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Features of the Method for Performance of Measurements of Mass Fraction of Water in Food By Thermogravimetric Method

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ABSTRACT: This article discloses all aspects of the methodology for measuring the mass fraction of water in food by the thermogravimetric method,as well as the physical essence of the methods for measuring the mass fraction of water in various types of food of raw materials and ready products. It is explained that in the food industry, where analytical measurements are the main ones, the error of the measurement result also depends on many other factors, one of such measurement methods is the determination of the mass fraction of water in food by the thermogravimetric method. It also explains the terms used in the thermogravimetric measurement method, such as free water and many others. The essence of the working and arbitration options for the implementation of the measurement method, requirements for measuring instruments, auxiliary devices, materials and solutions,the sequence of operations in preparation for taking measurements,drying regimes approved by national standards for certain food products are analyzed in this article.

KEY WORDS: methodic of measurement techniques (MMT), thermogravimetry, standards, measurements, technique, documents, error, result, mass fraction, certification, food product, sample, desiccator, drying modes.

I. INTRODUCTION

To provide the consumer with high-quality food, first of all, it is necessary to control carefully the characteristics of the raw materials as a final product, and to comply with the parameters of all relevant technological processes, the quality and quantity of energy carriers [1]. Thus, it can be argued that only the accuracy and economic efficiency of measurement are the most important part of the production of products, it is possible to achieve high production efficiency. At the same time, regarding scientific research on the development of new products, as well as equipment and technological processes, it can be argued that the main link here will be measurements, and then - the comprehension and interpretation of their results (measurement information). At the same time, the need to reduce the cost of production while increasing its quality level requires paying attention to improving the quality and effective use of measuring equipment, as well as to the received measuring information. This is directly related to the state of metrological support of production and scientific research [2]. It is in the field of the food industry that the most important measurement feature is associated with sources of error and reliability of measurement results [3,5,6,8]. Conventionally speaking, when it issaid about usage, "ordinary" types of measurements (mass, pressure, length, etc.), the error of the measurement result Δ_{pi} is determined mainly by the error of the used measuring instrument Δ_{si}. In the food industry, where analytical measurements are the main ones, the error of the measurement result also depends on many other factors, one of such measurement methods is the determination of the mass fraction of water in food by the thermogravimetric method.

II. LITERATURE REVIEW

Depending on the measuring instruments used, the methods are divided into measuring, registration, calculation, sociological, expert and organoleptic.

Measuring methods are based on information obtained using measuring and control instruments. Measuring methods are used to determine indicators such as mass, size, optical density, composition, structure, etc.

Measuring methods can be subdivided into physical, chemical and biological.

Physical methods are used to determine the physical properties of products - density, refractive index, viscosity, stickiness, etc. These methods include microscopy, polarimetry, colorimetry, refractometry, spectroscopy, rheology, luminescence analysis and others.

