

PHYSICOCHEMICAL MEASUREMENTS

ESTIMATE OF PRECISION OF THERMOGRAVIMETRIC METHOD OF MEASURING MOISTURE CONTENT: ESTIMATE OF PRECISION AND EFFECTIVENESS GAINED WITH THE USE OF THE METHOD IN THE AGRO-INDUSTRIAL COMPLEX

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The problem presented by the limited practical application of methods and instruments for automatic monitoring of the moisture content of products of the agro-industrial complex is studied. The determination of the moisture content of the agro-industrial complex by means of the thermogravimetric method is considered. The effectiveness of weighing and selection of samples by accelerated and arbitration thermogravimetric methods is analyzed. The errors in the determination of the moisture content of products of by the thermogravimetric method are estimated. It is established that the error of the accelerated method is several times that of the error of the arbitration method. The need for an improvement in the existing thermogravimetric method of determining moisture content in terms of speed and in terms of precision is noted. The importance of selecting the optimal number of parameters of a subject of study to be determined that assures maximal measurement precision is demonstrated. The basic requirements that must be imposed on the design of instruments employed in monitoring moisture content are formulated on the basis of an analysis and estimate of the precision of the thermogravimetric method of measuring moisture content.

Keywords: moisture content, error, measurement precision, instrument, monitoring, thermogravimetric method.

Introduction. The practical application of methods and instruments for automatic monitoring of the composition and properties of substances in the agro-industrial complex is limited due to the heterogeneity of raw material and the diversity of the physicochemical properties of raw material.

Moisture content is in fact a principal technological parameter that influences the quality of the products of the agro-industrial complex and the degree to which these products satisfy the requirements of state standards and technological conditions. Moisture content determines the length of storage time and degree of preservation of the nutritional and technological properties of products of the agro-industrial complex as well as the price of a product when marketed. Rapid and precise measurement of the moisture content of a product is the most important economic and technological problem that has to be solved.

Laboratory methods of analysis are mainly employed today for monitoring the moisture content of granular materials produced by the agro-industrial complex. Delay in entering information on processed raw material and products of the agro-industrial complex sharply reduces the value of this information and narrows down the possibility of using this information for operational management of the production process.

Instruments of increased precision and reliability for use under conditions of corrosive media, high pressures and temperatures, etc. are needed by enterprises of the agro-industrial complex to determine the moisture content of products manufactured by these enterprises. Moreover, the rates of introduction of modern instruments of automation in enterprises of

the agro-industrial complex are low. It is, therefore, necessary to develop new high-precision proximate analysis methods of measurement of electrophysical parameters of materials for the purpose of determining moisture content.

The moisture content of materials is determined from their thermophysical and acoustic properties by means of radiometric, magneto-resonance, optical, electrophysical, and other types of methods [1–4]. Methods of analysis are broken down into direct and indirect. With the use of direct methods of measurements materials are divided into dry matter and moisture. The thermogravimetric method is the most common direct method of determining moisture content. In indirect methods, a quantity functionally related to the moisture content of the material, for example, conduction or the dielectric properties of the material, is measured. Despite the host of methods of determining the moisture content of solid and granular materials [5–7], only some of these methods are used in practical applications [8].

The thermogravimetric method is considered the basic method of determining moisture content adopted in normative documents for products produced in the agro-industrial complex (All-Russia State Standard GOST 10856-96, "Inter-State Standard. Oil-Bearing Seeds. A Method of Determining Moisture Content"). The method is based on drying substances in a drying kiln at a temperature of 105°C to constant mass.

The thermogravimetric method is considered to be the most precise method and is used as a base control method in state testing, appraisal, and calibration of instruments used in monitoring moisture content (hygrometers), the operation of which is based on other methods. Less precise accelerated forms of the thermogravimetric method in which the length of drying is reduced as a result of increasing the temperature to 195–220°C are also known (GOST 32198-2013, "Margarines, Fats for Comestibles, Pastry, Bakery, and Milk Industry. Rules for Acceptance and Inspection") are also known.

