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UNCERTAINTY OF A MANOMETRIC METHOD FOR DETERMINING THE HUMIDITY OF FIBROUS MATERIALS

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Abstract

A manometric method for determining the moisture content of fibrous materials is considered. The scheme and principle of operation of the device. A formula is given for calculating the moisture content of a material. According to the obtained measurement model, the determination of the moisture content of materials is reduced to measuring the heating temperature, the initial and final values of the volumes of vessels and pressures in the vessels before and after their communication. It is emphasized that the two-chamber manometric method for determining the moisture content of materials is an indirect measurement. To determine the measurement result and its uncertainty, it is necessary to justify and choose one of the appropriate methods for determining the measurement results and assess their uncertainty – the linearization method or the reduction method. The main sources of uncertainty are identified. The pairwise correlation between the estimates of the input quantities and the verification of the condition for the insignificance of the remainder term in the expansion of the measurement model in the Taylor series are estimated. Formulas for determining the sensitivity coefficients of the total standard uncertainty to the standard uncertainties of input quantities are presented. Standard, summary and extended uncertainties are provided.

Keywords: uncertainty, gauge, humidity, fibrous material, bellows type vessel, temperature, pressure.

Introduction

One of the main parameters of fibrous materials, in particular raw cotton and cotton fiber, is their moisture content. The environment of numerous methods for determining moisture is the most common gravimetric [1]. The accuracy characteristic of the gravimetric [1] method for determining humidity, of other methods, too, is the "error". This does not comply with international requirements [2, 3].

Statement of the objectives of the article

Based on the results of our studies, we have identified the disadvantages of existing methods for determining the moisture content of fibrous materials. The accuracy characteristic of these methods for determining moisture is "error." This does not comply with international requirements [2, 3]. The elimination of these shortcomings by the development of a manometric method for determining the moisture content W of fibrous materials and estimation of the uncertainty of measurement results is the main goal of this work.

Statement of the main research material

One of the main parameters of fibrous materials, in particular raw cotton and cotton fiber, is their moisture content. The environment of numerous methods for determining moisture is the most common gravimetric [1]. The accuracy characteristic of the gravimetric [1] method for determining humidity, of other methods, too, is the "error". This does not comply with international requirements [2, 3].

In order to eliminate this drawback, we developed a manometric method [4-7], determining the moisture content W of fibrous materials and estimating the uncertainty of measurement results.

The block diagram of the device, based on the manometric method of measuring humidity, is presented in Figure 1. The device contains two sealed vessels 1 and 2, made in the form of bellows, one 1 of which is built into the other 2, having a common base and cover, a heating element 3, a temperature regulator 4, a heat-insulating casing 5, a differential pressure gauge made in the form of two transducers 6 and 7 pressures into an electric signal, summing blocks 8 and 9, a linear bellows-sized transducer 10 to an electric signal, multiplication blocks 11 to 14, a division block 15, a temperature to electric signal converter 16, and a control unit 17.

The device operates as follows.

A sample of fibrous material, for example, cotton material weighing 10^{-2} kg, is placed in vessel 1, and air is pumped out from it by compressing vessels 1 and 2 to a minimum volume V_0' with their subsequent sealing. After sealing, a certain amount of air remains in the vessel 1 with the sample, the mass of which

$$m_1 = \rho_1\left(V_0' - V_0\right), \quad (1)$$

where m_1 is the mass of air in the vessel 1 with a sample; ρ_1 is the air density under normal conditions; V_0' is the volume of the first (control) vessel after compression; V is volume of the sample (more precisely, the total volume of fibers in the sample).

Air mass m_2 in the second (control) vessel

$$m_2 = \rho_1 V_0', \quad (2)$$

Then make the expansion of blood vessels 1 and 2 to a value of V_0. Vessels 1 and 2 expand and, consequently, the pressure inside the chamber decreases, which creates favorable conditions for accelerating the evaporation process.

Then, heating is carried out until the moisture from the sample is completely evaporated at a constant drying temperature of the sample, for example, at $393\ K$. As the moisture evaporates from the sample, the volume of the bellows increases a second time due to the pressure of moisture vapor inside the chamber, thereby additionally creating conditions for intensive and complete evaporation of moisture from sample. The constancy of temperature with an accuracy of $\pm 2°$ is ensured by the heating element 3.

