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Improvement of Laboratory Services When using Sample Preparation in Microwave System

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ABSTRACT

Introduction: In the laboratory activity, the quality of testing analysis for determination of the toxic and heavy metals is considered as its ability to ensure the test validity. Improvement of measurement methods, sample preparation for analysis, the introduction of new equipment to improve the quality indicators of services are the most relevant tasks in the activities of testing laboratories.

Materials and Methods: With the purpose of comparison of results received by operation on atomic-absorption spectrometer "Quantum-2AT" with use of microwave laboratory system PLP(Pech Laboratory Probopodgotovka)-01M and at sample preparation according to Government Standards(GOST)-26929, four different groups of products were chosen as working samples. These products are fish, bread, milk, meat.

Results: The analysis of results of research of samples on the content of lead and cadmium by their determination on atomic absorption spectrometer "Quantum-2AT" with use of microwave laboratory system PLP(Pech Laboratory Probopodgotovka)-01M and sample preparation according to Government Standards(GOST)-26929 is presented. The analysis of the performed studies allowed estimating the time spent on one study taking into account the implementation of different types of sample preparation for analysis. It is established that higher accuracy indicators are achieved in case of microwave decomposition using microwave laboratory system PLP-01M. It is necessary to note a higher level of automation of the sample preparation process in case of application of microwave laboratory system PLP-01M in comparison with the traditional algorithm of sample preparation for analysis according to GOST 26929.

Conclusion: It is shown that the validity of the results is determined by several factors: the methods used, the equipment and reagents used, the competence of the personnel, the procedure of internal laboratory control. Using microwave laboratory system PLP-01M for the decomposition of samples before atomic absorption analysis has improved quantitative indicators of quality of the analysis.

Key Words: Safety; Quality; Control; Microwave decomposition, Xenobiotics, Lead, Cadmium; Indicators of accuracy

INTRODUCTION

The competitiveness of analytical laboratories, as well as of any organization, is determined by the quality of services provided, regardless of the specifics of its activities. Because quality is an economic category, the ratio "achieved indicators of quality of measurement (testing) results/price of the service, expended on quality indicators maintenance by the laboratory" is of key importance.

The quality of the provided service is closely connected with the competence of the laboratory. Laboratory competence is a proven ability to apply knowledge and skills in practice (under Group of standards for Russia GOST R ISO

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9000 which is National Standard of Russia). The quality of provided services and the competence of the laboratory determines its competitiveness. In the activity area of laboratories, quality is considered as the ability of laboratories to provide testing services, namely to provide reliable test results, while ensuring the required accuracy of measurements (tests). The following factors influence the accuracy of the results: the methods used, the equipment and reagents used, the competence of the personnel, the procedure of internal laboratory control. The improvement of measuring methods, preparation of the sample for analysis, new equipment launching, which allows improving the quality indicators of the provided services, are the most actual tasks in the activity of testing laboratories.

MATERIAL AND METHODS

In this paper the analysis of the results of lead and cadmium content determined by the atomic absorption spectrometer "Quantum-2AT" with the use of microwave laboratory system PLP-01M and in case of dry ashing under GOST 26929 "Mineralization of food samples to determine the content of toxic elements". To reduce the time of long and labour-intensive phase of sample mineralization, the microwave decomposition of the sample was carried out in PLP-01M, where the organic matrix was destroyed under the influence of high temperature, pressure and microwave fields in hermetic vessels following the instruction of microwave laboratory furnace PLP-01M (manufacturer "Ural-Gefest"). For comparison of results obtained at the atomic-absorption spectrometer "Quantum-2AT" with the use of microwave laboratory system PLP-01M and at sample preparation following GOST 26929, four different groups of products were selected on the working samples, most fully covering the range of results obtained in studies. The following testing samples were selected: river fish, wheat bread, cow's milk and beef. Standard samples of lead ion solution composition, cadmium, given in Table 1, were chosen as materials for the tests.