The objective of the present study is the present an analysis of the precision of the thermogravimetric method for the purpose of determining the moisture content of products manufactured in the agro-industrial complex and assessing the effectiveness achieved with the use of the method with the design of instruments used in monitoring moisture content in the agro-industrial complex.

We will consider solid, liquid, and dough-like products manufactured in the agro-industrial complex (grain, cotton seeds, cotton oil, margarine and similar products). Water present in these substances may have entered as a result of absorption on the surface and in pores, or in hydration of polar groups of macromolecules, and may also be found incorporated into the lattice of crystallohydrates of the mineral component [9].

Thermogravimetry method of analysis of moisture content. The process of monitoring moisture content by the thermogravimetric method is based on heating of the sample and weighing the residue obtained following evaporation of the moisture content. The content of moisture is inversely proportional to the dry mass of the substance.

There are several variant techniques for the measurement of moisture content by means of the thermogravimetric method [10].

Recommendations are given in the *Manual for the Analysis of Moisture Content* (<https://docplayer.ru/48695776-Analiz-produktov-pitaniya.html>) for the selection of methods and performance of studies, technological inspection, and calculation of output in the oils and fats industry where verified methods and procedures on the use of the thermogravimetric method of measuring moisture content are set forth.

The low level of representativeness of the sample, which is often taken from very large inhomogeneous masses of substances (weighing from a few tons to several hundred tons) is one of the problems encountered with the use of the thermogravimetric method in an agro-industrial complex. This feature is usually not taken into account when comparing the thermogravimetric method with indirect methods. Many instruments that are based on modern methods and that enable rapid assessment of the integral moisture content of large volumes of material, therefore, do not formally meet the requirements of state acceptance tests. Meanwhile, the determination of moisture content by means of the thermogravimetric method on many stages of the production of billets and processing of grain turns into a formal operation the results of which do not correspond to the true moisture content of the material, a situation that is in fact equivalent to the absence of every type of control and sometimes leads to worse conditions of the technological processes.

In actual practice, all methods of analysis of moisture content based on the principle of differential weighing may be referred to as thermogravimetric methods. Thermogravimetric analysis is used as a standard method and is described in many normative documents that regulate the grain processing industry.

A sample is weighed and heated in order to liberate all the moisture contained in it. Immediately after it is cooled in an exsiccator the sample is again weighed. The difference between the initial mass and the result found in the second weighing is used to calculate the content of moisture. In this procedure, the temperature and heating time are the important parameters. Reproducible and traceable results may be obtained only with identical conditions of analysis. For this reason, results that are obtained by alternative methods, for example, with the use of a moisture content analyzer, must be compared with the drying method employed in a drying kiln.

One of the drawbacks of the thermogravimetric method is the fact that, to one degree or another, many substances decompose in the course of heating, leading to additional loss of mass. In addition, errors in the course of processing the samples are possible along with errors in the calculations.

Variations that occur in the electrophysical parameters of the substances employed in a field of electromagnetic waves as functions of the moisture content and other technological parameters must be studied in order to improve high-precision hygrometry.

Features of multi-component substances, such as raw materials and the products manufactured by the agro-industrial complex, must be taken into account in the course of selecting the measurement method and designing instruments for monitoring moisture content.

Calculation of the error in the determination of the moisture content of products manufactured by the agro-industrial complex by the thermogravimetric method. Let us analyze possible errors in the determination of moisture content by the thermogravimetric method. If a series of measurements of some quantity l_1, l_2, \dots, l_n of equivalent precision is performed and there is no grounds for assigning a preference to any one of these measurements, then, according to a property of random errors, the arithmetic mean \bar{x} of the results of all the measurements must be adopted as the final value of the measured value [11]:

$$\bar{x} = (l_1 + l_2 + \dots + l_n)/n = [l]/n,$$

where the square brackets denotes the sum, as is done in the theory of errors.