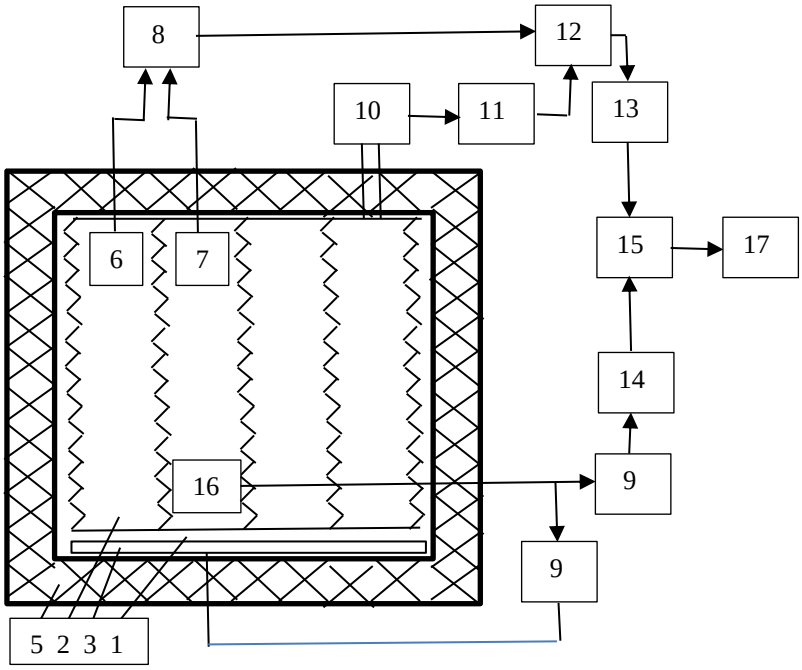

Fig1. The device diagram of the manometric method for determining the moisture content of fibrous materials

The latter is controlled by the temperature controller 4, and a temperature to electric signal converter 16 is connected to the input. The signals from the outputs of the transducers 6 and 7, proportional to the pressures in the vessels 2 and 1, respectively, are fed to the inputs of the first summing unit 8. In this case, the pressures P_1 and P_2 in vessels 1 and 2 according to the Dalton law and the Mendeleev – Clapeyron equation are determined by the expressions

$$P_1 = \left[\frac{m}{\mu} + \frac{m_1}{\mu_1}\right]\frac{RT}{V_0 - V}, \quad (3)$$

$$P_2 = \frac{m_2}{\mu_1}\cdot\frac{RT}{V_0}, \quad (4)$$

where μ is the molar mass of moisture (water); μ_1 is the molar mass of air; m is the mass of moisture evaporated from the sample; R is the universal gas constant; T is the absolute drying temperature.

By measuring the pressures P_1 and P_2 and the volume V_0 of the vessel after their expansion, the vessels communicate with each other. The volume occupied by water and air vapors increases and becomes equal to $2V_0$ - V, and the total pressure P_3 in the vessels is expressed by the formula

$$P_3 = \left[\frac{m}{\mu} + \frac{m_1 + m_2}{\mu_1}\right]\frac{RT}{V_0 - V}, \quad (5)$$

The magnitude of the signal at the inputs of block 8 summation is proportional to the pressure difference in the vessels. The signal from the output of the transducer 10, proportional to the height of the bellows, is fed to the input of the first multiplication block 11, into which the value of the base area of the vessel 1 is preliminarily entered. , the second input of which is connected to the output of the summing unit 8. The signal from the output of the multiplication block 12 does not receive a third multiplication block 13, into which the value of the molar mass of water multiplied by 100 is preliminarily entered, i.e. 100 μ. From the output of block 13, the signal is supplied to the first input of division block 15. The signal from the output of the temperature transducer 16, the value of which is proportional to the heating temperature, is fed to the input of the second summing unit 9, into which the figure 273,15 is previously entered (this is necessary to switch to the absolute temperature). From the output of block 9, the signal goes to the input of the fourth multiplication block 14, into which the mass of sample M, multiplied by the universal gas constant R, is preliminarily introduced, i.e. the value of MR. The signal from the output of the multiplication unit 14 is fed to the second input of the division unit 15, the output of which is fed to the input of the recording unit 17. At the output of the device (registration unit 17), the numerical value of the sample moisture in percent is recorded.

Solving (1)-(5) with respect to m/V and taking into account that $V=mc/\rho$, where mc and ρ are the mass and density of the sample after drying, we obtain the formula for calculating the moisture content W of the material as a percentage

$$W = \frac{100\mu}{\rho RT}\cdot\left[P_1\frac{V_0}{V_0} - P_1 - \frac{(P_3 - P_1)\cdot(P_2 - P_1)}{(P_3 - P_1) - (P_2 - P_1)}\right]. \quad (6)$$

Thus, the determination of the moisture content W of fibrous materials comes down to measuring the heating temperature, the initial and final values of the volumes of vessels and pressures in the vessels before and after their communication, and is expressed by the measurement model (6).

