Hardware-Software System for Measuring Thermophysical Characteristics of the Materials and Products

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Abstract

Accurate measurements the thermophysical characteristics of materials and products required in almost all modern fields of technology. The presented in article methods for measuring the thermophysical and thermal radiation characteristics can be applied not only for materials but for energy-efficient glasses and thin coatings, as well as methods for determining the thermophysical parameters of concrete mixtures over a long period of time and a wide range of temperatures. Proposed unified hardware-software measuring system that combine described methods for measuring thermophysical characteristics. The main characteristics which are necessary for carrying out measurements and metrological support are defined. Methods of analysis of metrological characteristics using working standards as well as indirect method are proposed for implementation in the system. An algorithm for determining the metrological characteristics of the measuring system is proposed. Presented the metrological studies results of the measuring system.

Keywords

thermophysical characteristics, measuring system, thermal conductivity, emissivity, heat flux

1. Introduction

Accurate measurements of the thermophysical characteristics of materials and products required in almost all modern fields of technology. Measuring information can be used in the priority areas of science and technology development such as: construction and energy, metallurgy and materials science, aviation and astronautics, electronics and mechanical engineering.

The main thermophysical characteristics are thermal conductivity, specific heat, thermal resistance. Various methods for measuring these characteristics have been developed, which can be classified according to the following general features:

- by the thermal state of the sample (or stage of the thermal process): stationary, which provide for the establishment of thermodynamic equilibrium in the system [1-4], and dynamic, which study the temperature-time dependence of the thermophysical properties of the sample material [5-7];
- by the method of finding the desired value: absolute (for example, by measuring the electric power consumed by the heater) [2,4] and differential (comparative);
- by the nature of measurements: direct, based on the measurement of heat flux, while for the calculation it is necessary to know only the geometric parameters of the sample, and indirect, based on the measurement of temperature [1,3,5];
- according to the shape of the sample (or its isothermal surfaces): flat, cylindrical and spherical.

The basic differential equations describe thermal phenomena only in the most general form, since they are derived taking into account the general laws of physics. In order to distinguish from the innumerable number of thermal processes the considered one and to give its full mathematical

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description, the conditions of unambiguity (boundary conditions) are formulated, which reflect the partial features of a concrete process. Conditions of unambiguity include:

- geometric conditions that characterize the shape and linear dimensions of the body in which the process takes place;
- physical conditions that characterize the properties of the environment and the body and the law of distribution of internal heat sources [8,9];
- initial (or temporal) conditions that formulate when considering non-stationary processes and characterize the distribution of temperatures in the body at the initial time;
- boundary conditions characterizing the interaction of the considered body with the environment.

A wide range of new materials, different in shape and structure, requiring comprehensive studies of their thermal characteristics in accordance with the above classification and variation of boundary conditions, requires the creation of various instruments and measuring systems [10]. Measuring instruments, in turn, require the development of specific methods of calibration and verification of research results. Construction of multi-stage calibration schemes is not always technically and economically viable. This is especially true in the field of non-destructive testing, when the decisive factor is not the measured thermophysical value, but its relationship with the characteristics that reflect the quality of the material or product.

Unification of measurements the ability to determine several defining thermal characteristics using a single intelligent information-measurement system, is a promising area of development.

Considering written above the next research tasks was formulated:

- Propose the methods for measuring thermophysical characteristics that can be unified in one measuring system;
- Define the main characteristics which are necessary for carrying out measurements and metrological support;
- Implement hardware-software unified system;
- Propose the methods for metrological support of the system.

2. System for measuring the thermophysical characteristics

One of the main thermophysical characteristics of modern materials is thermal conductivity or thermal resistance. The methods of thermal conductivity measuring for the particular group of materials should be chosen depending from the following criteria:

- a suitable range of values of the coefficient of thermal conductivity, as for low- and high-conductivity materials require different measurement methods;
- range of operating temperature values;
- the corresponding standard size and shape of the sample.

