Evaluation of the stability of the results of studies of beef for lead content using the additive method

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Abstract. The obtained monitoring results indicate that in the range of less than 0.01 mg of lead per 1 kg of beef prevails (51.9%) over other ranges. The least registered sample results (3.4%) with the range of obtained data - 0.1-1.0 mg / kg. The primary and important task remains to ensure the control of the quantitative content of lead, the introduction and application of fast and reliable methods of their research. The analysis of the obtained data results shows that all the results on the study of beef for the content of lead, obtained by stripping voltammetry and atomic absorption spectrometry methods for assessing the precision and operational control of the error using the additive method, are satisfactory. By implementing the methods of stripping voltammetry and atomic absorption spectrometry, the best precision of research results in the testing laboratory is achieved, both under repeatability conditions and under conditions of intermediate precision.

1. Introduction

In the foreign and domestic literature, the issue of the effect of nutrition on human health has been widely studied [1-6], information on contaminants, their types and effects on the body as a whole, routes of intake of toxicants and preventive measures to reduce the level of food contamination with toxic substances is presented in detail, as well as technological methods of reducing the residual amounts of contaminants in food products [7-12].

Ensuring testing of food products and food raw materials at the highest level of compliance with modern international quality standards is a component that determines the competitiveness of testing laboratory centers [11-18].

The development of effective algorithms for optimizing the laboratory equipment base, as well as the search for a solution that will allow laboratories to provide reliable test results at minimal cost in the shortest possible time, is an important and urgent task [17-21]. The relevance of the problem under
consideration is due to the need for hardware to cover a wide range in the determination of toxic elements. The primary and important task is to ensure the control of the quantitative content of toxic elements, the introduction and application of fast and reliable methods for their analysis [20-26]. Implementation of quality management systems is of great importance [27, 28].

According to the requirements of the Technical Regulations of the Customs Union 021/2011 (TR CU 021) “On food safety”, as amended on August 8, 2019, lead is mandatory for control in food (figure 1). Lead belongs to the highest hazard group toxicants.

**Figure 1.** Permissible levels of lead in meat and meat products according to the requirements of technical regulations, mg/kg, not more.

### 2. Material and methods

The development and approbation in the laboratory of modified test methods of the conformity confirmation procedure is an important and urgent task.

Carrying out comparative tests by stripping voltammetry and atomic absorption spectrometry in order to ensure the control of lead in beef will allow us to optimize test equipment with a prerequisite - preserving the metrological characteristics of the accuracy of the obtained research results.

The object of research is beef samples.

Equipment for research of lead content in food raw materials and food products is shown in figure 2. The methodological basis is the standards for research methods, test methods, operation manuals and instructions for the use of the specified equipment.

**Figure 2.** Research instrument base.
In the course of the research, the standard deviation (RMS) of the intralaboratory repeatability of \( S_r \) and the intermediate precision \( S_R \) of atomic absorption analysis and stripping voltammetry were evaluated.

The characteristic of a standard sample of the composition of a solution of lead ions is shown in figure 3.

![Figure 3. Characterization of a standard sample of the composition of a solution of lead ions.](image)

The main document of the research procedure is the test facility quality manual.

The control by the method of additions during the implementation of various methods in this work was carried out according to the approved research scheme.

During the operational control of the analysis procedure using the control procedure to control the error using the method of additions, the control means were working samples of a stable composition and the same samples with a known addition of lead.

Under the conditions of in-laboratory precision, samples were analyzed with and without added lead.

3. Results and discussion

Atomic absorption spectrometry is widely used in the determination of heavy metals (toxic elements) in various branches of science and industry.

It should be noted that the assortment of product groups for which lead standards have been established has significantly expanded in comparison with GB 2762-2005. In the national standard GB 2762-2005, only the maximum levels of lead for the following types of products were presented: cereals, flakes, cereals; legumes; potato products; meat; edible animal by-products; fish; fruits; small fruits, berries, grapes; vegetables (excluding stems, leaves and edible mushrooms); stem vegetables; vegetables - leafy; fresh milk; baby food (using milk as raw material, materials measured by milk liquid, milk diluted from powder); fresh eggs; wine; fruit juice; tea.

The national standard of the People's Republic of China GB 2762-2012, in comparison with the requirements of TR CU 021, does not standardize the maximum levels of lead in dietary supplements to food, food for pregnant and lactating women.

The maximum lead levels according to the national standard of the People's Republic of China GB 2762-2012 meet the requirements of the Codex Alimentarius standards, unless there is no Codex standard. The requirements of TR CU 021 correspond, and in some cases are lower than the requirements of the Codex Alimentarius standards, i.e. Lead MPCs for some types of products were found to be stricter in the Codex Alimentarius standards.

In comparison with the MPC for lead in food products regulated by the requirements of TR CU 021, the maximum levels of lead in accordance with the national standard of the People's Republic of China GB 2762-2012 correspond or in some cases are higher than the requirements of TR CU 021, i.e. The maximum permissible concentration of lead for some categories of products was stricter in the national standard of the People's Republic of China GB 2762-2012.

In the conditions of market relations at any enterprise, including in testing laboratories, the relevance of quality management is determined by its focus on providing such a level of quality of services that
can fully satisfy all consumer needs. The high quality of the services provided is the most significant component that determines the competitiveness of test centers.