We let X denote the true value of the measured quantity and calculate a series of corresponding absolute errors of the measurements:

$$\left. \begin{aligned} \Delta_1 &= X - l_1; \\ \Delta_2 &= X - l_2; \\ \Delta_n &= X - l_n. \end{aligned} \right\} \quad (1)$$

We add the right and left sides of Eq. (1) and obtain

$$[\Delta] = nX - [l], \quad X = [l]/n + [\Delta]/n. \quad (2)$$

From Eq. (2) it follows that where the number of measurements n increases to infinity, the arithmetic mean of all the measurements will equal to the true value of the measured quantity X .

Since in actual practice the number of measurements is limited, the arithmetic mean value of all the measurements differs from the true value of the measured quantity X . However, in any case for every n the arithmetic mean \bar{x} is adopted as a reliable estimate of the measured true quantity X [11].

In the thermogravimetric method, the quantity of moisture in a sample is established on the basis of the loss of mass as a result of weighing of the sample before and after drying. The quantity of moisture q in the sample may be related to the initial mass of the sample (to the mass q_1 of the moist substance) or to the ultimate mass of the sample (to the mass q_2 of the dry substance) and expressed in fractions or percentages. In this case, the moisture content W (mass fraction of moisture), or ratio of the mass of moisture to the mass of the moist substance, is used as a quantity characterizing the content of moisture in the sample:

$$W = q/q_1.$$

The total length of time needed to determine moisture content by means of the thermogravimetric method comprises the length of time needed for selecting and preparing the samples, delivering the samples to the laboratory from the acceptance point, warehouse, or shop, preparation of the samples for testing, drying, weighing, calculation of the moisture content, and transmission of information on the moisture content. The length of time needed for simple drying of the sample to

constant mass amounts to 6–10 h and that of accelerated drying, to 1.5–2 h. The simple thermogravimetric method and accelerated thermogravimetric method, therefore, are not suitable for quick testing and management of technological processes in an agro-industrial complex.

An analysis of published sources [12–14] together with improved designs of hygrometers that were developed on the basis of the present method confirm that the use of automatic samplers equipped with sensors that measure the mass of a sample significantly reduces the time it takes to measure the moisture content of a sample of a substance. Moreover, drying of a sample is performed by means of infrared radiation or in a high- or ultra-high frequency electric field and the result of a measurement of moisture content is arrived at by means of electronic computer instruments.

Through the use of improved methods it becomes possible to decrease the time it takes to measure moisture content, though because of their complexity, insufficient reliability, and the high cost of the instruments employed, the improved methods have not become sufficiently widespread. Moreover, the time needed for a single measurement by means of instruments that are used in the improved methods may be as much as 10–15 min. Therefore, unacceptable dynamic errors arise in the control systems with the use of these types of hygrometers as sensors of the automatic systems used to control the technological process. A further reduction in the length of time needed to dry the sample with an increase in the power of the heater is not possible, since this will lead to thermal decomposition of the material of the sample.

Conditions for the use of the thermogravimetric method recommended in GOST 32189-2013 must be especially observed in calibration of instruments for quick monitoring of moisture content, since the sampling error encountered in the analysis of samples is the principal source of error with the use of thermogravimetric methods. The magnitude of this error depends on the degree of heterogeneity of the material and may reach unacceptable levels, since the samples that are used in arbitrage and accelerated methods of determination weigh only a few grams.

Thus, thermogravimetric measurements of moisture content suffer from the following drawbacks.

1. The sampling error is determined by heterogeneity relative to the moisture content of the controlled substance and the mass of the sample. The sampling error is due to the fact the only some of the material is subjected to moisture monitoring, the volume of which is 10^{-6} – 10^{-8} times that of the volume of material the moisture content of which has to be determined. For example, the sampling error for such a solid dispersed material as raw cotton is close to 2%.