According to the international standard IS0/IEC 17025:2005 [3], the result of tests and|/or measurements must be presented, together with its accuracy characteristic, which is evaluated according to [2] "Guide to the expression of measurement uncertainty" (GUM:1993) Calculations of the

uncertainty of measurements require special knowledge in the field of mathematical statistics and the ability to work with statistical packages. These requirements sometimes create some difficulties, the way out of which is the automation of these calculations. The simplest implementation of the basic uncertainty estimation algorithm are programs developed in the Excel environment [8, 9].

The main sources of uncertainty in the manometric method for determining the moisture content W of fibrous materials, as follows from the functional dependence (6) of the measured quantity W on input quantities such as μ, ρ, R, T, P_1, P_2, P_3, V_0' and V_0.

Since the two-chamber manometric method for determining the moisture content W of fibrous materials, as follows from the measurement model (6), is indirect, to determine the measurement result and its accuracy characteristics, according to [10], it was necessary to justify and choose one of the corresponding methods for determining the measurement results and estimation of their uncertainty – a linearization method or a reduction method. For this purpose, we carried out multiple (n=5) measurements of input quantities, such as T, P_1, P_2, P_3, V_0' and V_0 (Table 1). The degrees of correlation between the values are estimated: P_1, P_2, and P_3; V_0' and V_0. Since the drying temperature T of the sample, the pressure in the first 1 vessel after its expansion P_1, the pressure in the second 2 vessel after its expansion P_2 and the total pressure P_3 in the vessels after their measurement were measured by different measuring instruments, at different times, etc., there is no need in conducting studies to evaluate the pairwise correlation between them. There was no need to evaluate the correlation between the molar mass μ of water, the density ρ of the sample after drying, the universal gas constant R, and other input quantities, since their values are tabular (standard.

The degrees of correlation between the measurement results of the input quantities xi and xj are determined by the well-known formula (7), and their significance is evaluated by the Student criterion (8)

$$r(x_i, x_j) = \frac{1}{n(n-1)} \cdot \frac{\sum_{k=1}^{n} \left(x_{ik} - \bar{x}_i\right)\left(x_{2j} - \bar{x}_j\right)}{u(\bar{x}_i) \cdot u(\bar{x}_j)}; \qquad (7)$$

$$t = \frac{\left|r(x_i, x_j)\right|\sqrt{n-2}}{\sqrt{1-r^2(x_i, x_j)}} < t_p(n-2), \qquad (8)$$

where x_i and x_j are, respectively, the i-th and j-th input quantities; $t_P(n\text{-}2) = 3{,}182$ is the student coefficient for the number of degrees of freedom (n-2) for the confidence level p, in particular p=0,95.

The fulfillment of condition (8) indicates the absence of a correlation between the results of the estimates of the above-mentioned values (Table 1). Since the verification of the condition of insignificance of the residual term when expanding function (6) in a Taylor series was confirmed, to estimate the indirectly measured quantity W and its accuracy characteristics, one can use the linearization method. The results are presented in table.1 and table 2.

The combined standard uncertainty $u_c(W)$ of the material moisture measurement result is estimated by the formula (9):

$$u_c(W) = \left[\left(\frac{\partial W}{\partial \mu}\right)^2 u^2(\mu) + \left(\frac{\partial W}{\partial \rho}\right)^2 u^2(\rho) + \left(\frac{\partial W}{\partial R}\right)^2 u^2(R) + \left(\frac{\partial W}{\partial T}\right)^2 u^2(T) + \left(\frac{\partial W}{\partial P_1}\right)^2 u^2(P_1) + \left(\frac{\partial W}{\partial P_2}\right)^2 u^2(P_2) + \left(\frac{\partial W}{\partial P_3}\right)^2 u^2(P_3) + \left(\frac{\partial W}{\partial V_0}\right)^2 u^2(V_0) + \left(\frac{\partial W}{\partial V_0^{'}}\right)^2 u^2(V_0^{'}) \right]^{0.5}, \qquad (9)$$

where $\frac{\partial W}{\partial \mu}, \frac{\partial W}{\partial \rho}, \frac{\partial W}{\partial R}, \frac{\partial W}{\partial T}, \frac{\partial W}{\partial P_1}, \frac{\partial W}{\partial P_2}, \frac{\partial W}{\partial P_3}, \frac{\partial W}{\partial V_0}, \frac{\partial W}{\partial V_0^{'}}$ are the coefficients the sensitivity of the combined standard uncertainty $u_c(W)$ to standard uncertainties in the estimates of μ, ρ, T, P_1, P_2, P_3, V_0' and V_0, the values of which (Table 2) are determined by equations (10) - (17):

