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Results of studies of wheat bread for lead content using the additive method

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Abstract. The relevance of the problem under consideration is due to the need for hardware to cover a wide range in the determination of heavy metals in food. One of the primary and important tasks remains to ensure control of the quantitative content of lead, the introduction and application of fast and reliable methods of their research. The analysis of the data obtained shows that all the results on the study of wheat bread for the content of lead, obtained by stripping voltammetry and atomic absorption spectrometry for the assessment of precision and operational control of the error using the method of additions are satisfactory. The obtained monitoring results indicate that in the range of less than 0.01 mg of lead per 1 kg of wheat bread prevail (75.6%) over other ranges. The least registered sample results are (2.2%) with the range of obtained data: 0.1-1.0 mg/kg.

1. Introduction

In the foreign and domestic literature, the issue of the effect of nutrition on human health has been widely studied [1-4], 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 for reducing the residual amounts of contaminants in food products [5-9]. 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 [10-16]. 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 as soon as possible, is an important and urgent task [17-



21]. The primary and important task remains to ensure the control of the quantitative content of toxic elements, the introduction and application of fast and reliable methods of their analysis [19-23]. Implementation of quality management systems is of great importance [24, 25].

This paper considers such a toxic element as lead, which is mandatory for control in food products, in accordance with the requirements of the Technical Regulations of the Customs Union 021/2011 (hereinafter referred to as TR CU 021) "On food safety" (figure 1).

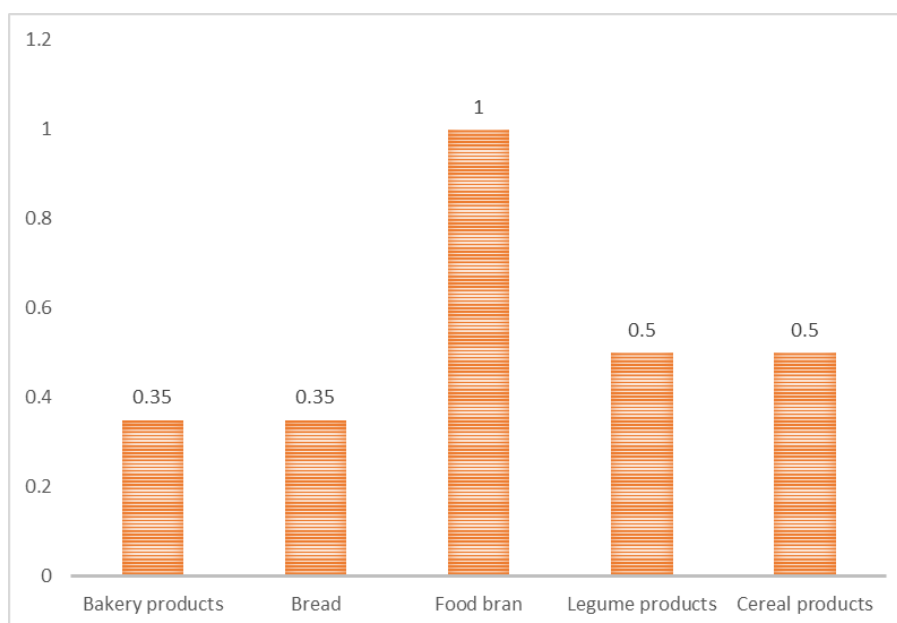


Figure 1. Permissible levels of lead in flour, cereals and bakery products according to the requirements of technical regulations, mg/kg, not more.

Lead belongs to the highest hazard group toxicants.

2. Material and methods

The development and approbation in the laboratory of modified test methods of the conformity confirmation method is an important and urgent task. The urgency of the problem under consideration is due to the expansion of supplies of products from other countries with a very diverse range of approved drugs. It seems important to ensure the necessary control of contaminants in food, to use sufficiently fast and reliable methods for their analysis. Conducting comparative tests with these methods in order to ensure the control of lead in food products, analyzing the accuracy, precision, repeatability of the results obtained makes it possible to judge the effectiveness of the application of these methods, and also allows a comparative analysis of the results obtained. An analysis was carried out in order to optimize the methods used and to develop the most economical and efficient option for optimizing the equipment used in the laboratory while maintaining the metrological characteristics of the accuracy of the results obtained.

The methodological basis is the standards for research methods, test methods, operating manuals and instructions for the use of equipment.

The methodological basis is the standards for research methods, test methods, operating manuals and instructions for the use of the specified equipment.

Quality control of the analysis results during the implementation of the method in the laboratory provides for the control of the stability of the analysis results.

The object of research is samples of wheat bread.

Equipment for research of lead content in food raw materials and food products is shown in figure 2.



Figure 2. Research instrument base.

Analytical lines of elements for atomic absorption spectrometry are shown in table 1.

