Evaluation of the use of the PLP-01M microwave laboratory system using working samples to control the accuracy of the results of examining product samples for lead content

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Abstract. In order to compare the results obtained when working on the Kvant-2AT atomic absorption spectrometer, taking into account the use of the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929 "Raw materials and food products. Sample preparation. Mineralization to determine the content of toxic elements", on the working samples, the products were selected that most fully covered the range of results obtained during the research. Within 30 working days, a set of research results and analysis of data obtained during operation on the Kvant-2AT atomic absorption spectrometer, taking into account the use of the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929, were carried out. Average values obtained under conditions repeatability were recorded in the table. In order to assess the accuracy of the obtained values, the analytic, lead, was added. The implementation of sample preparation methods taking into account the microwave decomposition of the sample in the case of using the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929-94 achieves the precision of the analysis results both under conditions of repeatability and under conditions of intermediate precision.

1. Introduction
Research into the influence of heavy metals in ecosystems and technological methods for reducing the residual amounts of contaminants in products is one of the topical issues [1-8].

Ensuring product testing for the content of xenobiotics requires constant improvement [9-18]. Acceptable levels of lead in products are shown in figure 1.
Figure 1. Permissible levels of lead in fishery products according to the requirements of technical regulations, mg/kg, not more.

The toxicity of lead is shown in figure 2.

Figure 2. Lead toxicity.
One of the important tasks of testing centers is to ensure the reliability of tests at minimal cost [19-23]. The relevance of the problem under consideration is confirmed by numerous studies of scientists from different countries [24-29].

In the PLP-01M microwave laboratory system (figure 3), a fundamentally new method of sample preparation is used. The decomposition was carried out in a closed system - sealed fluoroplastic vessels under the influence of high temperature, pressure and microwave field. The microwave field in the working chamber of the furnace was created by a special generator-magnetron.

The advantages of microwave decomposition of samples using PLP-01M over classical methods of sample preparation are undeniable.

2. Material and methods
This paper considers and analyzes the results of examining samples for cadmium content by determining them on a Kvant-2AT atomic absorption spectrometer, taking into account the use of the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929 “Raw materials and food products. Sample preparation. Mineralization to determine the content of toxic elements”.

Sample mineralization by microwave decomposition using a PLP-01M microwave laboratory system was carried out according to the general scheme in accordance with the instructions for a microwave laboratory oven from the Ural-Hephaestus TP.

In order to cover the entire range of results obtained during research and thereby simulate the obtaining of values of various concentrations in the analysis of working samples of food products, intervals were identified and control samples (OK Pb/OK Cd) with a certified value of the determined toxic element - cadmium - were selected for these intervals (table 1).

| Intervals, mg/dm³ | OK Pb, mg/dm³ |
|------------------|--------------|
| 0.01–0.03        | 0.015        |
| 0.03–0.05        | 0.04         |
| 0.05–0.10        | 0.085        |
| 0.1–1.0          | 0.55         |

For research purposes, we used standard samples of the composition of a solution of lead ions, shown in figure 4.
3. Results and discussion

3.1. Checking the accuracy of the results

In order to compare the results obtained when working on the Kvant-2AT atomic absorption spectrometer, taking into account the use of the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929 "Raw materials and food products. Sample preparation. Mineralization to determine the content of toxic elements ", on the working samples, the products were selected that most fully cover the range of results obtained during the research.

Within 30 working days, a set of research results and analysis of data obtained during operation on the Kvant-2AT atomic absorption spectrometer, taking into account the use of the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929, were carried out. Average values obtained under conditions repeatability were recorded in the table. In order to assess the accuracy of the obtained values, the analyte, lead, was added.

As a result, under conditions of intermediate precision, five average values were obtained.