Table 1

Measurement results and their accuracy characteristics (uncertainty)

i	T, K	P_1, Pa	P_2, Pa	P_3, Pa	V_0, m^3	V_0^I, m^3
1	400	5,40E+04	4,06E+04	1,37E+05	4,01E-03	1,59E-03
2	397	5,65E+04	3,08E+04	1,61E+05	5,14E-03	1,35E-03
3	396	4,93E+04	3,90E+04	1,24E+05	4,01E-03	1,27E-03
4	400	5,17E+04	3,98E+04	1,61E+05	5,01E-03	1,75E-03
5	397	5,94E+04	4,11E+04	1,47E+05	4,12E-03	1,42E-03
The average	398	5,42E+04	3,83E+04	1,46E+05	4,46E-03	1,48E-03
***W*, %**	11,1					
***u*$_A$(x), %**	8,37E-01	4,06E+03	2,99E+03	1,15E+04	3,88E-04	1,56E-04
***u*$_B$(x), %**	1,15	0,29	0,29	0,29	0,29	0,29
Sensitivity	0,028	0,000	0,000	0,000	2489,906	7520,325
Contribution,%	0,023	0,363	0,551	0,542	0,631	0,650
***r*(x_1,x_2)**	$r(P_1,P_2)$=0,433; $r(P_1,P_3)$=-0,681; $r(P_2,P_3)$=0,303; $r(V_0,V_0^I)$=0,682					
t	$t(P_1,P_2)$=1,593; $t(P_1,P_3)$=2,941; $t((P_2,P_3)$=1,004; $t(V_0,V_0^I)$=2,952					
***t*$_p$(n-2)**	3,182					
Relevance *r*(x_j,x_k)	$r(P_1,P_2)$, $r(P_1,P_3)$, $r(P_2,P_3)$, $r(V_0,V_0^I)$ - not significant					
***u*$_c$(W), %**	1,2					
***U*, %**	3,3					

$$\frac{\partial W}{\partial \mu}=\frac{100}{\rho RT}\cdot\left[P_1\frac{V_0}{V_0^{'}}-P_1-\frac{(P_3-P_1)\cdot(P_2-P_1)}{(P_3-P_1)-(P_2-P_1)}\right], \quad (10)$$

$$\frac{\partial W}{\partial \rho}=-\frac{100\mu}{\rho^2 RT}\cdot\left[P_1\frac{V_0}{V_0^{'}}-P_1-\frac{(P_3-P_1)\cdot(P_2-P_1)}{(P_3-P_1)-(P_2-P_1)}\right], \quad (11)$$

$$\frac{\partial W}{\partial {}^{'}V_0}=\frac{100\mu}{\rho RT}\cdot\frac{P_1}{V_0^{'}}, \quad (12)$$

$$\frac{\partial W}{\partial R}=-\frac{100\mu}{\rho R^2 T}\cdot\left[P_1\frac{V_0}{V_0^{'}}-P_1-\frac{(P_3-P_1)\cdot(P_2-P_1)}{(P_3-P_1)-(P_2-P_1)}\right], \quad (13)$$

$$\frac{\partial W}{\partial {}^{'}V_0^{'}}=-\frac{100\mu}{\rho RT}\cdot\frac{P_1\cdot V_0}{V_0^{'2}}, \quad (14)$$

$$\frac{\partial W}{\partial T}=-\frac{100\mu}{\rho RT^2}\cdot\left[P_1\frac{V_0}{V_0^{'}}-P_1-\frac{(P_3-P_1)\cdot(P_2-P_1)}{(P_3-P_1)-(P_2-P_1)}\right], \quad (15)$$

$$\frac{\partial W}{\partial P_1}=\frac{100\mu}{\rho RT}\left[\frac{V_0}{V_0^{'}}-1+\frac{(P_3-P_1)^2-(P_2-P_1)^2}{[(P_3-P_1)-(P_2-P_1)]^2}\right], \quad (16)$$

$$\frac{\partial W}{\partial P_2}=\frac{100\mu}{\rho RT}\left[\frac{2(P_3-P_1)(P_2-P_1)-(P_3-P_1)^2}{[(P_3-P_1)-(P_2-P_1)]^2}\right]. \quad (17)$$