As a result of the analysis of schemes of measurement of thermal conductivity of the materials in the form of a plate [1,3,11], in the developed complex information-measuring system the thermometric method with use of two identical heat flux sensors. In order to ensure a uniform thermal field in the sample in the measuring cell of the system, thermal stabilization of the heater edge zones and the cooler with the help of protective peripheral heaters is applied. It is this design and thermophysical solution of the measuring cell allows not only to minimize heat loss, but also provides the fastest access to a stationary mode of measurement [11].

Based on the measurements results of the heat flux passed through the sample and the temperature difference on its working surfaces, the thermal resistance is calculated R, m^2·K/W, and thermal conductivityλ (W/(m·K)) based on dependences presented in Table 1. The measurement range of the thermal conductivity from 0,02 up to 3 W/(m·K) in the temperature range from 240 up to 400 K, sample sizes from 100 × 100 to 300 × 300 mm with thickness up to 120 mm.

The Heat module of the measuring system during installing the sample for thermal conductivity measurements is shown in Fig. 1.

A special place among modern thermal insulators is occupied by thin-layer coatings, such as paints and mastics based on acrylic binders containing hollow ceramic or glass microspheres. Determination of their thermal resistance is usually carried out by the calculation method, based on data for ordinary paints, which is incorrect for complex inhomogeneous compositions. To objectively assess the effectiveness of such thermal insulation materials, a method for measuring the thermal resistance and thermal conductivity of thin coatings has been developed. A uniform layer of the studied paint is thickness $h_{\text{coat}} = 1...2$ mm applied to a substrate of rigid sheet dielectric material (for example glass), whose thermal resistance R_{gl} ($\text{m}^2\cdot\text{K/W}$) determined in advance. The coated sample is placed in the measuring cell of the system, and in the centers of the working surfaces of the sample are placed the joints of the tape differential thermocouple thickness 0,05 ... 0,07 mm and provide their pressing to the surface of the sample with thin elastic silicone gaskets, which eliminates possible micro-irregularities of the coating surface. Based on measurements of the heat flux and temperature the calculation of the thermal conductivity value for the investigated coating performed.

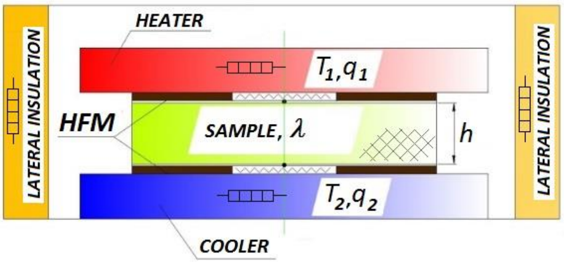
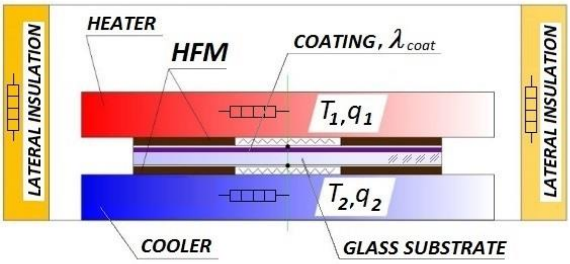
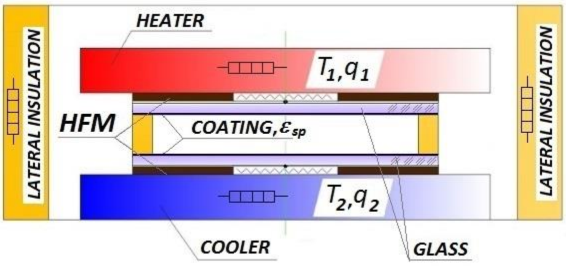
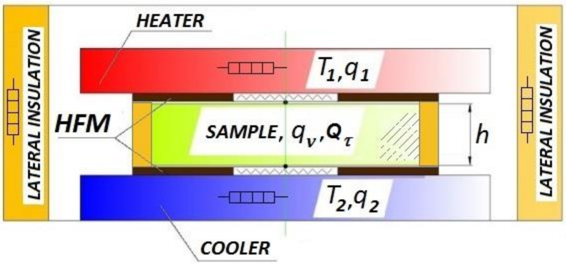
Figure 1: Heat module of the system during sample installation

