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The Combined Relative Uncertainty of Measurement Results by Prototype Semi-Automated Calorimetric Chamber

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The paper presents an evaluation of the combined relative uncertainty of the result of direct step temperature measurements aimed at evaluation of the indirect measurements of the specific thermal capacity of the heat insulating concrete by means of a pair of resistive cable thermometers fitted with Pt100 temperature sensors and integrated into a computer-controlled calorimetric chamber. In particular, it is a proposal of evaluation of the overall relative uncertainty of the measurement of partial temperatures measured in equidistant time steps, in a relatively wider time interval. In practice, the uncertainty of the result of step temperature measurements is most often declared only by the instrument uncertainty specified by the manufacturer. The exact evaluation of the result of the measurements of thermal and temperature material parameters measured by the calorimetric comparison method is required by the fact that the investigated samples are made of newly designed non-tabulated building materials and that the measurements are made by a prototype device.

Keywords: Combined relative uncertainty, step temperature measurement, specific thermal capacity, semi-automated calorimetric chamber.

1. INTRODUCTION

The basic documents of the field "Methodology of evaluation of measurement results and their uncertainties" recommend principles of evaluation of measurement results using standard uncertainties [1], [2]. The document GUM (Guide to the Expression of Uncertainty in Measurement) deals with the uncertainty of the measurement result in general, but it does not address the uncertainties of results of measurement performed by electrical means of measurement. The IEC (International Electrotechnical Commission), the approach of which is formulated in [3], [4], focuses on the evaluation of the results of practical measurements and of instrumental uncertainties. Other scientific and research publications, such as [5], [6] are based on basic metrology documents. Calorimetric measurement methods are also being developed [7], [8]. The development of measurements by electrical devices has altered standard evaluation procedures and forced a change in the understanding of the accuracy of the presented results [9], [10]. Standard procedures are not always easy to implement and time-saving, so they are often neglected in practice, or they may be conventionally modified [11], [12].

The sources of uncertainties must be identified in a standard manner, absolute statistical uncertainties must be assessed by method A, the degrees of freedom of uncertainty of type B must be analyzed and absolute systematic uncertainties must be evaluated by method B. If the accuracy of the used measurement devices guaranteed by the manufacturer can be considered satisfactory, the infinite degrees of freedom of uncertainties of type B and as a result, the degree of doubtfulness about the reported data need not be included in the evaluation of systematic uncertainty.

In practice, when evaluating the effect of statistical uncertainty of the step temperature measurement, the effect of statistical uncertainty is mostly neglected, and the effect of systematic uncertainty is expressed by the data declared by the manufacturer. "Methodology of uncertainties" is nowadays very advanced, but it does not address in a standard manner the evaluation of the uncertainties of the results of measurements of time-temperature intervals. In principle, the reason for this is the fact that the measured physical magnitude is not represented only by the mean value of the result obtained from repeated measurements but the probability interval, in which this mean value is found. The

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step temperature measurement should also be repeated several *n*-times, with the observance of the same measurement conditions. However, exactly the setting of the same measurement conditions for repeated measurements in consecutive time steps is problematic from the experimental point of view; the comparison of corresponding repeatedly measured partial temperatures with a resolution of hundredths of centigrade is also questionable. In particular, the step measured partial temperatures cannot be "averaged" so that they represent a single mean temperature. While the values of statistical uncertainties evaluated by method A decrease with an increasing number of measurements, the values of systematic uncertainties evaluated by method B are not dependent on the number of measurements. It can be assumed that a statistically higher number of subsequent partial temperature measurements (at least for $n = 30$) can compensate for the statistical requirement of *n*-repetitions of measurements of partial temperature. In the proposal of evaluation of the result of the step-temperature measurement, a standard and very effective tool for evaluating and comparing the accuracy of adequate measurement results is preferred, specifically the use of relative uncertainties, since the mathematical expression of the Gaussian uncertainty propagation law is specifically for relative uncertainties much simpler than for absolute uncertainties.