Table 2

The sensitivity coefficients of the combined standard uncertainty $u_c(W)$

$\partial W/\partial\mu$, (mol/kg)%	$\partial W/\partial\rho$, (m^3/kg)%	$\partial W/\partial R$, (Kmol/J)%	$\partial W/\partial T$, % / K	$\partial W/\partial V_0$, % / m^3	$\partial W/\partial V_0^I$, % / m^3	$\partial W/\partial P_1$, % / Pa	$\partial W/\partial P_2$, % / Pa	$\partial W/\partial P_3$, % / Pa
6,16E+02	1,80E+00	1,33E+00	2,78E-2	2,82E+03	1,45E+01	2,69E-4	1,24E+06	1,07E-6

The expanded uncertainty U, as is known, is obtained by multiplying the combined standard uncertainty $u_c(W)$ of the output quantity by the coverage coefficient k:

$$U=k\cdot u_c(W). \quad (18)$$

To determine the coverage coefficient k, the effective degree of freedom ν_{eff} was estimated for the combined standard uncertainty $u_c(W)$ using the Welch – Satterthwaite formula [11]. As shown in [11], when $\nu_{eff}=n-1=4$, which corresponds to the measurement considered by us, the coverage coefficient is $k=2{,}746$. Therefore, according to (18), the expanded uncertainty U of measuring the moisture content W of fibrous materials, in particular of the carbon fiber, by the manometric method was 3,3%.

Thus, the total standard uncertainty of humidity measurement is not more than 1,5%. At the same time, the main contribution to the total standard uncertainty is to make the measurement uncertainty of volumes V_0 and V_0'.

Table analysis 1 shows a relatively small contribution to the uncertainty of T and $\underline{P}_1$ in the total standard uncertainty of humidity measurement. The contributions of the uncertainty of the estimates of P_2 and P_3 to the total standard uncertainty of humidity measurement are of the same order of magnitude 0,6-0,7%).

Conclusions

Summarizing the research results, it should be noted that the following main scientific results were obtained in the work:

1. Uncertainties of the measurement results of the manometric method for determining the moisture content of fibrous materials, not previously investigated;

2. For the first time, equations have been obtained for estimating the uncertainties of measurements of moisture in fibrous materials by the manometric method, and the uncertainties of these measurements have been estimated;

3. Standard uncertainties of input quantities are not correlated in pairs;

4. The use of the linearization method to determine the measurement result and estimate uncertainty is justified;

5. Comparison of the theoretical curve with experimental values shows that the correspondence can be considered satisfactory;

6. The data obtained by us are in good agreement with the results obtained by other authors.

Анотація

Розглянуто манометричний метод визначення вологості волокнистих матеріалів. Описано схема і принцип роботи пристрою. Наведено формулу для розрахунку вологості матеріалу. Згідно з отриманою моделлю вимірювання, визначення вологості матеріалів зводиться до вимірювання температури нагріву, початкових і кінцевих значень обсягів судин і тисків в судинах до і після їх спілкування. Підкреслюється, що двокамерний манометричний метод визначення вологості матеріалів є непрямими вимірюваннями. Для визначення результату вимірювання і його невизначеності необхідно обґрунтувати і вибрати один з підходящих методів визначення результатів вимірювань і оцінки їх невизначеності – метод лінеаризації або метод редукції. Виявлено основні джерела невизначеності. Оцінюється попарно кореляція між оцінками вхідних величин і перевіркою умови неістотності залишкового члена в розкладанні моделі вимірювання в ряд Тейлора. Наведено формули для визначення коефіцієнтів чутливості. Наведено стандартні, сумарні і розширені невизначеності.

Ключові слова: невизначеність, датчик, вологість, волокнистий матеріал, посудину сильфонного типу, температура, тиск.

Аннотация

Рассмотрен манометрический метод определения влажности волокнистых материалов. Описаны схема и принцип работы устройства. Приведена формула для расчета влажности материала. Согласно полученной модели измерения, определение влажности материалов сводится к измерению температуры нагрева, начальных и конечных значений объемов сосудов и давлений в сосудах до и после их сообщения. Подчеркивается, что двухкамерный манометрический метод определения влажности материалов является косвенным измерением. Для определения результата измерения и его неопределенности необходимо обосновать и выбрать один из подходящих методов определения результатов измерений и оценки

их неопределенности – метод линеаризации или метод редукции. Выявлены основные источники неопределенности. Оценивается попарная корреляция между оценками входных величин и проверкой условия несущественности остаточного члена в разложении модели измерения в ряд Тейлора. Приведены формулы для определения коэффициентов чувствительности. Приведены стандартные, сводные и расширенные неопределенности.

Ключевые слова: неопределенность, датчик, влажность, волокнистый материал, сосуд сильфонного типа, температура, давление.

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