Table 1. Analytical lines of elements for atomic absorption spectrometry (determination of lead).

Analytical line wavelengths, nm	Relative ratio weakening of sensitivity
217.0	1.0
283.30	2.0
261.41	36
202.20	50
205.32	340

Table 2 shows the distinctive features of voltammetric methods of analysis using the analyzer "TA-4".

Table 2. Range of determined lead content on the analyzer "TA-4".

Determined content range, mg/kg	Indicator electrode
from 0.01 to 6.0 inclusive	Mercury film on a silver backing
from 0.01 to 6.0 inclusive	Silver modified
from 0.005 to 5.0 inclusive	Gold-carbon

During the research, the standard deviation of the intralaboratory repeatability S_r and intermediate precision S_R of atomic absorption analysis and stripping voltammetry were assessed.

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

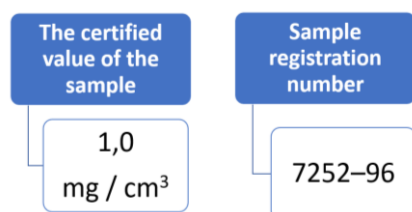


Figure 3. Characterization of a standard sample of the composition of a solution of lead ions.

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 additive method, the control means were working samples of a stable composition and the same samples with a known lead addition.

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

The purpose of the study is the development and testing in the laboratory of modified test methods of the conformity confirmation methodology. To achieve the goal, the following tasks were set:

- implement a program of comparative tests, which allows to evaluate and compare the accuracy of the results obtained with the existing laboratory equipment;
- evaluate the accuracy of the test results.

The initial and, for most analytical schemes, the inevitable stage of sample preparation for the determination of components remains long, multi-operational, makes a significant contribution to the overall analysis error and prevents the implementation of the capabilities of instrumental methods.

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. The most common classification of xenobiotics is the following, representing the greatest danger in terms of prevalence and toxicity:

- 1) metabolites of microorganisms (aflatoxins, patulin, staphylococcal toxins, botulinum toxin, mycotoxins);
- 2) toxic elements (lead, cadmium, arsenic, mercury, copper, zinc, tin, iron);
- 3) substances used in animal husbandry (amino acids, minerals, enzymes, antibiotics (chloramphenicol), tranquilizers, antibacterial substances, antioxidants, flavors, dyes, hormones and hormone-like compounds);
- 4) pesticides (HCCH, DDT, organomercury compounds and hexachlorobenzene; herbicides used to control weeds, zoocides to control rodents, insecticides to control harmful insects);
- 5) nitrates, nitrites, nitrosamines;
- 6) dioxins and dioxin-like compounds (polychlorinated dibenzodioxins (more than 75 isolated), a group of polychlorinated dibenzofurans (more than 135 isolated) and polychlorinated biphenyls (more than 80 PCBs isolated));
- 7) polycyclic aromatic hydrocarbons;
- 8) radionuclides (strontium-90, rubidium-87, cesium-137, etc.);
- 9) food additives (sweeteners, flavorings, colors, antioxidants, stabilizers, etc.).

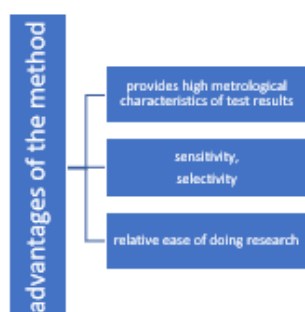


Figure 4. Advantages of stripping voltammetry.

In the conditions of market relations at any enterprise, including testing laboratories, the relevance of quality management is determined by its focus on ensuring 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.

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

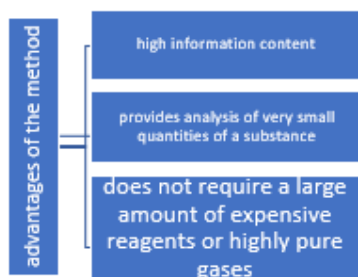


Figure 5. The advantages of atomic absorption spectrometry.

3.1. Control of intermediate precision

Under the conditions of repeatability and intermediate precision, five average measurement results were obtained for a wheat bread sample.

The critical range factor $f(n)$ for the five results is 3.9. The repeatability standard deviation S_r is presented in table 3.

For the Kvant-2AT spectrometer:

$$(CR_{0.95}(5), \%) = f(5) \times S_r (\text{Pb}) = 3.9 \times 18 = 70.2 \%$$

For the analyzer "TA-4":

$$(CR_{0.95}(5), \%) = f(5) \times S_r (\text{Pb}) = 3.9 \times 13 = 50.7 \%$$

Table 3. Results of studies of wheat bread for lead content (standardized level of permissible concentration 0.5 mg/kg).