The systematic uncertainty of direct temperature measurement is declared by the manufacturer by the relative systematic uncertainty of the thermometer for a certain temperature range. However, there are usually several partial systemic uncertainties participating in the overall uncertainty of the temperature measurement measured by a more complex thermometer. In the illustrated case, it is necessary to express and cohere the relative uncertainty of the temperature sensor, the uncertainty of dependence of the measured temperature on the change of the temperature of the investigated material, the uncertainty of the converter and the uncertainty of its linearity, specifically for each partial temperature. The set of systematic partial uncertainties of a given step temperature measurement can then be statistically processed and represented by a single information value, namely by the combined relative uncertainty.

At present, data loggers are commonly used to continuously record temperature changes over time and store data in an energy-independent electronic memory. This data can then be transferred to a computer via an RS232 interface, USB, Ethernet, or GSM modem using the appropriate adapter. The application of the presented combined uncertainty of the step temperature measurement is broad, covering all analogous situations in which the given temperature cannot be repeatedly measured under the given conditions in a standard way. This is not only the case of step temperature measurements in a prototype calorimetric chamber, but it also includes further step temperature measurements performed over a specified time interval, especially in technological operations, warehouses and museums or in meteorology.

Regarding the novelty of the presented problem in the "methodology of evaluation of measurement results and their uncertainties", there are many known and frequently used methods today; it is especially possible to classify direct and indirect methods, absolute and relative, compensatory, interpolation, restrictive, special, etc. It can be said that all these methods are based on the principle of statistically significant repetition of measurement of the studied physical quantity under comparable conditions, with the exception of the successive method. The successive method is relatively close to step measurement in the sense that the individual sub-measurements directly follow one another. The principle of the successive method lies above all in the numerical processing of the measured data, so the method is used in those cases where the measured values approximately form an arithmetic sequence (e.g., in the case of periodic time measurements, capillary constant measurements by the drop method, determination of the wavelength of the standing waves in the resonator, etc.). However, the measured temperatures during the heating of the substance (in the case of porous concrete) do not even create an arithmetical sequence; depending on the material and the heating intensity, the temperatures can fluctuate too much! Therefore, a new presented evaluation of the statistical uncertainty of the type A of continuous, step measurements is based on the principle of evaluating the partial relative uncertainties, not the absolute uncertainties. The calculation of the measurement uncertainty is generally estimated to be infinitive, i.e. it is always associated with a certain degree of uncertainty whose degree is closely related to the value of the measured magnitude and the conditions under which it was determined. In the case of step temperature measurements, the statistical uncertainty of type A is not negligible, namely within the complex uncertainty of type C.

2. METHOD OF MEASUREMENT AND MEASURING EQUIPMENT

The measurement by a comparative calorimetric method was performed in the prototype thermostatic semi-automated calorimetric chamber (Fig.1.) [13], [14] as a measurement of the investigated sample and then as a measurement of the comparative etalon under the same measurement conditions. The sample was placed between selected support layers of the thermally conductive and non-conductive materials, and it was continuously heated. The partial temperatures on the opposite walls of the sample were measured at a pre-set time interval (1-60 min) and in the pre-set time steps (1-30 s) by a pair of resistive cable thermometers Pt30 fitted with temperature sensors Pt100. Both thermometers were coupled with an A/D converter in order to convert the measured data and with a Programmable Logic Controller (PLC) in order to automate heating processes in real time. Close contact between the probe and the surface of the sample was ensured by a groove recessed into the sample surface according to the geometric dimensions of the temperature probe.

In this manner, it is possible to evaluate several material parameters: specific thermal capacity, specific volume capacity, specific thermal conductivity, temperature diffusivity factor, and specific heat-storage capacity. The paper illustrates the uncertainty of the result of the measurement of the specific thermal capacity of the selected thermal insulation concrete. Theoretically, it is a comparison

of the calorimetric equations of the investigated sample and the comparative etalon (16), including the evaluation of the combined relative uncertainty of the measurement result (17) to (20). Calculation of the mean value of the specific thermal capacity of the investigated sample is performed by means of a tabulated value of the specific thermal capacity of the comparative etalon, by weighing of both samples and by indirect measurements of the temperature differences between the opposite walls of the investigated sample and of the comparative etalon during the stationary heat flow. The uncertainty of the mean value of the specific thermal capacity of the investigated sample is evaluated according to the Gaussian uncertainty propagation law for weighing both samples and for indirect measurement of temperature differences between the opposite walls of the investigated sample and the comparative etalon. Relative uncertainties of temperature differences are evaluated indirectly by standard statistic methods according to the Gaussian uncertainty propagation law, by direct temperature measurements on the opposite wall of the samples.

