objects to determine concentrations of individual elements or relatively small groups of them (typically 3–4 metals) in the range of 10^{-9} to 10^{-5}%. This is due to the fact that the optimal conditions for determining individual elements by the developed method vary significantly depending on the mode and potential of preconcentration, the nature and type of the indicator electrode, as well as the composition and concentration of supporting electrolytes and buffer solutions. Therefore, even in synthetic model solutions, the number of elements that can be determined using a single polarogram or voltammogram should not exceed five to six.

On the other hand, when solving analytical problems under industrial conditions or in research work – especially in the absence of the aforementioned multi-element analytical methods – it becomes necessary and important to determine the maximum number of elements from a single portion of the analyzed material. Examples of such objects include expensive high-purity materials and substances, as well as samples with limited quantities due to their uniqueness or the complexity of their manufacturing process, such as space objects, lunar soil fragments, and others. These critically important tasks become even more relevant when analyzing micro-objects, such as localized areas of crystals and films, environmental samples, biological materials, blood, food products, beverages, and so on.

It should be noted that due to the wide range of applications for the same materials and objects across various fields of science and technology, the requirements for impurity content can vary significantly. Therefore, it is both reasonable and relevant to develop multi-element analysis schemes, followed by the selection of methods that are optimal for the determination of individual elements or their specific groups. Thus, the challenges of multi-element analysis also apply to inversion voltammetry, a method that is widely used in the analysis of micro-objects.

From the theoretical foundations and results of experimental studies in the inversion voltammetry method, it follows that the simultaneous determination of 10 or more elements using a single voltammogram is practically impossible and unfeasible at the current stage of the method's development. In our opinion, a promising approach is the creation of theoretical and experimental bases for a developed method of multi-element sequential analysis, which combines the use of a single initial sample portion with optimal conditions for the final determination of individual elements or fragments and groups with similar properties.

Since zinc(II) ions possess chromophoric properties, most methods use reagents that do not contain chromophoric groups. Zinc(II) ions form very strong coordination bonds with any donor atoms. Selective reagents for zinc(II) are compounds containing phenolic OH groups and nitrogen donor atoms. We have developed photometric methods for determining zinc(II) in the form of various ligand complexes with the organic reagent Cupferon, which exhibits high sensitivity and selectivity.

For the successful resolution of nutritional problems, it is important to timely consider and evaluate the potential use of new sources of dietary proteins, including those previously neglected. However, even more important and relevant is the determination of toxic impurities in food industry products, since the content of carcinogens, mutagens, and toxic trace elements is strictly regulated and must not exceed the maximum permissible concentrations.

Existing standard atomic absorption and photometric methods for zinc determination are labor-intensive and not rapid; in many cases, they require preliminary separation of the target metals from the main matrix before analysis. Therefore, the development of electrochemical methods for determining toxic metals in food products – offering improved metrological characteristics and economic advantages – is a pressing task in modern analytical chemistry and public health protection.

The developed methods were applied for the determination of trace amounts of zinc(II) with the possibility of using the organic reagent Cupferon ($C_6H_5N(NO)ONH_4$) in the analysis of meat products.

Experimental Section

Sampling and Preliminary Preparation of Analyzed Samples

The high sensitivity of inversion voltammetry necessitates the exclusion of all possible sample contaminations, which may originate from reagents, laboratory glassware, or any apparatus contacting the samples. It has been established that the accuracy of analytical results largely depends on errors in sampling and sample preparation; therefore, these operations require the utmost and careful attention from researchers. As a rule, polyethylene bottles should be pre-treated with various cleaning agents, since even perfectly clean polyethylene surfaces tend to adsorb metals.

Selection of Determination Stage Conditions

When determining metal contents by the inversion voltammetric method, a sample solution of 25–30 mL is introduced into the cell, and several drops (up to 10) of 0.02 M mercury(II) nitrate solution of analytical grade or high purity are added. A pre-electrolysis of the analyzed solution is carried out for 30–90 seconds (depending on the nature and concentration of the components) at –1.0 V using a magnetic stirrer (1500 rpm). After the pre-electrolysis time has elapsed, the solution is allowed to rest for 15–30 seconds, and the voltammogram (peak) is recorded in the potential range from –0.9 to –0.1 V. Then, the electrode is held at –0.1 V for 45 seconds to fully dissolve all concentrated components from the electrode. Based on the peak heights recorded, the working sensitivity of the instrument and the necessary pre-electrolysis time are selected. The analysis procedure is then repeated under the chosen conditions until reproducible peaks and accurate results for zinc ion determination are obtained. The standard addition should be chosen such that the concentration of the determined metal in the sample solution approximately doubles.

Since hydrogen ions participate in the electrode reaction, the half-wave potential ($E_{1/2}$) of both waves depends on the background pH according to the equation:

$$E_{1/2} = E^0 - 0{,}059\frac{m}{n}pH$$

where:

E^0– standard electrode potential;

m– number of protons;

n– number of electrons involved in the electrode reaction.