Heat-protective properties of thin coatings are characterized not only by the thermal conductivity, but also the thermal radiation characteristics. The emissivity which is determined using a specialized expensive device - Fourier spectrophotometer [13]. For unify the measurements, an experimental and computational method for measuring the emissivity of energy-saving glass and other coatings proposed calorimetric method. This method requires two identical samples of glass or coated material are collected in a package having a layer of air of known thickness between the two test surfaces. This package is placed in the measuring cell of the system and determine its thermal resistance, which depends on the conductive-convective and radiative heat transfer in the package. Based on the obtained results the emission of the coatings is determined by calculation (see Table 1) [11, 14].

The scheme of the study and calculation formulas presented in Table 1.

Table 1

Measurement schemes and main data for determining thermal properties

Sought quantity, measurement scheme	Design equation	Data characteristics
Thermal conductivity λ, thermal resistance R of material 	$\lambda = h \cdot \bar{q}/\Delta T$ $R = \Delta T/\bar{q}$	$\bar{q} = (q_1 + q_2)/2$ – average heat flux recorded by HFM$_{1,2}$ $\Delta T = T_1 - T_2$ – temperature difference on the sample surfaces h – sample thickness
Thermal conductivity λ, thermal resistance R of a thin coating 	$\lambda_{coat} = \dfrac{h_{\text{coat}}}{R_{\text{coat}} - R_{\text{gl}}}$ $R_{\text{coat}} = \Delta T/\bar{q}$	h_{coat} – coating thickness R_{coat}, R_{gl}, R_{air} – thermal resistance of coating, glass and air layer
Emissivity ε_{sp} of thin coating 	$\varepsilon_{\text{sp}} = R_{rad}/4\sigma\bar{T}^3$ $R_{rad} = \dfrac{1}{R - 2R_{\text{gl}}} - R_{air}$ $R = \Delta T/\bar{q}$	$\bar{T}$ – average sample temperature σ – Stefan-Boltzmann constant
Volumetric q_v and integral Q_τ heat release of thermolabile materials 	$q_v = \Delta q/h$ $Q_\tau = \int_{\tau_0}^{\tau} q_v \, d\tau$	$\Delta q = \lvert q_1 + q_2 \rvert$ – heat flux recorded by HFM$_{1,2}$

Particular interest for modern technologies of monolithic frame construction is the study of the process of hydration of concrete mixtures and forecasting the strength and durability of building structures. Design of the information-measuring system, as well as technical characteristics allows to study the thermophysical parameters of thermolabile substances during long-time range (up to several days). In one experiment, it is possible to measure the current values of heat output during hydration of binders or concrete and their thermal conductivity for 72 hours, as required by the relevant standards, in the range of temperature values, which is of greatest interest in these studies. A special cuvette with a prepared sample of concrete mixture is inserted into the measuring cell of the system and the intensity of its heat release during hydration is recorded according to the signals of heat flux sensors (see Table 1) [11]. The result of calorimetric analysis is to obtain thermokinetic information, which allows to give recommendations on the composition of concrete and conclude the need to adjust the composition in a certain direction, the favorable modes of hardening of concrete, taking into account the influence of technological factors. From the analysis Table. 1, the main information parameters when measuring several thermophysical characteristics are the values of heat flux and temperature, as well as the geometric parameters of the sample.

3. Hardware-software modules of the system

The hardware part of the system consists from modules for setting and maintaining measurement modes and modules for recording primary data [14].

The module for setting and maintaining measurement mode consists from four units that perform the functions of setting and monitoring the thermal modes of the system.

The unit for setting and maintaining the reference temperature is implemented using a temperature regulator of the reference junctions of thermocouples, which controls the thermostat device of the reference junctions to set a stable value of the temperature of the thermocouples of the reference junctions of all measuring thermocouples.