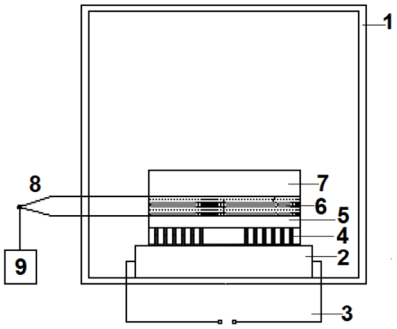

Fig.1. Diagram of the calorimetric chamber/device: 1 thermostatic container; 2 plate of heater; 3 electrical circuit; 4 heat exchanger; 5 support plates; 6 board of studied material; 7 support plates; 8 sensors; 9 timer and spreadsheet.

The determination of the systematic relative uncertainty of the measurement result using the method [15] is an integral part of the evaluation of the overall relative uncertainty of the final measurement result. Specifically, it is the evaluation of the relative uncertainties of the measurement results carried out during the verification of the identical dimensions of the investigated sample and of the comparative etalon, as well as of the relative uncertainties of settings of the same heating times of the investigated sample and of the comparative etalon, and of relative uncertainties of fluctuations of the measured temperatures as a result of the relatively more intensive heating. The spreadsheet processor comprises software for determination of the measurement results, including their respective uncertainties, which accelerates and refines more precisely the evaluation. However, it is also desirable to compare the result of the measurement of the investigated material parameter by the used method and the used measuring equipment, with the result of a comparable measurement carried out by another method and by another equipment. It is also desirable to compare the uncertainties declared by both measuring devices under comparable measurement conditions. In the illustrated case, a comparison of the measurement results and of their uncertainties was performed using a commercial instrument Isomet 2114.

Thermal transmittance in building materials is always dependent not only on the current state of the investigated material, i.e. on its temperature and humidity, but particularly on its properties, i.e. on the type of material (metallic or non-metallic), on the arrangement of the internal structure of the material, in the case of thermal insulating materials particularly on porosity and bulk density. The illustrated investigated sample is thermal insulation concrete, the proposed formula of which is based on cellular concrete and on a hydraulic binder with addition of waste aluminosilicate [16]. The cellular concrete - Ytong - was chosen as a comparative etalon because this building material exhibits, due to its homogeneous structure, the same properties in all directions, both static and thermal-technical. Material parameters of Ytong are tabulated by the manufacturer, under dry and room temperature. Considering the considerable thermal inertia of the core cooling of most building materials, the pauses between repetitive step temperature measurements of the same sample were chosen to be 24 hours.

3. Relative uncertainties of direct step temperature measurements on the sample opposite walls

At each step temperature measurement, the minimum limit of the number of measurements was exceeded in order to enable their relevant statistical evaluation, namely 30 partial measurements. In the illustrated case, altogether 150 partial temperature measurements were performed with an equidistant step of 30 s, i.e. from 23.04 °C to 49.81 °C for 75 minutes. The evaluated linear part of this interval contained 44 partial measurements from 23.34 °C to 25.37 °C recorded over 22 minutes. Fig.2. shows a linear part of the heating of the adjacent wall of the investigated sample for $n = 44$ partial temperatures t_{1B}, t_{2B}... t_{nB}, where $i = 1, 2,.., n$, namely the temperatures measured in equidistant time steps of 30 s.

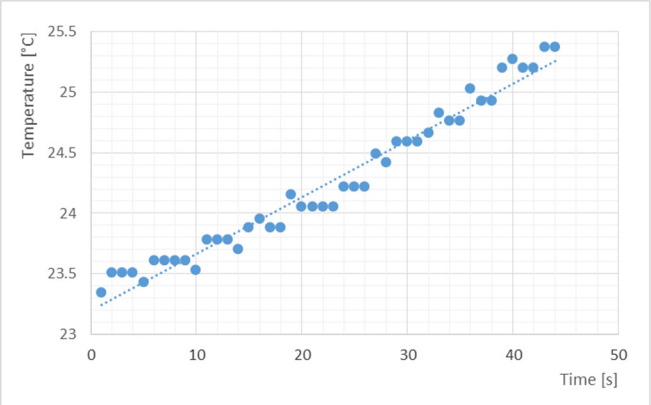

Fig.2. Step temperature measurement when heating the adjacent wall of the studied sample of heat insulating concrete.