№	TA-4	Kvant-2AT	C _D = 0.03		C _D = 0.03	
			sample preparation stage		measurement stage	
1	0.011	0.011	0.037	0.036	0.036	0.041
2	0.0094	0.013	0.033	0.041	0.037	0.044
3	0.0101	0.015	0.037	0.034	0.041	0.038
4	0.0113	0.0095	0.036	0.035	0.032	0.037
5	0.0107	0.01	0.039	0.037	0.038	0.031
X _{avr}	0.0105	0.0117	0.0364	0.0366	0.0368	0.0382
<i>Assessing the precision of results $(X_{max} - X_{min}) \leq CR_{0.95}(5)$</i>						
	0.002<0.005	0.006<0.008	0.006<0.018	0.007<0.026	0.009<0.019	0.013<0.027

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 4.

Table 4. Study of samples for lead content.

№	additive at the stage of sample preparation				additive at measurement stage			
	TA-4		Kvant-2AT		TA-4		Kvant-2AT	
	K _к	K	K _к	K	K _к	K	K _к	K
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 obtained results, we state that the condition $(X_{max} - X_{min}) \leq CR0.95(5)$ is satisfied 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 operational control of the laboratory test procedure using the control procedure to control the error using the addition method are presented in table 5.

Table 5. Bread research results (permissible level 0.15 mg/kg).

	TA-4	Kvant-2AT	TA-4	Kvant-2AT	TA-4	Kvant-2AT
			addition at the stage of sample preparation $c = 0.05$		additive at the stage of measurement with $= 0.05$	
$X(5)_{avr}$	0.0105	0.0117	0.0364	0.0366	0.0368	0.0382
X_{avr}	0.0111		0.0365		0.0375	
<i>Evaluation of the precision of results obtained by different methods</i>						
	5.4 % < 17 %		0.3 % < 17 %		1.9 % < 17 %	
			K_K	K	K_K	K
			-0.0046	0.00544789	-0.0036	0.00558467

The analysis of the presented results in table 5 shows that all the results of studies of bread samples for lead content 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 bread from January 2019 to October 2020. We assessed the distribution of research results depending on the concentration of lead in bread (figure 6).

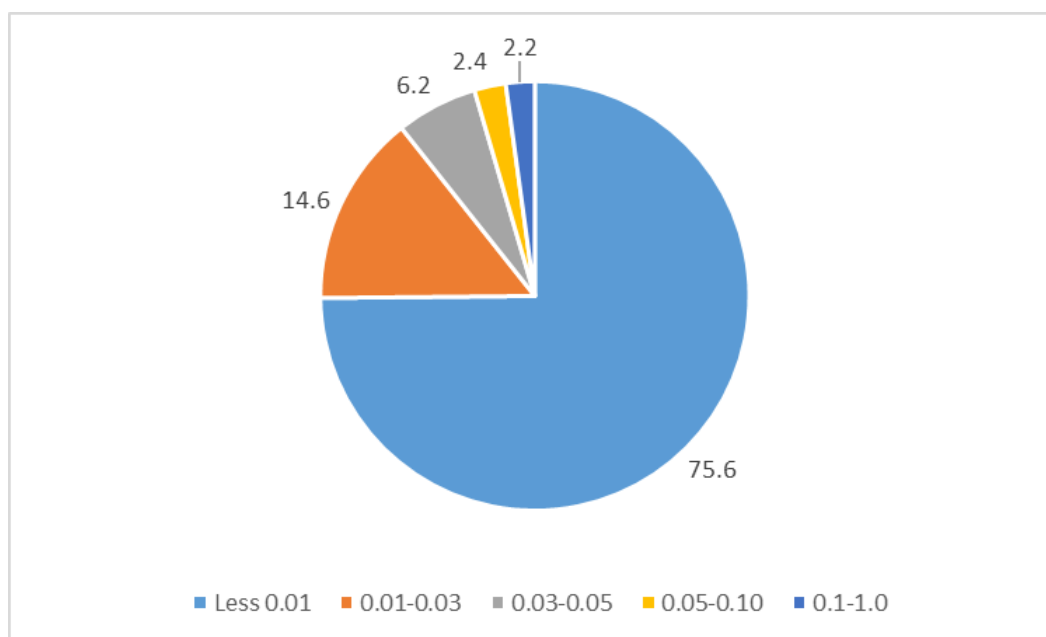


Figure 6. Distribution of test results depending on the concentration (mg/kg) of lead in wheat bread samples, %.

The obtained monitoring results indicate that in the range of less than 0.01 mg of lead per 1 kg of wheat bread prevail (75.6%) over other ranges. The least registered sample results (2.2%) with the range of obtained data: 0.1-1.0 mg/kg.

4. Conclusion

The analysis of the data obtained shows that all the results on the study of the content of lead in bread by assessing the 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 in the results of lead studies in the testing laboratory, both under conditions of intermediate precision and under repeatability conditions.

Wheat bread should be monitored for lead on an ongoing basis.

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