Sample preparation of the selected food samples was carried out in accordance with GOST 26929 and in accordance with the instructions of the PLP-01M microwave laboratory oven from the Ural-Gefest TP, the volume of the sample taken for analysis is shown in table 2.

| Table 2. Sample weight in g.                        |
|---------------------------------------------------|
| Sample preparation according to with GOST 26929   |
| Microwave decomposition on PLP-01M                |
| 10                                                |
| 2                                                 |

The research results are presented in table 3.

| Table 3. Results of Lead Testing of Fish Samples. |
|-------------------------------------------------|
| Day | GOST | PLP-01M | GOST | PLP-01M |
|-----|------|---------|------|---------|
|     |      |         |      |         |
| 1   | 0.069 | 0.072  | 0.131 | 0.125  |
| 2   | 0.071 | 0.072  | 0.121 | 0.123  |
| 3   | 0.066 | 0.077  | 0.112 | 0.123  |
| 4   | 0.064 | 0.071  | 0.102 | 0.118  |
| 5   | 0.073 | 0.075  | 0.113 | 0.125  |
| Xcp | 0.0686| 0.0734 | 0.1158| 0.1228 |

Assessing the precision of results \((X_{max}−X_{min}) ≤ CR_{0.95}(5)\)

\[
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0.009<0.024 0.007<0.033 0.029<0.059 0.029<0.078
\]

Analyzing the results obtained during the experiment, we assert that the condition \((X_{max}−X_{min}) ≤ CR_{0.95}(5)\) is fulfilled.

3.2. Monitoring the stability of analysis results using the addition method

When carrying out operational control of the analysis procedure using the control procedure for error control (SSC) using the additive method, the control means were working samples of a stable
composition and the same samples with a known addition of the analyte.

The control by the addition method during the implementation of various types of sample preparation in this work was carried out according to the following scheme.

The sample was taken in double size; the analyzed sample was divided into two parts. One part remained unchanged, the second was supplemented with the determined element $C_d$. The addition was carried out at the stage of sample preparation.

Under the conditions of intralaboratory precision, the analysis of samples was carried out with the added additive of the determined element and without the additive.

In accordance with the analysis methods, taking into account various types of sample preparation, the results of control measurements of the concentration of the determined element in the averaged working sample - $X_{(n)}$ and in the averaged working sample with a known addition of the determined element – $X_{(n)+d}$.

As the results of control measurements of the concentration of the determined element in the sample and in the sample with the additive, the arithmetic means of two results of a single analysis were used, the discrepancy between which does not exceed the repeatability limit.

The result of the control procedure $K_k$ and the control standard $K$ were calculated according to the approved methods. The fulfillment of the comparison condition was also checked.

The results of the operational control of the analysis procedure using the control procedure to control the error using the addition method are summarized in table 4.

| Table 4. Results of operational control of the analysis procedure using the method of additions for lead content. |
| --- |
| GOST 26929 | PLP-01M |
| $K_k$ | $K$ | $K_k$ | $K$ |
| -0.0028 | 0.02035 | -0.0006 | 0.02163133 |
| -0.0038 | 0.00428 | -0.0038 | 0.0050202 |
| -0.0048 | 0.00829 | -0.0044 | 0.00913438 |
| -0.012 | 0.07798 | -0.008 | 0.08227281 |

The research results presented in table 4 constitutes that the condition $|K_k| \leq K$ is fulfilled for all measurement results. Evaluation of the precision of the analysis results obtained taking into account the use of two different types of sample preparation.

The analysis procedure was considered satisfactory if condition 3.12 was met.

The results of evaluating the precision of the results obtained taking into account the use of different types of sample preparation are presented in table 5.

| Table 5. Lead analysis of food samples. |
| --- |
| № | GOST | PLP-01M |
| --- | --- | --- |
| $X_{(5)}$ | 0.0686 | 0.0734 |
| $X_{av}$ | 0.071 | 0.1158 |

Evaluation of the precision of the results obtained by different sample preparations

$K_k$ | $K$ |
| --- | --- |
| -0.0017 | 0.01982478 |
| -0.0038 | 0.00438914 |
The analysis of the data obtained concludes that the results for the assessment of precision and operational control of the error using the method of additions are satisfactory.

4. Conclusion
The results of analyzes carried out under conditions of repeatability and intermediate precision are considered satisfactory. The results for the assessment of precision and operational control of the error using the method of additions are satisfactory.

The implementation of sample preparation methods taking into account the microwave decomposition of the sample in the case of using the PLP-01M microwave laboratory system and during sample preparation in accordance with GOST 26929-94 achieves the precision of the analysis results both under conditions of repeatability and under conditions of intermediate precision.

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