The following systematic relative uncertainties were evaluated: the uncertainty of the temperature sensor, the uncertainty of its linearity, the uncertainty of the measurement card - A/D converter card, and the uncertainty

of temperature dependence due to the heating of the investigated material.

The maximum relative uncertainty of resistance of the Pt100 sensor z_{tBmax1} given by the manufacturer is 0.3 % for the calibration range of the sensor with the converter 0 °C to 150 °C, which means that the measured temperature interval entirely falls within this range. It is possible to calculate by direct proportion that the maximum absolute uncertainty of the read temperature $z_{tBmax1} = 0.45$ °C. The corresponding absolute uncertainty $u_{tB1} = 0.26$ °C is given by a uniform distribution according to (1)

$$u_{tB1} = \frac{z_{tB\max 1}}{\sqrt{3}} \tag{1}$$

and each corresponding relative uncertainty ρ_{tiB1} is related to the relevant partial temperature t_{iB} according to (2)

$$\rho_{tiB1} = \frac{u_{tB1}}{t_{iB}} \cdot 100\,\% \tag{2}$$

in the measured temperature interval the partial relative uncertainties ρ_{tiB1} drop from 1.1 % to 1.0 %. The maximum relative uncertainty of the measuring card - A/D converter z_{tBmax2} given by the manufacturer is 0.02 % of FSR (from the maximum value of the measuring range) ± 1 LSB (the value of the least significant bit, the position of which indicates in the binary number uneven or even parity of the number), i.e. $z_{tBmax2} = z_{tBmax2'} + z_{tBmax2''}$. It is possible to calculate by direct proportion that the maximum absolute uncertainty of the converted temperature of $z_{tBmax2'} = 0.030$ °C and according to (3) that $z_{tBmax2''} = 0.037$ °C

$$z_{tB\max 2''} = \frac{150}{2^{12}} \tag{3}$$

the maximum absolute uncertainty of the converted temperature $z_{tBmax2} = 0.067$ °C, with the corresponding absolute uncertainty $u_{tB2} = 0.039$ °C given by uniform distribution (4)

$$u_{tB2} = \frac{z_{tB\max 2}}{\sqrt{3}} \tag{4}$$

each partial relative uncertainty ρ_{tiB2} corresponding to it is related to the relevant partial temperature t_{iB} (5)

$$\rho_{tiB2} = \frac{u_{tB2}}{t_{iB}} \cdot 100\,\% \tag{5}$$

in the measured time temperature interval of the partial relative uncertainty ρ_{tiB2} of the converted temperatures decrease from 0.17 % to 0.15 %.

The maximum relative uncertainty of the converter linearity z_{tBmax3} given by the manufacturer is 0.2 % for the calibration range of the sensor with the converter 0 °C up to 150 °C. It is possible to calculate by direct proportion that the maximum absolute uncertainty of the linearly converted temperature $z_{tBmax3} = 0.30$ °C. The corresponding absolute uncertainty $u_{tB3} = 0.17$ °C is given by uniform distribution (6)

$$u_{tB3} = \frac{z_{tB\max 3}}{\sqrt{3}} \tag{6}$$

and each partial relative uncertainty ρ_{tiB3} corresponding to it is related to the relevant partial temperature t_{iB} (7)

$$\rho_{tiB3} = \frac{u_{tB3}}{t_{iB}} \cdot 100\,\% \tag{7}$$

in the measured time temperature interval of the partial relative uncertainty ρ_{tiB3} the linearly converted temperatures decrease from 0.73 % to 0.67 %.