Table 1: Characteristics of certified reference samples of lead and cadmium ion solution composition

Name of the standard sample (by GOST 8.315-2019)	State standard sample number	Type Approval Certificate	Tested value of the sample, mg/cm	Producer
State standard sample of lead ion solution composition	7252	№ 4347	1,0	Ural Research Institute of Metrology
State standard sample of cadmium ion solution composition	7472	№ 3046	1,0	

A sampling of the selected food products was carried out by GOST 26929 and the instructions of microwave laboratory oven PLP-01M "Ural-Gefest". The volume of the sample taken for analysis is given in Table 2.

Table 2: Quantity of sample taken for analysis

Product category (samples)	Sample weight in g (sample volume in cm)			
	Sample preparation according to GOST 26929		Microwave decomposition on PLP-01M	
	Pb(Lead)	Cd(Cadmium)	Pb	Cd
beef	10	10	2	2
freshwaterfish	10	5	2	2
cow'smilk	100	50	2	2
whitebread	20	15	2	2

The result of the control K procedure was calculated by the formula:

$$К_к = Xcp(n)+д - Xcp(n) - С_д, \text{ mg/kg} \quad (1)$$

where $С_д$ – additive value, mg/kg;

Xcp(n)+д – concentration of a determining element in an average work sample with a defined additive of a determining element, mg/kg;

Xcp (n) – concentration of the measuring element in the average work sample, mg/kg.

The K control standard is calculated by the formula:

$$K = \sqrt{\left(\Delta\pi_{Xn+\partial}\right)^2 + \left(\Delta\pi_{Xn}\right)^2}, \text{ mg/kg} \quad (2)$$

where ± Δл – measurement error characteristic corresponding to the content of the element to be determined in the additive sample, calculated by the formula:

$$\Delta л = 0{,}01 \times \delta л \times X, \text{ mg/kg} \quad (3)$$

±Δл – measurement error characteristic corresponding to the content of the element to be determined in the additive sample, calculated by the formula:

$$\Delta л = 0{,}01 \times \delta л \times Xn, \text{ mg/kg} \quad (4)$$

±δл – the relative value of the measurement error characteristic, established during the procedure implemented in the laboratory and ensured by controlling the stability of the analysis results:

$$\delta л = 0{,}84 \times \delta, \% \quad (5)$$

±δ – method accuracy index, %.

The procedure of analysis was considered satisfactory when the condition was met:

$$|Кк| \leq K \quad (6)$$

RESULTS

Independent measurement results were obtained by the same method on the same test objects, in the same laboratory, by the same operator, using the same equipment - atomic absorption spectrometer "Quantum-2AT", within a short period, i.e. conditions of repetition were ensured. To check the reproducibility of the measurement results, the studies were performed by different operators using the same equipment on different days, i.e. intermediate (intra-laboratory) precision conditions were met. Within 30 working days, the selection of observation results and analysis of data obtained during the work on the atomic absorption spectrometer "Quantum-2AT" with the use of microwave laboratory system PLP-01M and sample preparation by GOST 26929. The average values obtained under conditions of repeatability were registered in the intermediate table. The accuracy control was carried out by the method of additions: at the stage of sample preparation the additive of the determined substance - lead and cadmium - was added. Under intermediate precision conditions, five mean values for each product type were obtained: for samples with and without the addition of a defined element. To evaluate the metrological characteristics, an average of five average values for each product type was recorded in the final table 3.

Table 3: Comparative characteristics of determination of lead and cadmium content based on various sample preparation types

Sample	Determining element	Mean value, mg/kg		Additive amount, mg/kg	Control measurement result of the sample with additive, mg/kg	
		GOST	PLP-01M		GOST	PLP-01M
Fresh water fish	Pb(Lead)	0.0686	0.0734	0.05	0.1158	0.1228
	Cd(Cadmium)	0.0444	0.0522	0.03	0.0728	0.0826
White bread	Pb	0.0136	0.0172	0.015	0.0248	0.0284
	Cd	0.0342	0.0400	0.015	0.0438	0.0528
Cow's milk	Pb	0.0304	0.0342	0.02	0.0456	0.0498
	Cd	0.00854	0.01056	0.01	0.01674	0.018832
Beef	Pb	0.3180	0.3360	0.10	0.4060	0.4280
	Cd	0.0334	0.0426	0.03	0.0590	0.0694

The result of the control procedure K and the control standard K were calculated according to formulas 1, 2 considering the calculations according to formulas 3, 4, 5. Also, compliance with the comparison condition according to Formula 6 was checked. Tables 4 and 5 summarize the report on the quality control procedure for the implementation of the additive method analysis.