The unit for setting and maintaining the temperature of the heater is implemented using a temperature regulator that controls the electric heater for setting the temperature depending on the operating mode.

The units for setting and maintaining the temperature of the cooler and the side insulation have separate temperature regulator, which respectively controls the mode of operation of the cooler and the side insulation.

The module of registration of primary data consists of units for registration temperature and heat fluxes. These units receive signals from analog temperature sensors and heat flux sensors, which are built into the heat and cooler units. ADC modules with a bit rate of 16 bits and a conversion frequency of 10 Hz were used for these tasks.

The software part of the system can be divided into a module for control system of the system and a module for processing measuring information [15,16].

The Module for control system of the system operation provides system settings, general control of operating modes, system settings also it responds for displaying measurement information using 2D graphs and tables. Also, the unit for control of the system operation connected with pre-censorship unit in receiving and processing measurement information. In the case of an error, the control module decides to send an additional request to the ADC modules or stop the measurement process and provides the relevant information to the operator.

The module of processing of the measuring information consists of units for pre-censorship, processing of the measured data, and data storage. This module also includes a unit for analysis of metrological characteristics that can be used for calibration and recalibration of the system according to the main indicators.

The pre-censorship unit of measurement data is designed to avoid cases of storage of data with errors, such as those arising from the transmission of information and the exclusion of measurement data with excessive errors.

The measured data processing unit converts the signals received from the ADC using polynomials into temperature (K) and heat fluxes (W / m^2). A separate polynomial is defined for each channel. In the future, data processing is carried out in accordance with the formulas given in Table 1.

The data storage unit stores the processed thermophysical characteristics and the values of the signals used for processing. This allows if necessary to conduct post signal processing after recalibration of the system.

