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Metrological Support and Improvement of Reliability and Efficiency of Quality Tests of Oil-Fat Yeilding Products

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ABSTRACT: The article describes the assessment of the competence of testing laboratories of the oil and fat industry, and also describes the development of a procedure for the certification of testing laboratories to increase the reliability of measurement results and the implementation of international standards ISO, O'zDSt and ISO/IEC. Three levels of competence of testing laboratories are recommended in accordance with ensuring the reliability of the results according to methods including the errors of type I, II or III.

KEYWORDS: Measurement, testing, measurement process, measurement error, laboratory competence.

I. INTRODUCTION

The control of technological processes and the quality of oil and fat products will always be among the most important tasks of production. The most important component of the technical base for ensuring the control of technological processes is measurement, especially analytical control.

A comprehensive integrated approach to the problem of controlling technological processes and the quality of oil and fat products implies the use of methods that allow to establish the value of a defined indicator with minimal error and to conduct research in competent testing laboratories (TL), which are one of the main elements of an enterprise that determine product quality.

For oil and fat industry, an important indicator that primarily characterizes the quality of raw materials and determines the quality of the extracted oil is the acid number. the requirements for this indicator are constantly being tightened. an equally important indicator is the color number, a standardized indicator of the oil produced. it allows you to quickly assess the effectiveness of cleaning oils, and in some cases their authenticity.

II. PROBLEM DEFINITION AND ITS DECISIONS

Complex fat-and-oil system is emulsion products, currently produced with different fat content. The mass fraction of fat in such products is one of the main indicators determining the quality and authenticity.

Therefore, the improvement and certification of measurement and control methods of the above parameters seems relevant.

On the other hand, the transition of the Uzbekistan to the level of world standards also requires ensuring the uniformity of measurements, which, in accordance with the Law on Technical Regulation, is a link connecting the economic, social and environmental safety of society.

The legal basis for the uniformity of measurement is the documents listed in Table 1.

Table 1.
Normative legislative

Technical Regulation Act
O'zDSt ISO / IEC 17025: 2007 General requirements for the competence of testing and calibration laboratories
GOST R ISO 5725-2002 Accuracy (trueness and precision) of measurement methods and results
RMG 58-2003 Assessment of the quality of the testing laboratory of food products and food raw materials
RMG 61-2003 Indicators of accuracy, correctness, precision of methods for quantitative chemical analysis

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The current legislation (table 1) on the one hand requires interlaboratory testing, on the other hand. It lays the foundation for the formation of the certification procedure for TL. These aspects are interrelated, as for conducting interlaboratory tests knowledge of the competence of the involved subjects is necessary.
Currently, TL competence is confirmed as a result of the accreditation procedure according to O‘zDSt ISO/IEC 17025: 2007 General requirements for the competence of testing and calibration laboratories. These standards put forward general requirements for TL and lead to the form of accreditation, using the concept of certification as a checking procedure of the testing laboratory to determine its compliance with the established requirements, which have a general organizational legal (legal) character. The above documents describe accreditation procedures for testing laboratories and general requirements, but do not set standards on the criteria by which accreditation is made. The above documents also do not imply an assessment of the activities of the staff [1].
The present work on the studied frequent (special) examples allows us to create methodological basics to solve such a problem. As can be seen from Figure 1, the actual subject of measurement is not an individual analytic operator, but a measurement laboratory as a whole. In general, the structure of the testing laboratory can be described by the following scheme presented in Figure 1, where the method and factors are highlighted as objects and procedures that determine the measurement error in general and they form particularly the subject - the laboratory.