As a result of the continuous heating of the investigated material, each partial temperature t_{iB} is measured as a temperature dependent, with the absolute uncertainty u_{tiB4} given by the manufacturer according to (8)

$$u_{tiB4} = 0.15 + 0.002 \cdot t_{iB} \tag{8}$$

and each relative partial uncertainty ρ_{tiB4} corresponding to it is related to the pertinent partial temperature t_{iB} according to (9)

$$\rho_{tiB4} = \frac{u_{tiB4}}{t_{iB}} \cdot 100\,\% \tag{9}$$

in the measured time-temperature interval the partial relative uncertainties ρ_{tiB4} decrease as a result of continuous heating from 0.84 % to 0.79 %.

For the evolution of the resulting partial relative systemic uncertainties ρ_{tiB} of the result of the temperature measurement of the wall of the investigated sample that is adjacent to the heater, in the given time-temperature interval, the Gaussian uncertainty propagation law applies (10)

$$\rho_{tiB} = \sqrt{\rho_{tiB1}^2 + \rho_{tiB2}^2 + \rho_{tiB3}^2 + \rho_{tiB4}^2};\ i = 1, 2, 3, ..., n \tag{10}$$

in the measured time-temperature interval, the resulting partial systematic relative uncertainties ρ_{tiB} drop from 1.58 % to 1.47 %.

Fig.3. shows evolution over time of the partial relative systematic uncertainties ρ_{tiB} of the results of direct temperature measurements on the adjacent wall of the investigated sample at a given time interval; the mean value of these uncertainties is expressed in red as their geometric mean. The geometric mean is suitably used for calculation of the average trend of change: growth/decrease in values. In principle, it is an expression specifying that the combined relative uncertainty would reach the value of the geometric mean if the rate of growth/decrease was constant.

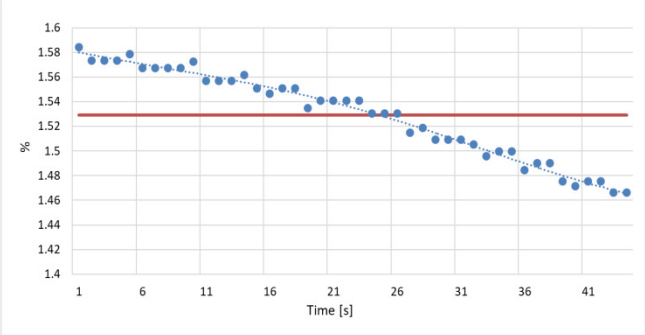

Fig.3. Time change of temperature relative uncertainties of sample wall (blue), their median value (red).

For the evaluation of partial relative systematic uncertainties $\rho_{tiB'}$ of the result of temperature measurements on the opposite wall of the investigated sample the relation applies analogically (11)

$$\rho_{tiB'} = \sqrt{\rho_{tiB'1}^2 + \rho_{tiB'2}^2 + \rho_{tiB'3}^2 + \rho_{tiB'4}^2};\ i = 1,2,3,...,n \quad (11)$$

For the evaluation of the partial relative systematic uncertainties ρ_{tiY} of the result of temperature measurements on the wall of the comparative etalon adjacent to the heater in the given time-temperature interval, the following relation applies according to the Gaussian uncertainty propagation law (12)

$$\rho_{tiY} = \sqrt{\rho_{tiY1}^2 + \rho_{tiY2}^2 + \rho_{tiY3}^2 + \rho_{tiY4}^2};\ i = 1,2,3,...,n \quad (12)$$

For the evaluation of the partial relative systematic uncertainties ρ_{tiY} of the result of temperature measurements on the opposite wall of the comparative etalon the relation (13) applies analogically

$$\rho_{tiY'} = \sqrt{\rho_{tiY'1}^2 + \rho_{tiY'2}^2 + \rho_{tiY'3}^2 + \rho_{tiY'4}^2};\ i = 1,2,3,...,n \quad (13)$$

For the evaluation of the combined relative uncertainty ρ_{tB}, which represents the result of measurement of the temperature of the adjacent wall of the investigated sample in the given time interval and analogically for the evaluation of the combined relative uncertainty ρ_{tB}, which represents the result of the temperature measurements on the opposite wall of the investigated sample at the same time, the relation (14) applies. The combined relative uncertainties in question are represented by the mean values (by geometric averages) of the sets of partial relative systematic uncertainties of the adjacent and opposite walls of the investigated sample.