The advantages of the voltammetric test method are shown in Figure 4.

![Advantages of stripping voltammetry](image)

**Figure 4.** Advantages of stripping voltammetry.

The advantages of atomic absorption spectrometry are shown in figure 5.

![Advantages of atomic absorption spectrometry](image)

**Figure 5.** Advantages of atomic absorption spectrometry

3.1. Intermediate precision control
Under repeatability and intermediate precision conditions, five average measurements were obtained for the beef sample.

The critical range factor \( f(n) \) for the five results is 3.9. The repeatability standard deviation \( S_r \) is presented in table 1.

For the Kvant-2AT spectrometer:

\[
(CR0.95(5), \%) = f(5) \times S_r (Pb) = 3.9 \times 18 = 70.2 \%
\]

For the analyzer "TA-4":

\[
(CR0.95(5), \%) = f(5) \times S_r (Pb) = 3.9 \times 13 = 50.7 \%
\]
Table 1. Results of studies of beef for lead content (standardized level of permissible concentration 0.5 mg/kg).

| №  | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT |
|----|------|-----------|------|-----------|------|-----------|
|    |      |           |      |           |      |           |
| 1  | 0.27 | 0.32      | 0.48 | 0.57      | 0.59 | 0.59      |
| 2  | 0.24 | 0.35      | 0.52 | 0.53      | 0.52 | 0.63      |
| 3  | 0.32 | 0.27      | 0.49 | 0.49      | 0.61 | 0.61      |
| 4  | 0.26 | 0.37      | 0.55 | 0.54      | 0.63 | 0.57      |
| 5  | 0.29 | 0.34      | 0.53 | 0.56      | 0.54 | 0.51      |

Assessing the precision of results $(X_{max}-X_{min}) \leq CR_{0.95}(5)$

|    |      |           |      |           |      |           |
|----|------|-----------|------|-----------|------|-----------|
|    | 0.08 | 0.14      | 0.07 | 0.26      | 0.08 | 0.38      |
|    | 0.11 | 0.29      | 0.12 | 0.41      |

The results of operational control of the research procedure using the control procedure for controlling the error using the addition method are presented in Table 2.

Table 2. Study of samples for lead content.

| №  | additive at the stage of sample preparation | additive at measurement stage |
|----|---------------------------------------------|-------------------------------|
|    | K | K | K | K | K | K | K | K |
|    | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT |
| 1  | -0.0092 | 0.02045 | -0.011 | 0.02896 | -0.0012 | 0.021246 | -0.0018 | 0.030215 |
| 2  | -0.0041 | 0.00414 | -0.0051 | 0.00581 | -0.0037 | 0.004179 | -0.0035 | 0.006041 |
| 3  | -0.0068 | 0.00697 | -0.0072 | 0.010134 | -0.0036 | 0.007272 | -0.005  | 0.01042  |
| 4  | -0.062 | 0.06371 | -0.092 | 0.09543 | 0.002  | 0.069944 | -0.048  | 0.10116  |

Analyzing the results obtained, we state that the condition $(X_{max}-X_{min}) \leq CR_{0.95}(5)$ performed for the measurement results.

3.2. Evaluation of the precision of laboratory test results obtained from the implementation of different research methods

The results of the operational control of the laboratory test procedure using the control procedure to control the error using the addition method are presented in Table 3.

Table 3. Research results.

| TA-4 | Kvant-2AT | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT |
|------|-----------|------|-----------|------|-----------|
|      | additive at the stage of sample preparation | additive at measurement stage |
|      | K | K | K | K | K | K | K | K |
|      | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT | TA-4 | Kvant-2AT |
| 5(5) | 0.276 | 0.33 | 0.514 | 0.538 | 0.578 | 0.582 |
| $X_{av}$ | 0.303 | 0.526 | 0.58 |

Evaluation of the precision of results obtained by different methods

$8.9 \% < 17 \%$  
$2.3 \% < 17 \%$  
$0.35 \% < 17 \%$

| K | K | K | K | K | K |
|---|---|---|---|---|---|
| -0.077 | 0.08668383 | -0.023 | 0.09344501 |

The analysis of the presented results in Table 3 shows that all the results of investigations of samples for the content of lead are satisfactory (according to the assessment of precision and operational control of the error using the method of additions).
3.3. Research results monitoring
We monitored the results of research on the content of lead in beef from January 2019 to October 2020. We assessed the distribution of research results depending on the concentration of lead in beef (figure 4).

![Figure 6. Distribution of test results depending on the concentration (mg/kg) of lead in beef samples, %.

The obtained monitoring results indicate that in the range of less than 0.01 mg of lead per 1 kg of beef prevails (51.9%) over other ranges. The least registered sample results (3.4%) with the range of obtained data - 0.1-1.0 mg/kg.

4. Conclusion
The analysis of the obtained data results shows that all the results on the study of the content of lead in beef on the assessment of precision and operational control of the error using the method of additions are satisfactory.

The implementation of the methods of stripping voltammetry and atomic absorption spectrometry achieves the best precision of the results of lead research in the testing laboratory, both under conditions of intermediate precision and under conditions of repeatability.

Beef should be monitored for lead on an ongoing basis.

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