Application of the PLP-01M microwave laboratory system using control samples to assess the accuracy of the results of studies of cadmium content

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Abstract: 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". The PLP-01M microwave laboratory system uses a fundamentally new method of sample preparation. 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. Based on the results of the assessment of the operational control of the measurement procedure using samples for cadmium control during the implementation of the PLP-01M microwave laboratory system in the laboratory, the analysis procedure was recognized as satisfactory.

1. Introduction

Research on 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].
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].

The PLP-01M microwave laboratory system uses a fundamentally new method of sample preparation. 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.

Advantages of microwave decomposition of samples using PLP-01M over classical methods of sample preparation:

- an increase in the rate of acid decomposition of inorganic and organic samples by 10-100 times;
- obtaining a sample of improved quality;
- reproducibility of the research process;
- reduction of consumption of acids used for sample decomposition;
- realization of the ability to retain volatile components in the sample due to the use of closed vessels;
- automation of the sample decomposition process thanks to a microprocessor control unit.

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."

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

Acid mineralization using the PLP-01M microwave laboratory system was carried out according to the following scheme:

- a sample weighing 2g (0.5-2g, depending on the type of sample) was placed in a sealed fluoroplastic vessel;
- the addition of 8 cm³ of concentrated nitric acid was carried out, followed by sealing the vessel;
- in parallel, mineralization of the reagents added to the sample was carried out to control their purity (control solution);
- a carousel with fluoroplastic vessels was placed in a PLP-01M microwave laboratory system, the sample was decomposed in accordance with the program from the Ural-Gefes TP for this product group;
- upon completion of decomposition, the vessels are cooled and opened in accordance with the instructions of the microwave laboratory oven from the Ural-Hephaestus TP.

Mineralization of samples based on an inorganic matrix was carried out in one stage (OC analysis). During the mineralization of samples with an organic matrix, the process was carried out with a gradual increase in pressure (maximum working pressure 1300 kPa) and the second stage of mineralization. For this, 2 cm³ of hydrogen peroxide was added to the cooled sample, the vessel was hermetically sealed and placed in a PLP-01M microwave laboratory system, and re-mineralization was carried out in accordance with the program from the Ural-Hephaestus TP.

The resulting solution was filtered through a paper filter, transferred quantitatively into a 25 cm³ volumetric flask, the volume was brought to the mark with a background solution of nitric acid, and thoroughly mixed.
3. Results and discussion

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 (OKp/OKCd) with a certified value of the determined toxic element - cadmium - were selected for these intervals (table 1).

| intervals, mg/dm³ | OKCd, mg/dm³ |
|-------------------|-------------|
| 0.001–0.005       | 0.0035      |
| 0.005–0.010       | 0.0075      |
| 0.01–0.05         | 0.03        |

Table 1. Intervals and control samples.

For research purposes, we used standard samples of the composition of a solution of cadmium ions, shown in figure 1.

![Characteristics of a standard sample composition of the solution of cadmium ions.](image)

Independent measurement results were obtained by the same method on identical test objects, in the same laboratory, by one operator, using the same equipment - an atomic absorption spectrometer "Kvant-2AT", within a short period of time, i.e. the repeatability conditions were met.

In order to check the reproducibility of the results, the studies were carried out by different operators using the same equipment on different days, i.e. the conditions of intermediate (intralaboratory) precision were met.

The accumulation and processing of data was carried out within three days. In one day, sample preparation and analysis of two parallels were carried out, the arithmetic mean for each parallel was calculated and the average research result for the day was found.

All the obtained values were entered in tables 2-4 in the corresponding columns.

| COKCd=0.0035 mg/dm³ | X₁    | X₂    | Xₐr   | Xₐ/day |
|---------------------|-------|-------|-------|--------|
|                      | PLP-01M |       |       |        |
| I day                | 0.003116 | 0.003305 | 0.003067 | 0.003303 |
|                      | 0.003018 | 0.003112 | 0.003095 | 0.003095 |
| II day               | 0.003321 | 0.003274 | 0.003309 | 0.003106 |
|                      | 0.003296 | 0.003254 | 0.003264 | 0.003264 |
| III day              | 0.003115 | 0.003081 | 0.003146 | 0.003146 |
|                      | 0.003177 | 0.003007 | 0.003044 | 0.003044 |
| Accepted result      | 0.003115 | 0.003081 | 0.003146 | 0.003146 |

Table 2. Results of studies of the control sample of cadmium concentration of 0.0035 mg/dm³.
A sample of control of cadmium concentration of 0.0035 mg/dm³, analyzed taking into account the use of the PLP-01M microwave laboratory system, is defined as 0.00315 mg/dm³, in the case of sample preparation in accordance with GOST 26929 - 0.00299 mg/dm³.

