Analysis of the results of control of toxic elements in food

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Abstract. The article discusses the experimental substantiation of the use of the stripping voltammetry method in order to organize and control the process of measuring toxic elements in food products, the quantitative criteria of which are direct indicators of safety. Comparative results are given for the example of dairy and milk-containing products and criteria for the amount of toxic elements in products and their norms, in particular. The analysis of the monitoring points related to the work logs for the control of these elements was carried out, the processes of the quantitative chemical analysis of the collected samples were studied. The sample data was tracked by the specified method of IV measurements on a «ТА-1» voltammetric analyzer. Also, the analysis of regulatory and various technical documentation for the level of pollution by toxic elements of our environment was carried out and the possibilities of ways of excretion and their entry into products were considered. Intralaboratory control instruments were used in the form of control charts; according to the results of the construction of which, the results of correctness and accuracy were determined and interpreted. Managing these safety processes is one of the stable analysis of evaluating the internal control of the parameters of results and obtaining close-reliable conclusions. As a result of the research, indicators of the correctness and accuracy of the data were derived.

1. Introduction
Contamination of various food products with toxic foreign substances is part of the globalization of problematic environmental pollution, therefore, at this modern time, a very important aspect of obtaining safe quality is the determination of trace amounts of anti-positive substances in food, in particular the dairy industry, and the selection of means to reduce their content. Lead and cadmium are metals that exhibit strong toxicological properties at the lowest concentrations and do not perform any useful function. Such elements are not vital, beneficial, but even in small doses they lead to disruption of the normal metabolic functions of the whole organism.

The food industry, like other branches of industrial production, requires a complex analytical complex, equipped in the technological process with devices that have sufficient sensitivity and selectivity to the determined substances, in particular to the above.

In the analysis of food products for the content of toxic metals, the method of stripping voltammetry is widely used.

To improve the quality of laboratory work during production, an internal quality control system for its results is required. Control charts are used to control the stability of the measurement process and to identify situations of exit of this process from a statistically controlled state [1–3].

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2. Materials and methods
Physicochemical, mathematical and empirical methods were used: controlled quantitative chemical analysis of the content of cadmium and lead ions in the samples of dairy products using the method of IV measurements. The dependence of the current on the potential - voltammogram - for stripping voltammetry has the shape of a peak. The analyzer voltammetric type «TA-1» is a complex that consists of the actual voltammetric analyzer «TA-1/1» and an IBM-compatible personal computer with the installed software package «TA-1». IV is based on the preliminary electrochemical accumulation of the determined component on the surface or in the volume of the indicator electrode with the subsequent registration of the voltammogram reflecting the electrochemical reaction of the accumulation product.

The method of voltammetric analysis is based on measuring the current flowing in the circuit of an electrochemical cell, depending on the polarizing voltage applied to its electrodes. To determine the content of toxic elements, a voltammetric analyzer «TA-1» is used. IV measurements of the content of cadmium and lead in product samples are carried out using a mercury film electrode.

Mineralization of test samples is also carried out [4–6].

3. Results and discussion
On-line control of intra-laboratory precision is carried out using working samples by comparing the result of the control procedure, which is equal to the discrepancy between the two results of measurements of the component content in the same sample, obtained under different conditions [7–9].

Table 1. The values of the standards for operational control of repeatability, intra-laboratory precision and error for each analysis result

| The element being defined | The operational control standard, drel, % (for two results of parallel determinations) | In-laboratory monitoring standard precision, drel, % (for two results of parallel determinations) | Intra-laboratory control standard for error, Krel, % (P=90) |
|--------------------------|--------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------|------------------------------------------------------------------|
| Cadmium                  | 35                                                                                   | 42                                                                                        | 27                                                              |
| Lead                     | 46                                                                                   | 55                                                                                        | 34                                                              |

The operational control scheme of the analysis procedure provides for:
- choice of control procedure;
- implementation of the control procedure;
- calculation of the result of the control procedure;
- calculation of the control standard;
- implementation of the decisive control rule.

The control procedure was implemented using the addition method.

To organize the stability control and control the analysis results using control charts, the following is carried out:
1 The required number of control procedures for a reliable assessment of each of the controlled quality indicators of the analysis results, while the assessment of the characteristics of the controlled quality indicator is considered reliable if the uncertainty of this assessment does not exceed 0,33;
2 The time range for obtaining the required number of control procedures, established taking into account: the duration of the analysis procedure and the relationship between the number of control procedures and the number of working samples analyzed for a certain period of time.

Since the concentration of cadmium ions in a working sample of milk and dairy products is less than 0.05 mg/kg, operational control is carried out in a working sample with an additive, and the concentration of lead is determined in a working sample and in a working sample with an additive. Operational control of repeatability in determining the concentration of lead ions is carried out in working samples and working samples with an additive.
Control of intra-laboratory precision and analysis procedure in working samples with additive. To control the repeatability, a control chart is used, on which the results of control procedures are plotted - discrepancies in the results of control determinations. To construct a control chart, the values of the center line, warning limit and action limit are calculated and plotted on the control chart [10, 11].

Shewhart's control charts for the repeatability of analysis results for determining the content of cadmium and lead ions are shown in Figures 1, 2.

When monitoring the repeatability of the analysis results to determine the content of cadmium and lead ions, no overshooting of the control limits was observed, the process is considered stable.

**Figure 1.** Shewhart’s control chart for control of the repeatability of the analysis results for the determination of the content of cadmium ions

**Figure 2.** Shewhart’s control chart for repeatability control of lead ion analysis results
Shewhart's control charts of the in-laboratory precision of the analysis results for the determination of the content of cadmium and lead ions are presented in Figures 3, 4.

**Figure 3.** Shewhart’s control chart for monitoring the intralaboratory precision of the analysis results for the determination of the content of cadmium ions

![Shewhart's control chart for cadmium ions](image)

**Figure 4.** Shewhart’s control chart for monitoring the intra-laboratory precision of the analysis results for the determination of the content of lead ions

![Shewhart's control chart for lead ions](image)

The errors in the analysis results for determining the content of cadmium and lead ions are presented in Figures 5, 6.
Based on the results of the control procedures, calculated in terms of the measured contents and obtained within the planned time range, it is possible to control the accuracy and accuracy of the analysis results.

The estimated value of the intralaboratory precision indicator for determining the content of cadmium ions is 0.0043, and that of lead ions is 0.0047. The index of intralaboratory precision of the analysis results for the determination of the content of cadmium ions is 0.0219, lead is 0.0489, which are lower indicators. With the converted value of the indicators into a percentage, we get 7% of cadmium, 4% of lead.

4. Conclusion
The management of food safety, in particular from dairy products, can be achieved through a concentrated interaction of measurements of indicators of toxic components and the construction of
control charts based on the identified data. Thus, you can achieve a bar with a statically controlled result. These results were obtained and analyzed in a continuous research system. The error of the obtained results does not exceed 10%.

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