As the pH of the medium increases, both waves shift toward more negative potentials.

The number of protons participating in the electrode reaction can be found from the slope (tangent of the angle) of the $E_{1/2}$ vs. pH dependence using the formula above.

$$m = \frac{n}{0{,}0591}tg\alpha$$

The accuracy of trace metal determination by inversion voltammetry using the method described above is very high.

Results and Discussion

Solutions and Reagents

A standard zinc(II) solution (1 mg/mL) was prepared by dissolving a weighed amount of Mohr's salt in water with the addition of 10 mL of concentrated H_2SO_4 to acidify the solution. Solutions with lower zinc(II) concentrations were prepared by successive dilution of the standard solution. Cupferon ($C_6H_5N(NO)ONH_4$) was dissolved in water to prepare a 0.1% solution in a volumetric flask.

Cupferon reagent was purified by reprecipitation from ethanol solutions through the addition of water, followed by distillation. The ionic strength of the solutions ($\mu = 0.1$) was maintained constant by adding a calculated amount of KNO_3. An acetate buffer solution was used to achieve the required acidity of the solutions. All reagents used were of analytical grade (reagent grade or chemically pure). Preliminary experiments showed that Cupferon ($C_6H_5N(NO)ONH_4$) forms a colored complex with zinc(II), which is well soluble in non-polar organic solvents.

Effect of pH.

In the study of solution acidity, it was found that at pH = 1, an acidic medium is formed; at pH = 3–6, the solution remains clear with a red color; and at pH = 8–10, a dark brown precipitate forms.

Analysis Procedure Algorithm

1. A portion of the crushed sample (5–6 g) is weighed in a porcelain crucible or beaker with an accuracy of ±0.01 g and placed in a muffle furnace for ashing. The sample is first gently heated for one hour, after which a few drops of nitric acid (1:1) are added, and ashing is continued at a temperature not exceeding 550 °C until a light-colored ash of constant mass is obtained. The ash is then dissolved in 5–10 mL of hydrochloric acid (1:1), and the solution is quantitatively transferred to a 50 mL volumetric flask and brought to volume with 0.1 M HCl. 20.0 mL of the prepared sample solution (aliquot) is introduced into the voltammetric cell, and the analysis is then carried out.

2. A 3 g portion of beef was placed in a porcelain crucible and dried to remove moisture. The sample was then ashed – first over an open flame, followed by ashing in a muffle furnace at 500 °C. To accelerate the ashing process, the crucible was periodically removed from

the furnace, cooled, and the contents moistened with a 3% hydrogen peroxide (H_2O_2) solution. The sample was then dried on a water bath and in a drying oven, and further ignited until a uniform brown-colored mass was obtained.

The cooled ash was treated twice with 2 mL portions of 2 M hydrochloric acid (HCl), heated on a water bath, and stirred with a glass rod. The resulting solution was filtered into a beaker and heated to 60 °C in a water bath. Then, 5 mL of a 1% solution of ammonium alum, 5 g of ammonium chloride (NH_4Cl), and concentrated ammonia solution (NH_4OH) were added. The solution was maintained at 60 °C to ensure coagulation of the iron and aluminum hydroxide precipitate.

The beaker and precipitate were washed five times with hot water containing a few drops of NH_4OH. The precipitate was then dissolved in 10 mL of 2 M HCl, filtered into a 100 mL volumetric flask, and the volume was brought to the mark with distilled water. An aliquot of the resulting solution was taken, and the zinc(II) content was determined using Cupferon.

In all cases, the relative standard deviation (Sr) did not exceed 0.33, and the total time required for the simultaneous determination of all components in the analyzed sample – without extraction-based separation or the use of additional techniques – was no more than 30–40 minutes. To obtain more comprehensive information on the metrological characteristics of the proposed methods, we determined the lower detection limit of zinc(II) based on methods reported in the literature, which was found to be 0.001 µg in 10.0 mL of the analyzed solution.

The developed inversion voltammetric methods are not only comparable to GOST-certified and officially validated methods in terms of accuracy and selectivity, but in some cases even surpass them in certain metrological characteristics and analytical parameters.

Conclusions

The developed inversion voltammetric methods for the determination of zinc demonstrate high accuracy and reproducibility. The results obtained once again confirm the validity, necessity, and importance of the analyses performed on meat samples.

For the purpose of metrological evaluation of the proposed metal determination methods, as well as to assess their competitiveness and potential inclusion among the arsenal of well-established and widely used electroanalytical techniques, we compared the results obtained using the developed inversion voltammetric methods with those acquired through atomic absorption and GOST-certified methods. The comparison showed that our proposed inversion voltammetric techniques for zinc determination are comparable in terms of speed, accuracy, and reproducibility, as well as in their lower detection limits. Moreover, in certain metrological parameters, they even surpass the reference methods – namely, atomic absorption and GOST (photometric) techniques.

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