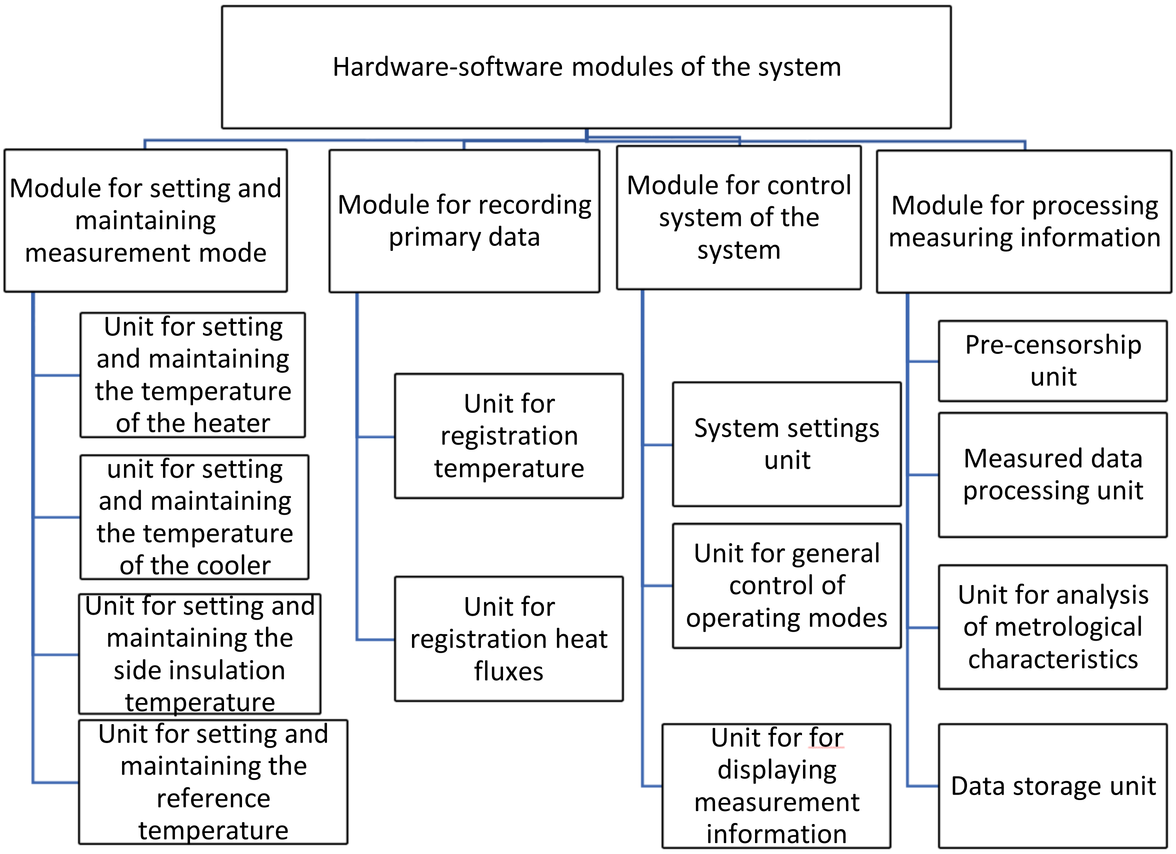


Figure 2: The structure of Hardware- software system modules

The metrological characteristics analysis unit can be used for verification of the calibration. For this task unit able work with methods based on using reference materials and working standards as well as an indirect method.

4. Determination the metrological characteristics of the system

4.1. Technique for determining metrological characteristics using reference materials and working standards

In practice, working standards act as a means of maintaining the uniformity of measurements of product properties. One of the most important advantages of their use is the ability to calibrate the working device in conditions close to operational ones, and to verify it directly at the operation site [3]. The working standard acts as a material carrier of the corresponding units, its main metrological characteristics are the values of the physical quantity and its error.

According to a standardized technique [1-3], the process is carried out by comparing the thermal conductivity measured value of the reference standard with the value indicated in its certificate or corresponding regulatory document. As thermal conductivity reference materials Polymethyl methacrylate, optical glasses LK5 and KV6, High Density Expanded Polystyrene are used. Error of the thermal conductivity working standard have to be no more than ± 3%, which must be confirmed by the appropriate certificate.

Thermal conductivity measurement range of the system is determined in experimental studies of the basic relative error at one fixed sample temperature from the values range close to room temperature using standard thermal conductivity measures and a control sample from granite.

To determine the basic relative error of the thermal conductivity measuring and establish its boundaries, experiments are performed at three temperature points of the working temperature range corresponding to its beginning, middle and end using one thermal conductivity working standard – optical glass LK5, because its thermal conductivity lies in the middle part measuring range. The list of materials – working standards of thermal conductivity and their characteristics at certain points of the temperature range are given in Table 2.

Table 2

Reference standards characteristics at fixed temperature points for metrological certification

Reference standard	Sample temperature T, K	Thermal conductivity λ_R, W/(m·K)
Polystyrene EPS HD	290 ± 3	0,047 ± 0,0014
Polymethyl methacrylate	290 ± 3	0,195 ± 0,0059
Optical glass LK5	290 ± 3	1,165 ± 0,0350
Optical glass KV6	290 ± 3	1,350 ± 0,0405
Granite	290 ± 3	2,907 ± 0,0872
Optical glass LK5	250 ± 5	1,094 ± 0,0328
Optical glass LK5	320 ± 5	1,214 ± 0,0364
Optical glass LK5	390 ± 5	1,317 ± 0,0395

In the process of the metrological characteristics studying the reference or control sample is placed in the system thermal block measuring cell. After establishing a stationary temperature regime, a series of measurements the sample's thermal conductivity values is carried out. As a result of measurement data processing in each temperature mode the basic relative measurement error limit is ± 6%, since it includes the thermal conductivity working standards error ± 3% [11].

So, the metrological characteristics of the system evaluation by the comparison method need to have several working standards, their characteristics have to cover the range of the desired thermophysical quantity and be confirmed by studies on the standard device. In this case, the error value of the working measuring instruments certified with their help includes the error of the working standard and, based on the example of the thermal conductivity study devices, it is twice the normalized in [1, 3]. To eliminate these inconsistencies and simplify research, a technique for determining the metrological characteristics of a measuring system by an indirect method has been developed.