$$\rho_{tB} = \frac{1}{n} \cdot \left(\prod_{i=1}^{n} \rho_{tiB} \right)^{\frac{1}{n}} \wedge \rho_{tB'} = \frac{1}{n} \cdot \left(\prod_{i=1}^{n} \rho_{tiB'} \right)^{\frac{1}{n}} \quad (14)$$

Analogically, the relation (15) for an evaluation of the combined relative uncertainty ρ_{tY}, which corresponds to the result of the temperature measurement of the adjacent wall of the comparative etalon at the same time-temperature interval, as well as to the combined relative uncertainty ρ_{tY}, which corresponds to the result of temperature measurements of the opposite wall of the comparative etalon in the same time-temperature interval

$$\rho_{tY} = \frac{1}{n} \cdot \left(\prod_{i=1}^{n} \rho_{tiY} \right)^{\frac{1}{n}} \wedge \rho_{tY'} = \frac{1}{n} \cdot \left(\prod_{i=1}^{n} \rho_{tiY'} \right)^{\frac{1}{n}} \quad (15)$$

To express and compare the accuracy of the performed evaluations, standardized statistical "relative uncertainties of relative uncertainties" for the results of step temperature measurements were also determined. In the measured time-temperature interval, the combined relative uncertainties of the results of the step temperature measurements of the investigated sample and the comparative etalon were comparable within an order: ρ_{tB} = 1.53 %, $\rho_{tB'}$ = 1.57 %; ρ_{tY} = 1.49 %, $\rho_{tY'}$ = 1.53 %. With respect to the usual recording of the value of relative uncertainty with the accuracy of a maximum of 2 valid digits, it can be stated that the combined relative uncertainties of the results of the step temperature measurements of the investigated sample and the comparative etalon were 1.5 % up to 1.6 %.

Relative uncertainties of relative uncertainties of the result of temperature measurement of the wall of the investigated sample and the comparative etalon adjacent to the heater were well comparable within an order: $\rho_{\rho tB}$ = 0.33 %; $\rho_{\rho tY}$ = 0.13 %. Relative uncertainties of relative uncertainties of the result of temperature measurement of the wall of the investigated sample and the comparative etalon opposite to the heater were evaluated with the highest accuracy: $\rho_{\rho tB}$ = 0.06 %; $\rho_{\rho tY'}$ = 0.04 %.

4. Relative uncertainties of the result of measurement of temperature differences

The relative uncertainty $\rho_{\Delta tB}$ of the result of measurement of the temperature difference between the opposite walls of the investigated sample and the relative uncertainty $\rho_{\Delta tY}$ of the result of measurement of the temperature difference between the opposite walls of the comparative etalon can again be evaluated according to the Gaussian uncertainty propagation law (16)

$$\rho_{\Delta tB} = \sqrt{\rho_{tB}^2 + \rho_{tB'}^2} \wedge \rho_{\Delta tY} = \sqrt{\rho_{tY}^2 + \rho_{tY'}^2} \quad (16)$$

In the measured time-temperature interval the following relative uncertainties of temperature differences were evaluated $\rho_{\Delta tB}$ = 2.2 % a $\rho_{\Delta tY}$ = 2.1 %.

5. Relative uncertainty of the result of indirect measurement of the specific thermal capacity of the investigated sample

The specific thermal capacity c_B of the investigated sample was evaluated on the basis of a comparison of the calorimetric equations (17) valid for the heating of the investigated sample and the comparative etalon

$$c_B = \frac{c_Y \cdot m_Y \cdot \Delta t_Y}{m_B \cdot \Delta t_B} \tag{17}$$

wherein c_Y is a tabulated value of the specific thermal capacity of the comparative etalon, m_Y is the weight of this etalon, Δt_Y is the difference of temperatures between its opposite walls; m_B is the weight of the investigated sample, Δt_B is the temperature difference between its opposite walls. In the illustrated case $c_B = 453$ J·kg^{-1}·K^{-1}, for the mean values of the measured variables: $m_Y = 1.01250$ kg, $m_B = 0.93825$ kg; for the constant temperature differences $\Delta t_Y = 0.69$ °C; $\Delta t_B = 1.64$ °C and for tabulated value c_Y=1 000 J·kg^{-1}·K^{-1}. In accordance with the Gaussian uncertainty propagation law, the pertinent relative uncertainty ρ_{CB} was evaluated (18)