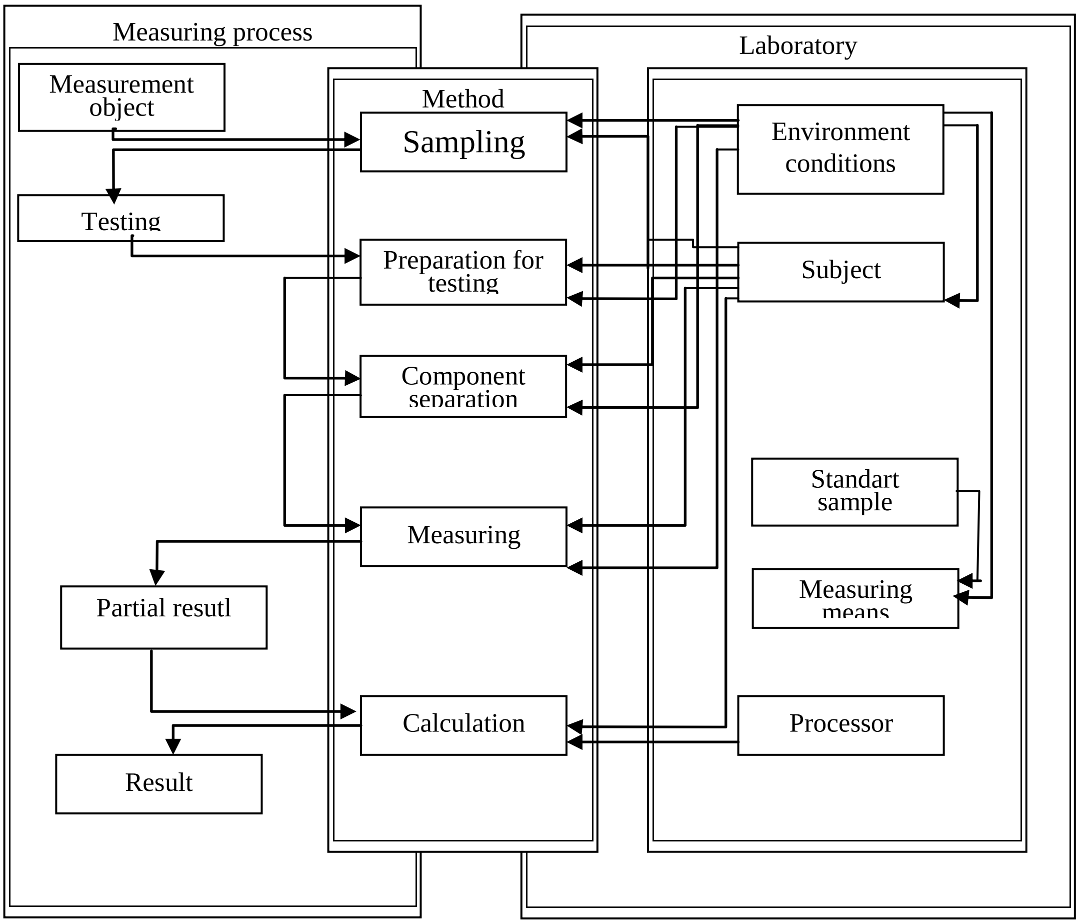

Fig.1. The scheme of interrelations of metrological important elements of the laboratory in the measurement process.

Since the laboratory is a basic element of the measurement system, the objective criterion of its competence is the ability to perform measurements of known complexity with the necessary accuracy, i.e. general metrological characteristic. Such an ability, in principle, can be established only as a result of a special experiment - intra and inter-laboratory metrological certification, based on a system account of the effects of equipment, personally, external conditions and methodological principles on the accuracy of measurements. From the entire array of significant factors affecting the error of the laboratory, we can distinguish several main groups and their corresponding sources of error:

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method, person, object of measurement, completeness of separation (separation), measurement, instrument, reagents, measurement conditions.
In general, the measurement error in the laboratory, which characterizes their quality, can be represented in the following in formula (1):

$$\Delta_{lab} = \sqrt{\Delta^2_{pers} + \Delta^2_{obj} + \Delta^2_{sep} + \Delta^2_{inst} + \Delta^2_{react} + \Delta^2_{cond} + \ldots} \quad (1)$$

where, Δ_{object} is a component of the error introduced by the properties of the object;
Δ_{inst} - component of the error due to measuring instruments;
Δ_{react} is a component of the error introduced by the quality of reagents and materials;
Δ_{sep} is the error component caused by separation and measurement procedures;
Δ_{cond} is a component of the error introduced by fluctuations of conditions;
Δ_{pers}- component of the error due to the actions of person.

Formula (1) is a mathematical description of intra-and interlaboratory interactions, in general, it corresponds to the principles of the element-by-element attestation method. The essence of the element-by-element certification consists in determining the total error of the measurement result on the basis of particular estimates of its components. Detailed examination of all these objects makes it clear that it is necessary to study all the factors which affect the accuracy and quality of measurements [2].
The methods used for the analysis of both fat-containing and other products can be divided into three groups, in accordance with Table 2. The first group represents the elementary units of analysis that are universal for any industry and carry the error of the first kind only. These are relatively simple methods with an error of only the 1st kind. The second group is relatively simple methods that have an error of the 2ndkind. The third group includes more complex methods, often specific to the industry [3].

Table 2.
Compliance with the level of competence of the laboratory manifested in the methods of kinds error.

Error	Indicator and method	Laboratory competency level
1stkind	weight (weighing); density (pycnometric method)	Elementary
1stkind 2ndkind	acid content in aqueous solutions (aqueous acid-base titration);	Average
1st kind 2nd kind 3rd kind	acid number in organic solutions (non-aqueous acid-base titration); mass fraction of fat (extraction); chromaticity (comparative);	high

The assessment of laboratory quality is carried out by the value of the Z-index RMG 58-2003. The assessment can not be made, objectively due to their heterogeneity and relatively low stability over time, thus, there is a need to create special samples.
Certification samples are the means of metrological certification of testing laboratories in the form of the substance or material which composition or properties are established upon certification and are encrypted with respect to the laboratory to be certified. Attestation samples retain all basic requirements and properties of standard samples [5, 6].
An essential step in the construction of the above-described system for analyzing the competence of testing laboratories is assessing the errors, which is used to certify methods when applied to certification samples. Knowledge of the error of these methods provides basis for evaluating the results obtained in laboratories. During the certification of selected MMTs, emphasis was also placed on their refinement and improvement.
The presence of mass and density samples is emphasized both by the availability of these procedures in the form of elementary analysis units in most methods and by the mandatory control of the finished product by the indicators of net quantity [2].

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As samples of the mass, should be used the set of samples that meet the requirements for RS and having different densities. As a sample similar in density to weights, bars or cylinders (or any other convenient form) made of metal can be used, the density of such samples should be about $8{\cdot}10^3$ kg/m^3. As a sample that is close in density to natural weighing objects (food products), you can use, for example, glass ampoules filled with water or other material providing the total density of the ampoule with material less than 10^3 kg/m^3. It is also possible to use ampoules filled with gas. The mass of such samples must be certified using accurate weighing methods.

III. CONCLUSION

It is convenient to use liquids their mixtures and solutions as the samples of density, the properties of which are well studied, and the density is known as a reference material. Certification of this group of samples is carried out according to the preparation procedure or according to standard reference data.

Introduction to the oil and fat industry developed procedure for certification of testing laboratories increases the reliability of measurement results. Three levels of competence of testing laboratories are distinguished in accordance with ensuring the reliability of results by methods including errors of kind I, II or III.

The proposed certification samples reproduce the properties of fat-and-fat food products, adequately and provide the ability to control the quality of tests in the fat-and-oil industry.

Laboratory certification is the final act confirming its competence. By virtue of this certification must be included in the accreditation procedure TL.

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