4.2. Technique for determining metrological characteristics by indirect method

The measurement system calibration is carried out according to the technique [20]. The individual static transfer functions each of two HFM are determined by the method of two measurements using a special certification electric heater. Individual calibration of primary thermocouples is carried out using a reference resistive temperature transducer Pt100 – a 3rd category working standard in the temperature range from 220 K to 430 K.

The information-measuring system metrological characteristics determination is reduced to the evaluation the range and the main relative measurement error of the desired value and the range of the operating temperature. Taking into account that $\lambda = f\,(T, q)$, $R = f\,(T, q, G_1 \dots G_n)$, $\varepsilon = f\,(T, q, G_1 \dots G_n)$, where $G_1 \dots G_n$ are geometric parameters of the sample, this procedure can be implemented according to the indirect measurements method by evaluating the measurement error components of the parameters used in determining the thermophysical characteristics: heat flux density q, temperature values T, as well as geometric parameters of the sample $G_1 \dots G_n$.

As a basis the definition of thermal conductivity metrological characteristics can be accepted [18, 19]. In this case, according to ISO [1, 3], are determined:

- the standard deviation of the measurement result error (RMSD);
- non-excluded systematic error (NSE);
- standard measurement uncertainties.

The total error of measuring the thermal conductivity in the developed system characterized by the RMSD and NSE was determined by the computational and experimental method for studying individual errors in measuring basic physical quantities. Components of the error in measuring the heat flux density and the temperature values difference were determined experimentally using a special certification electric heater and a reference resistive temperature transducer, respectively [20]. Standard measuring instruments, such as digital multimeters Picotest M3500A and Fluke 8845A, reference electrical resistance coil, DC voltage regulator and a digital caliper, are also used [22–25].

The measuring system metrological characteristics determination is carried out according to the developed algorithm (Figure 3).

The experiments are performed in 10 consecutive thermal regimes in the process of investigating the components of the error in measuring the heat flux density. The first five modes are carried out at one constant temperature value, but at different total heat flux values set by the calibration heater, which correspond to 5%, 25%, 50%, 75% and 95% of the heat flux density measurement range. The following five modes are carried out at five points of the measuring system operating temperature range corresponding to 5%, 25%, 50%, 75% and 95% of its value, but at one constant heat flux density value equal to the middle of its operating range. In each mode, the temperature of the heater, cooler and lateral isolation is set identical by the appropriate regulators.