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Chemical methods are used to determine the composition and amount of substances included in the product. They are subdivided into quantitative and qualitative - these are methods of analytical, organic, physical and biological chemistry [7,9].
Biological methods are used to determine the nutritional and biological value of products. They are divided into physiological and microbiological. Physiological is used to establish the degree of assimilation and digestion of nutrients, harmlessness, biological value. Microbiological methods are used to determine the degree of contamination of products by various microorganisms.
Registration methods are methods for determining indicators of product quality, carried out on the basis of observing and counting the number of certain events, items and costs. These methods rely on information obtained by registering and counting certain events, for example, counting the number of defective items in a batch, etc.
Calculation methods reflect the use of theoretical and empirical dependences of product quality indicators on its parameters. These methods are used mainly in the design of products, when the latter cannot yet be an object of experimental research. The same method can be used to establish relationships between individual indicators of product quality.
Sociological methods are based on collecting and analyzing the opinions of actual and potential consumers of products; carried out orally, by means of a survey or distribution of questionnaires, through conferences, meetings, exhibitions, tastings, etc. This method is used to determine the weighting factors.
Expert methods are methods carried out on the basis of a decision made by experts. Such methods are widely used to assess the level of quality (in points) on establishing the range of indicators taken into account at various stages of management, determining generalized indicators based on a set of single and complex quality indicators, and also on certifying product quality. Expert methods for assessing product quality are used when it is impossible or inexpedient to use calculation or measurement methods for specific assessment conditions.They are used independently or in combination with other methods in assessing the normative and technical documentation for products and product quality, in choosing the best solutions implemented in product quality management, as well as for: classifying the evaluated products and consumers; determination of the range and weighting factors of quality indicators; selection of baseline samples and determination of baseline values; measuring and evaluating indicators using the senses; evaluations of single indicators, the values of which are determined by a calculation or measurement method; determination of complex quality indicators and in other cases [3].
Toassess the quality of products using expert methods, expert commissions (technical, tasting, etc.) are created. The expert commission consists of two groups: working and expert. The psychophysiological capabilities of the expert and the state of his health are taken into accountto form the expert group. The expert must be competent, business-like and objective.
The working group prepares and conducts an expert assessment of product quality and analyzes its results.
Assessment of the level of product quality is a set of operations, including the selection of a nomenclature of quality indicators of the product being assessed, the determination of the values of these indicators and their comparison with the bases. For conducting an expert assessment of the quality of products, they are presented in the form of a hierarchical structure.
Generalized indicators are referred to the highest level, and group complex indicators are referred to the lower level. At the bottom level of the structural diagram are single indicators. The number of hierarchy levels is determined by the complexity of the product, the number of indicators, the goal and the required accuracy.
Organoleptic methods are methods based on the analysis of sensory perception. The values of quality indicators are found by analyzing the received sensations on the basis of available experience. The interpretation of the term "organoleptic" comes from the Greek word "organon" (tool, instrument,organ) plus "lepticos" (inclined to take or receive) and means "identified with the help of the senses."
Organoleptic properties are the properties of objects assessed by the human senses (taste, smell, texture, color, appearance, etc.). Organoleptic analysis of food and flavoring products is carried out through tastings, i.e. research carried out with the help of the sense organs of a specialist taster without the use of measuring instruments [3,4].

III. SYSTEMATIZATION OF BASIC REQUIREMENTS AND TASKS

This MM (Measurement Methodology) in physical essence is an analogue of the methods for measuring the mass fraction of water in various types of food raw materials and finished products, standardized in the documents given in Table 1, and their design and certification in accordance with the requirements of O'zDSt (UzSSt) 8.016: 2002. In this regard, this MM should be considered in parallel with the corresponding standardized MMO'zDSt(UzSSt) 8.016: 2002

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as a certificate of their certification, in connection with additional certification which is not required later. The technique is intended for quality control of food raw materials and ready products (further as food products), as well as for performing arbitration and metrological works [9,10,12,13,14]. The technique can be used for the analysis of food products containing substances, the volatility of which is comparable to water, with a mass concentration of no more than $0.02C_{mm}$, where C_{mm} is the mass fraction of water in the analyzed object (for example, alcohols C_2-C_4, aldehydes C_1-C_6, ketones C_3-C_7, acids C_1-C_2, ethers C_6-C_{10}, esters C_4-C_8, primary, secondary and tertiary amines in the C_4-C_8 field, etc.) and components which are capable of being destroyed under these conditions with the formation of volatile substances.

Note: in the range of the value of the mass fraction of water more than 50% is applicable to the name of the measured quantity "mass fraction of dry residue", equal to $1\text{-}C_{mm}$.

IV. RESULTS AND DISCUSSION

A. Water in food is free water removed from the test sample at a temperature (80 ° C under vacuum) 105 ° C at normal atmospheric pressure. A sign of the completeness of water separation during drying is the constancy of the mass of the dried sample within ± 0.01 g (or: 0.1% of the original mass of the sample and moisture is a source of systematic error [11].