**Table 3.** Research results of the control sample of cadmium concentration 0.0075 mg/dm³.

| COCA=0.0035 mg/dm³ | X₁ | X₂ | Xₐr | Xₐr/day |
|---------------------|----|----|-----|---------|
| **PLP-01M**         |    |    |     |         |
| I day               | 0.007412 | 0.007424 | 0.0074 | 0.00739 |
| II day              | 0.007382 | 0.007394 | 0.00738 | 0.007401 |
| III day             | 0.007229 | 0.007246 | 0.007246 | 0.007265 |
| Accepted result     |    |    |     | 0.007352 |
| **GOST 26929**      |    |    |     |         |
| I day               | 0.006681 | 0.006953 | 0.006817 | 0.006924 |
| II day              | 0.007121 | 0.007101 | 0.007111 | 0.00696 |
| III day             | 0.007104 | 0.007001 | 0.007053 | 0.007025 |
| Accepted result     |    |    |     | 0.00697 |

A sample of control of cadmium concentration of 0.03 mg/dm³, analyzed taking into account the use of the PLP-01M microwave laboratory system, was determined as 0.0285 mg/dm³, in the case of sample preparation in accordance with GOST 26929 - 0.0268 mg/dm³.

**Table 4.** Research results of the control sample of cadmium concentration of 0.03 mg/dm³.

| COCA=0.0035 mg/dm³ | X₁ | X₂ | Xₐr | Xₐr/day |
|---------------------|----|----|-----|---------|
| **PLP-01M**         |    |    |     |         |
| I day               | 0.02864 | 0.02911 | 0.028875 | 0.029435 |
| II day              | 0.02776 | 0.02754 | 0.02765 | 0.027695 |
| III day             | 0.02786 | 0.02793 | 0.027895 | 0.028458 |
| Accepted result     |    |    |     | 0.028529 |
| **GOST 26929**      |    |    |     |         |
| I day               | 0.02652 | 0.02647 | 0.02684 | 0.026394 |
| II day              | 0.02743 | 0.02711 | 0.02727 | 0.027138 |
| III day             | 0.02706 | 0.02695 | 0.027005 | 0.026745 |
| Accepted result     |    |    |     | 0.026759 |
systematically significant error into the analysis results). The error of the certified CC value does not exceed one third of the error characteristics of the analysis results.

Under the conditions of repeatability and intra-laboratory precision, the results of the CC analysis were obtained.

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 5.

Table 5. Evaluation of the results of operational control of the measurement procedure using samples for control of cadmium during the implementation of the PLP-01M microwave laboratory system in the laboratory.

| The entered value Cd, mg/dm³ | Test result, mg/dm³ | Control measurement result, Xav, mg/dm³ | The result of the control procedure, Xav, mg/dm³ | Control standard, Ka | Evaluation of the acceptability of results, | Kav, mg/dm³ | < Kav |
|-----------------------------|--------------------|-----------------------------------------|-----------------------------------------------|--------------------|-----------------------------------------------|----------|-----|
| 0.0035                      | 0.0030628          | 0.00328628                              | 0.003095                                      | 0.00314801         | 0.00035199                                    | < 0.00097 | satisfactorily |
| 0.0035                      | 0.0028985          | 0.00299925                              | 0.0030735                                     | 0.00299042         | -0.00050958                                   | < 0.00097 | satisfactorily |
| 0.0075                      | 0.00739            | 0.00740075                              | 0.0072648                                     | 0.00735183         | -0.00014817                                   | < 0.002079 | satisfactorily |
| 0.0075                      | 0.0069243          | 0.00695975                              | 0.007025                                      | 0.00696967         | -0.00053033                                   | < 0.002079 | satisfactorily |
| 0.03                        | 0.029435           | 0.027695                                | 0.0284575                                     | 0.02852917         | -0.00147083                                   | < 0.008316 | satisfactorily |
| 0.03                        | 0.0265943          | 0.0271375                              | 0.026745                                      | 0.02675892         | -0.00324108                                   | < 0.008316 | satisfactorily |

4. Conclusion

Based on the results of the assessment of the operational control of the measurement procedure using samples to control the tests for the content of cadmium during the implementation of the PLP-01M microwave laboratory system in the laboratory, the analysis procedure was recognized as satisfactory.

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