2. The drying rate depends on the temperature in the drying kiln, its gradient relative to volume, the water-sorption properties of the materials that are being monitored, and the temperature and moisture content of the environment.

3. The inaccuracy of weighing, which depends on the type of scale used, the temperature of the weighing bottle containing the material, as well as the temperature of the environment and the length of time the weighing bottle is held in the exsiccator, with the error of measurements of moisture content on the order of ± 0.2 abs.%.

The magnitude of some of the above errors may be reduced with the use of improved designs of drying ovens equipped with systems for removing moist air from the chamber, devices for rotating the weighing bottle in the space of the chamber, and built-in scales. However, the sampling error, which makes the principal contribution to the resultant measurement error, remains high. It does not seem possible to decrease the sampling error by increasing the weight of the sample in the thermogravimetric method, since this will lead to an unacceptable growth in the overall dimensions of the instrument or to the drying time due to the low heat capacity of the disperse material, to an uneconomical expenditure of electricity and of the volume of material subjected to analysis.

Thermogravimetric methods of measuring moisture content require the performance of time-consuming operations required in the selection and preparation of samples on the part of the personnel. Drying kilns that are permanently incorporated increase the air temperature in the laboratory, which creates difficult conditions for the staff, especially in summer time.

One way of decreasing the error is to increase the mass of the sample. This entails the creation of portable devices for monitoring moisture content to make it possible to monitor significant volumes of test material [15].

Thus, the present thermogravimetric method of analysis of the moisture content of the materials we are considering must be improved not only in terms of speed of performance but also in terms of degree of accuracy.

In designing measurement means of monitoring moisture content, it is essential to determine the list of standard metrological characteristics, including the basic error of reproduction of the measurement instrument relative to the nominal calibration characteristic, the limit of the permissible value of the basic error.

With the use of the high-frequency dielectric method for measuring the moisture content of substances, the lower limit of the measurements in instruments used to monitor moisture content may reach several tenths of a percent (cottonseed), with the upper limit reaching 30% (cottonseed oil), while for grain the average moisture content is in the range 15–30% [16]. The fundamental error must be in the range ± 0.10 pF and the random error in the range ± 0.05 pF.

Instruments for measurement of moisture content. Requirements imposed on the instruments. The fraction of granular friable material represents a significant volume of the raw material, semi-finished products, and finished goods in the grain-processing branch of industry. Automation of the agro-industrial complex requires the use of high-precision quick methods for monitoring the electrophysical properties of friable materials. These types of methods will have a decisive influence on the quality indicators of the final product and assure an appropriate effect with the introduction of modern systems of automatic control and management.

Many primary sources of information are used with automatic and automatized measurement of the technological control parameters of the process of monitoring the moisture content of bulk materials. These methods exert a decisive influence on the quality indicators of the final product and ensure an appropriate effect from the introduction of modern systems of automated control and management.

Many primary sources of information are used in automatic and automated measurement of the technological parameters of the process of monitoring the moisture content of friable materials. Often due to unreliable information, however, the results of these measurements exhibit great errors and cannot be used in the management of the technological process.

Instruments for monitoring moisture content that assure a required degree of precision, reliability, and degree of efficiency of the primary production and technological information needed for operation of automated systems of management of technological processes are used in the agro-industrial complex [17].

The design of instruments for monitoring the moisture content of grain and products of commercial processing of grain is based on deciding on a method of generating primary information as well as a method of converting the output signal of the sensing element into the output signal of the instrument. Selection and theoretical evaluation of the parameters of the circuit and design of the instrument are undertaken on the basis of calculations of the static and dynamic characteristics and the precision and reliability of the instrument.

Local measurements of the moisture content of solids are needed for scientific studies and the solution of practical problems, i.e., information is needed not about integral values of moisture content, but instead concerning the distribution of moisture content in the individual object and the moisture content at individual points of the particular medium under investigation.