Table 1
Products and standards regulating MM of water in food (fragment).

Product	interstatestandardGOST
Cigarette raw materials	3714-79
Laundry solid soap and toilet soap	790-89
Powdered cosmetics	28768-90
Cosmetic products. method for the determination of water and volatile matter or dry matter	29188.4-91
Fish, marine mammals, invertebrates, algae and their products	13930-68
Powdered flavors	15113.4
Confectionery	5904-82
Flour	27668-88
Cereals	263121-84
Oilcakes and meal	13979.0-86
Grain and products of its processing	P 50436
Seaweed and herbs and products of their processing	20438-75, 13496.0-80
Food concentrates	15113.0-77

B. Measurement conditions

Measurements for this MM are performed in laboratory conditions: ambient temperature (15 ... 25) ^{o}C, relative humidity (40 ... 95)%, atmospheric pressure - not regulated).

C. Attributed characteristics of measurement error.

The use of this MM makes it possible to obtain a measurement result in the range of values of the mass fraction of water in the product from 3 to 97% with a confidence interval (with a confidence probability of 0.95) of a relative total error of no more than ± 10% at working and ± 5% at arbitration and metrological methodsmeasurements.

D. Measurement method

The essence of the thermogravimetric measurement method consists in the separation of the analyzed component (water) from the test sample by means of a certain thermal effect on it and measurement by direct weighing of the mass values of the test sample before and after separation.

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Thermogravimetric measurement method can be implemented:
during work control;
during arbitration control;
for metrological research and work (verification, calibration, calibration, etc.).
The essence of the working version of the implementation of the measurement method consists in the impact on the test sample at a temperature of 130 ° C for 40 minutes, *the arbitration* - at a temperature of 105 ° C until a constant value of the mass of the test sample and *metrological* - 80 ° C under vacuum at an absolute pressure of 10 KPa until a constant value of the mass of the test subject sample [1.5].

E. Requirements for measuring instruments, auxiliary devices, materials and solutions are indicated in Table 2.

Table 2
Technical equipment. (fragment)

Name	Metrological requirements	An object measurements
Laboratory balance any type	absolute weighing error: For the working version of the method 0.01 g For the arbitration variant of the method 0.001 g	
Any type of thermostat	Temperature (100-140) ^{o}C; stability ± 2 ° C	
Vacuum thermostat of any type	Operating temperature range (100-140) ^{o}C; stability ± 2 ° C,	During metrological work
Desiccator of any type	Capacity not less than 250 cm3	
Calcium chloride	Dehydrated	
River coarse sand	cleaned and calcined	
Glass or metal cuvette with a lid (any type)		
tinplatetrays	Area 120 cm2, height 1 cm	Cigar raw materials
	Area (120 ± 2) cm2, height (10 ± 2) cm	Smoking shag
Mesh bucks	Mesh size 1x1 mm, wall height (40 ± 3) mm	

F. Operations in preparation for taking measurements.
In preparation for taking measurementsit is done the following:
a). activate (if necessary) the sorbent and pour it into the desiccator.
b). the thermostat is heated up to a temperature of 130 ° C (with a working version of measurement) or 105 ° C (with an arbitration version of measurement).
c). place two cuvettes with lids in a desiccator for at least two hours.
d). take 2 samples of a sample weighing (5-6) g.
For analyzing viscous substances and materials which are difficult to mix (margarine, butter, etc.) and materials thatcan be bakedon dried (for example, some types of fish and fish products) and cleaned calcined river sand is used.
Samples are taken and prepared in accordance with the requirements given in Table 1, Interstate standard GOST 31904-2012.
7. Operations on performing measurements.
The following operations are performed for using the working version of the measurement method:

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a). Weigh each cuvette with a lid, empty and then with a sample, recording the measured values of mass **m** and $\mathbf{m_1}$, respectively, in grams to the third decimal place according to [95].
b). Open cuvettes with samples together with lids are placed in a thermostat at a temperature of 130 ± 1 °C for (40 ± 1) minutes.
c). The cuvettes with the samples covered with lids are placed for (20 ± 1) minutes in a desiccator, then they are weighed with a record of the measured value of the mass $\mathbf{m_2}$ in grams to the third decimal place.
For using *the arbitration* version of the measurement method, the following operations are performed:
a). Weigh each cuvette with a lid, empty and then with a sample, recording the measured values of the mass **m** and $\mathbf{m_1}$, respectively, in grams to the fourth decimal place.
b). Place open cuvettes with lids in a thermostat at a temperature of (105 ± 1) ^{o}C for 2 hours.
c). The cuvettes with samples are closed with lids and placed in a desiccator for (20 ± 1) minutes.
d). Weigh the closed cuvettes with the measured mass $\mathbf{m_3}$ in grams to the fourth decimal place.
e). Repeat heating according to item "b" for (30 ± 1) minutes and the subsequent procedures according to items "g" and "d" until two consecutively measured values of the mass differ by no more than 0.01 g.
Note: in all cases, the cuvettes should only be taken with tweezers (clamp), lint-free cloth or chamois leather.

G. Modes of drying.

fish samples (except for dried, dried and cold smoked) are dried for the first 2 hours at (60-80) ^{o}C, Samples of products with a fat mass fraction of more than 20% are dried for the first 2 hours at a temperature of (60-65) ^{o}C, and with a fat content above 40% (cod fish liver, etc.) - under these conditions in a stream of inert gas ..
cigar raw materials: normal and dry dried for 10 minutes at 105 °C and with high humidity and in case of disagreement - 40 minutes at (100-105) °C
tobacco raw materials 20 minutes at 105 °C and with disagreements 40 minutes at (100-105) °C
raw tobacco is dried for 10 minutes at 105 °C and with high humidity and in case of disagreement - 40 minutes at (100-105) °C
smoking shag (30 ± 1) min (108 ± 2) ^{o}C
tobacco, tobacco in cigarettes, cigars (180 ± 2) min (92 ± 2) ^{o}C and accelerated (30 ± 1) min (108 ± 2) ^{o}C
margarine: on a hotplate at (160-180) ^{o}C stirring continuously, avoiding splashing. Finish - in the absence of crackling and discoloration to light brown. Moisture is removed from the walls by drying in a cabinet at (100-105) ^{o}C. accelerated: the same, but the end - by the absence of fogging of the hour glass after the crackling stops and by the change in the color of margarine to light brown
canned milk: option - in paraffin (GOST 30305.1-95)
meat products: (150 ± 2) and (103 ± 2) ^{o}C
salt: (140-150) ^{o}C

V. CONCLUSION

The introduction of unified MM turns out to be difficult due to the existence of standards for product testing methods, which are perceived by both manufacturers and regulatory authorities as more "authoritative" than unified ones. At the same time, it is not taken into account that in most cases the former contain uncertified MM, and therefore, in accordance with the law "On Metrology" and O'zDSt(UzSSt) 8.016: 2002, they are illegal. MM becomes a commodity for its developer and he has an economic incentive to carry out certification (validation) of MM. The user has the opportunity to select MM, based on the required control accuracy of this parameter (quality indicator);
The unified methodology is the way to solve the problem of the unity of analytical measurements in the food industry. Among a number of significant features of the food industry, which impede the introduction into the theory and industry of the provisions of the uniformity of measurements, regulated by the Law of Uzbekistan "On Metrology", the dominant role belongs to the methods of measuring the composition at all stages of production: food raw materials, intermediate products and finished products. The elimination of this obstacle lies in the rejection of the differentiated method of creating a base of analytical methods for measuring the composition of food objects, which is adopted in practice, focused on certain groups (bread, pasta, oils, tobacco, etc.), and the transition to integration based on the physical essence of the measured property the analyzed component, which takes into account the influence of the matrix, but as a secondary influencing factor. The latter requires the unification of measurement objects into certain, possibly larger groups.

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