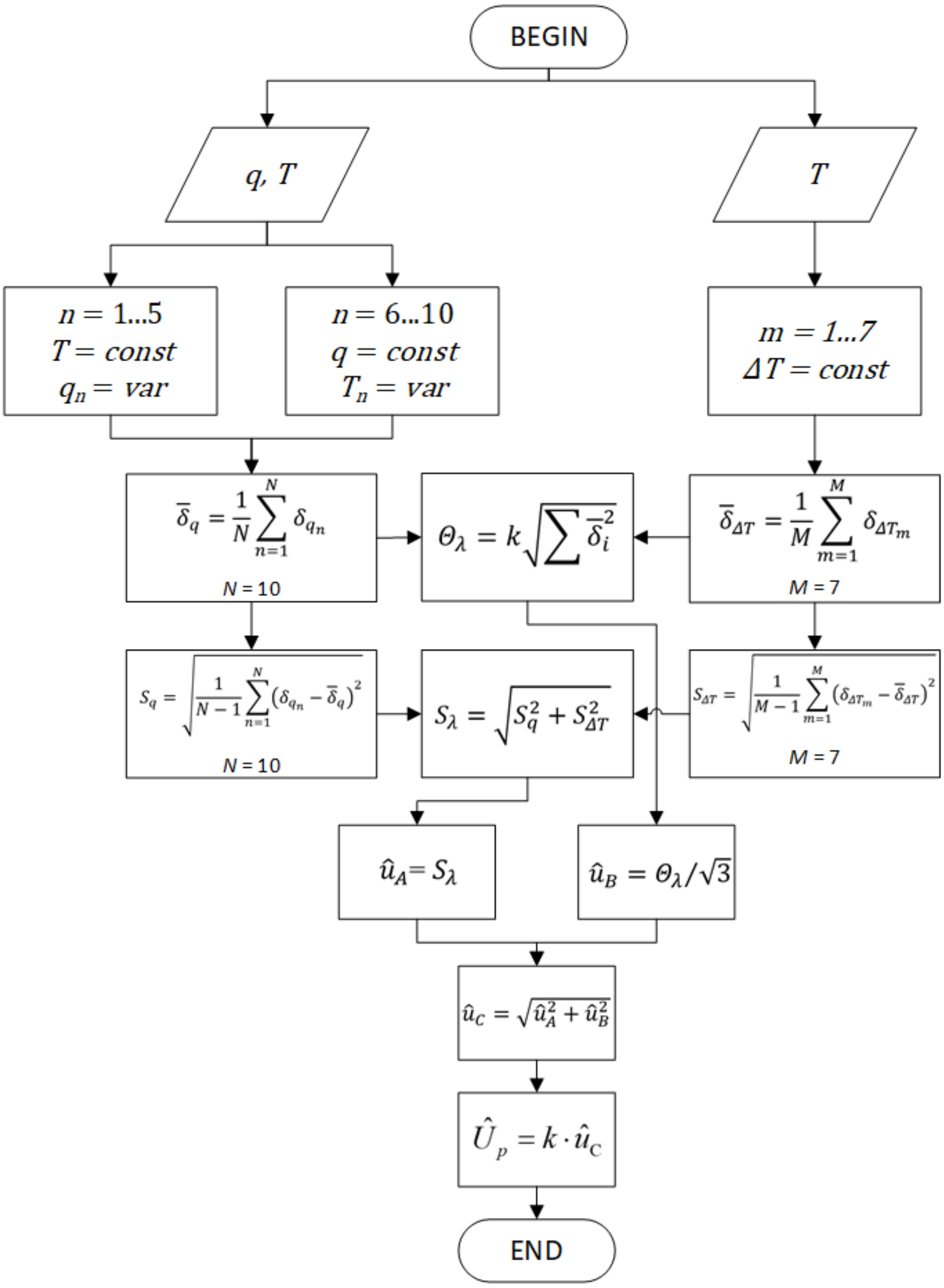


Figure 3: Algorithm for determining the metrological characteristics of a measuring system

In each n-mode after reaching a thermal steady state the system software records and averages data from the heat flux sensors and the electric heater during 20 minutes minimum. The NSE component of the heat flux density measurement θ_q is taken equal to the value of the series of measurements relative error $\overline{\delta}_q$:

$$\theta_q = \overline{\delta}_q, \tag{1}$$

were $\overline{\delta}_q$ − the relative error of the series of measurements in 10 modes, calculated by formula:

$$\overline{\delta}_q = \frac{1}{N}\sum_{n=1}^{N} \delta_{q_n}, N = 10 \tag{2}$$

The root-mean-square deviation (RMSD) of the heat flux density measuring results S_q is estimated according to the measurement data in 10 modes by formula:

$$S_q = \sqrt{\frac{1}{N-1}\sum_{n=1}^{N}\left(\delta_{q_n} - \overline{\delta}_q\right)^2} \tag{3}$$

The components of the temperature difference measurement error are investigated in seven consecutive temperature modes of the operating temperature range. In each m-mode, the heater, cooler and lateral isolation regulators are simultaneously set to the same beginning and ending temperatures, wherein $\Delta T = 10 \pm 2$ K.

In each m-mode, the stationary state is monitored according to HFM data, the parameters for regulating the heat flux density are not more than 2 W/m^2, the set temperature values permissible fluctuations have to be no more than ±0,5 K.