Table 4: The results of the control of the procedure of analysis using the additive method (testing of samples for lead content)

GOST 26929		Using PLP-01M	
Кк	K	Кк	K
-0.0028	0.0204	-0.0006	0.0216
-0.0038	0.0043	-0.0038	0.0050
-0.0048	0.0083	-0.0044	0.0091
-0.0120	0.0780	-0.0080	0.0823

Table 5: The results of the control of the procedure of analysis using the additive method (testing of samples for cadmium content)

GOST 26929		Using PLP-01M	
Kк	K	Kк	K
-0.0016	0.0086	0.0004	0.0098
-0.0054	0.0056	-0.0022	0.0067
-0.0018	0.0019	-0.0017	0.0022
-0.0044	0.0068	-0.0032	0.0082

The analysis of the conducted tests made it possible to establish the time spent on performance of one test taking into account realization of various types of preparation of the sample for the analysis, the obtained data are summarized in tables 6, 7.

Table 6: Time spent on one test

Name of work	Time allowance, h	
	Sample preparation by GOST 26929	Microwave decomposition on PLP-01M
Sample collection and registration, sample identification, journal entries	0.1 0.2	0.1 0.2
Taking the awning	0.1	0.1
Reagent preparation	0.2	0.2
Sample Mineralization	2.2	–
Microwave sample decomposition in microwave laboratory system PLP-01M	–	1.2
Preparation of the device for operation, construction/ refinement of the graduation chart	0.4	0.4
Performing sample analysis on atomic absorption spectrometer "Quantum-2AT"	0.11	0.11
Conducting internal laboratory control, making calculations, results from registration	0.3	0.3
Preparation of utensils	0.3	0.3
Document Processing	0.3	0.3

Table 7: Calculation of net profit with the application of microwave laboratory system PLP-01M (in Russian rubles)

Name of Items	Expenses
Profit before tax of one sample	156.86
Net profit per sample	90.75
Annual net profit in laboratory	1 456 989.56

The capital investment for the introduction of new equipment is 1,917,790 rubles for the atomic absorption spectrometer "Kvant-2AT" with the application of the microwave laboratory-tor system PLP-01M (introduced "Kvant-2AT" and PLP-01M simultaneously). The total cost of testing, taking into account the capital costs of the introduction of the new equipment will be 4,456,713.53 rubles. The payback period of the "Quantum-2AT" PPL-01M capital investment is 1 year 9 months and 7 days. To reimburse the capital costs for the introduction of the new equipment of the testing laboratory it is necessary to perform 28,412 tests.

DISCUSSION

Based on the results of the control evaluation of the measurement procedure using lead and cadmium control samples, the analysis procedure is considered satisfactory.

However, it was found that the best accuracy data are achieved by microwave decomposition (using microwave laboratory system PLP-01M). The introduction of the microwave laboratory system PLP-01M will be beneficial if the performance of the new equipment meets the laboratory's needs in terms of the number of tests. Application of microwave laboratory system PLP-01M will allow reducing the time of sample preparation in 19,5 times, and expenses item on "Raw materials and basic materials" at conducting one research will be reduced by 2,6 times. It is established that the reduction of the sample portion in case of microwave decomposition up to 2 g will reduce the number of reagents used by 3.7 times. It is necessary to note the higher level of automation of the process of sample preparation in case of application of microwave laboratory system PLP-01M in comparison with the traditional algorithm of sample preparation, providing various methods of mineralization according to GOST 26929.

CONCLUSION

The use of microwave laboratory system PLP-01M for sample decomposition has a significant impact on quantitative indicators of service quality, namely improvement of intra-laboratory control indicators, service delivery time is signifi-

cantly reduced, the accuracy of testing and, consequently, the reliability of test results increases and the costs of the reagents used are reduced.

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