Thus, two basic constituent elements of moisture content monitoring instruments should be identified: the sensor and the measuring device. By a sensor we understand the structural set of transformers and devices designed to introduce the test material into the transducer and mixing and discharge of the material, as well as additional devices for generating information about the values of external steps or for stabilization and compensation of these steps [18].

In deciding on which method of monitoring moisture content to employ [19], optimal selection of the number of measurable parameters of the subject of study to assure maximal precision of the particular quantity is of principal importance.

In general form, the function of converting moisture content into an output electrical signal I of the measuring device may be represented in the form

$$I = f(W, \rho, t, d, m, k, r, \dots),$$

where W , ρ , t , d , and m are the moisture content, density, temperature, thickness, and weight of the sample, respectively; k – concentration of electrolytes; and r – electrochemical criterion of the electrode/material boundary.

In automatic devices for monitoring moisture content, operation of the sensor and the measuring devices does not require human participation. In nonautomatic devices, operations that are needed to perform measurements or some component of these measurements (loading or unloading the sample, adjustment of the measuring device) is performed by the operator. These devices are generally designed for discrete steps [20].

From an analysis [21, 22] general requirements on the structural forms of sensors may be formulated. These include the stability of the transformation of the capacitance of the sensor $C = f(W)$ over time, i.e., the mechanical and temperature stability of the capacitance of the sensor; low overall dimensions and mass; resistance against corrosion; mechanical strength; and technological effectiveness.

For certain products manufactured by the agro-industrial complex, certain conditions besides those enumerated above must be satisfied, for example, in the analysis of liquids for flow-type transducers, minimum conditions imposed on the hydraulic resistance of the flowing liquid and of the mechanical strength of the sensors must be satisfied in order to maintain the pressure the flowing liquid constant.

The design of sensors must guarantee ease of cleaning and observance of the state of the sensors in the course of use, since pollution of the parts of the sensor that are in contact with the material being monitored is possible in the course of operation in most measuring devices. It is especially important to satisfy this condition when working with substances of high viscosity, which includes cottonseed oil and margarine.

The possibility of using of capacitance sensors without preliminary measuring off or weighing of the material being monitored is an important factor that must be considered in the design of capacitance sensors.

The use of microprocessors is an especially important means of increasing the efficiency of measuring devices. The use of microprocessors increases the precision of instruments, expands their functional capabilities, simplifies management of their operation, and increases their reliability and rate of response [23].

The use of microprocessors in the measuring devices that have been considered here expands their range of application, making it possible to correct the results of measurements of moisture content in light of non-informational parameters of the materials, obtain output information in different forms with the capacity to automatically adjust and periodically calibrate the measuring device incorporated into the instrument, automatically select the range of measurements, and automate the measurement process and processing of the results.

These drawbacks were eliminated in the automated ultra-high-frequency hygrometer proposed by the present author in [24].

Conclusion. The results of a determination of the moisture content of raw material and products of the agro-industrial complex by the thermogravimetric method are inexact. Due to the low mass of the samples, the use of the thermogravimetric method according to the standard technique in monitoring large volumes of material leads to errors greater than the prescribed error. Therefore, the results obtained in thermogravimetric determination of the mass fraction of the moisture content in products manufactured in the agro-industrial complex should not be used for calibration of devices used in proximate monitoring of moisture content. Different types of errors lead to a decrease in the reliability of primary information about the materials under investigation, which produces incorrect monitoring steps in the technological process, thereby inducing a reduction in the efficiency indicators of automated monitoring and management. Consequently, in view of the specific nature of the products manufactured by the agro-industrial complex, more precise methods of analysis and, correspondingly, new techniques for the design of devices for measurement of moisture content must be developed. An analysis of published data led us to formulate general requirements to impose on sensors and devices used in the measurement of moisture content, in particular, that will assure a required degree of precision, reliability, and efficiency of primary production and technological information as well as low dimensions, strength, technological ease, and stability of the capacitance of the sensors.

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