After reaching a thermal steady state the system software records and averages data from the temperature sensors and reference resistive temperature transducer during 20 minutes minimum. The NSE component of the temperature difference measurement $\theta_{\Delta T}$ is taken equal to the value of the series of measurements relative error $\overline{\delta}_{\Delta T}$:

$$\theta_{\Delta T} = \overline{\delta}_{\Delta T}, \tag{4}$$

were $\overline{\delta}_{\Delta T}$ − the relative error of the series of measurements in 7 modes, calculated by formula:

$$\overline{\delta}_{\Delta T} = \frac{1}{M}\sum_{m=1}^{M} \delta_{\Delta T_m}, M = 7\,. \tag{5}$$

RMSD of the temperature difference measuring results $S_{\Delta T}$ is estimated according to the measurement data in 7 modes by formula:

$$S_{\Delta T} = \sqrt{\frac{1}{M-1}\sum_{m=1}^{M}\left(\delta_{\Delta T_n} - \overline{\delta}_{\Delta T}\right)^2} \tag{6}$$

NSE boundaries of the thermal conductivity measurement result Θ_λ are determined as a positive square root of the sum of squares of all components of the systematic error, in measuring the heat flux density and temperature difference, as well as the errors of all measuring devices calculated based on data taken from manufacturer's specifications and calibration certificates, by formula:

$$\Theta_\lambda = k\sqrt{\sum \overline{\delta}_i^2} \tag{7}$$

were k =1,96 – a coverage factor, which is chosen depending on the effective degrees of freedom at the level of confidence p = 0,95 (see Table G.1 in Annex G [17]).

RMSD of the thermal conductivity measuring S_λ is estimated based on the results obtained as the square root:

$$S_\lambda = \sqrt{S_q^2 + S_{\Delta T}^2}\,. \tag{8}$$

Assessment of measurement uncertainties (type A, B, total and extended) is carried out according to the standard technique [17]. The results are presented in Table 3.

Table 3
Metrological characteristics

Components	Designation	Value, %
Non-excluded systematic error:		
• heat flux density measurement	θ_q	0,32
• temperature difference measurement	$\theta_{\Delta T}$	0,06
• thermal conductivity measurement	Θ_λ	1,25
RMSD:		
• heat flux density measurement	S_q	0,72
• temperature difference measurement	$S_{\Delta T}$	0,63
• thermal conductivity measurement	S_λ	0,96
Type A evaluation of standard uncertainty	$\hat{u}_A$	**0,96**
Type B evaluation of standard uncertainty	$\hat{u}_B$	**0,72**
Combined standard uncertainty	$\hat{u}_C$	**1,20**
Expanded uncertainty (k = 1,96)	$\hat{U}_P$	**2,35**

These results demonstrate the validity of the developed technique with the requirements of modern standards [1, 3]. This technique allows to pass the measurements range of the desired value with the appropriate step in a wide range of temperatures. The software allows to set the required temperature parameters, assess the stationarity of a stable thermal regime, record the readings of all measuring sensors in a series of measurements, form a data bank, process information and switch to the next temperature regime.

5. Conclusions

Analyzed methods for measuring thermophysical and thermal radiation characteristics of materials, energy-efficient glass and thin coatings. Proposed method for measuring thermophysical characteristics of concrete mixtures in a long-time interval and a wide temperature range. The main characteristics which are necessary for carrying out measurements and metrological support are defined.

The hardware-software module of unified system for research of thermophysical characteristics based on proposed methods is suggested and implemented. The proposed software allows to set the required temperature parameters, assess the thermal regime, record the readings of all measuring sensors in a series of measurements, form a data bank, process information. Also, the proposed construction of heat module system meets requirements of the standard ISO 8301 [1].

Methods of analysis the metrological characteristics using working standards as well as indirect method are implemented in system. An algorithm for determining the metrological characteristics of a measuring system is proposed. Presented the metrological studies results of the measuring system.

The results of this study can be used to simplification measurements of thermal characteristics using a single intelligent information- measurement system. Also, the results of this study improve the data processing and exclude subjective factor during measurements.

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