$$\rho_{CB} = \sqrt{\rho_{mY}^2 + \rho_{mB}^2 + \rho_{\Delta tY}^2 + \rho_{\Delta tB}^2} \tag{18}$$

where the relative uncertainty of the weighing of the comparative etalon ρ_{mY} and the relative uncertainty of the weighing of the investigated sample were evaluated in a standard manner as relative combined uncertainties from the 30 partial weightings of both samples, from the uncertainty of the weight declared by the manufacturer and from the uncertainty of the calibration weight: $\rho_{mB} =$ 0.11 %, $\rho_{mY} = 0.098$ %; $\rho_{\Delta tB} = 2.2$ %, $\rho_{\Delta tY} = 2.1$ %. In the illustrated case, then $\rho_{CB} = 3.1$ %.

6. Relative uncertainty and used comparative calorimetric methods

The used calorimetric equipment uses the principle of an adiabatic system, in which the combustion heat is used for heating the internal volume and only the temperature change inside the instrument is measured. The core of the equipment is a proportional integration control system that guarantees continuous heating and maintaining temperature while optimizing energy consumption. Temperature measurements are continuously recorded to a memory card in a commercially programmable logic controller. The finished record of measurement results is downloaded, saved and converted to the Excel spreadsheet.

The times and intensities of heating both samples are the same, but the setting of these measurement parameters is reflected in the uncertainty of the comparative calorimetric method by means of relative uncertainties $\rho_{autoB} = 0.2$ %, $\rho_{autoY} = 0.2$ % of the result of time measurement of the pre-set PLC, i.e. with the use of the programmable logic controller ORBIT MERRET OMC 8000.

The equipment for data recording and display has a limited number of digits, i.e. decimal places, which means that less significant digits of the displayed number are not displayed anymore. Digital devices do not allow displaying a value with smaller “error” than the value corresponding to the smallest possible change of the least significant digit on display (± 1 digit). The error due to the resolution of the imaging unit can be in practice neglected.

The investigated sample and the comparative etalon were manufactured as samples of identical geometric dimensions (0.15 m, 0.15 m, 0.05 m). The geometric dimensions of the samples were verified by a sliding gauge. Three basic dimensions of both samples were measured 30 times at the same room temperature, with a negligible statistical relative uncertainty (in hundredths of a percent). The systemic uncertainty was evaluated on the basis of the uncertainty of the measuring instrument specified by the certificate (with a resolution of 0.05 mm for the measured range) and on the basis of the maximum possible personal error of the experimenter (with a resolution of 0.1 mm) and it was statistically processed under the assumption of a uniform rectangular distribution. As a relative uncertainty, ρ_M of the comparisons of the geometric dimensions of the samples, the combined relative uncertainty was evaluated and finally also the relative uncertainty corresponding to it (with the coefficient of expansion $k = 2$). In the illustrated case, $\rho_M = 0.18$ %.

Particularly in the case of intensive heating of thermal insulating materials, the continuously measured partial temperatures fluctuate not only stochastically, but the heated air in the pores is rapidly expanding primarily as a result of the effect of the porous structure of the material. Significant fluctuations of partial temperatures often lead to the fact that in practice, the time-temperature evolution is approximated in order to evaluate the uncertainty of the result of the step temperature measurement. The uncertainty of the measurement result is then evaluated from the difference between the corresponding measured and theoretically predicted values. However, approximation itself is a source of uncertainty, and therefore the evaluation of the uncertainty of results of direct temperature measurement cannot be based only on an approximation as an "etalon". The combined relative uncertainty of direct temperature measurement as a result of fluctuations was comparable for the walls of both samples because both samples were heated with the same control. For thermal insulating concrete, this uncertainty was slightly higher than for Ytong, for the walls adjacent to the heater this uncertainty was slightly higher than for the external walls. In the illustrated case, fluctuation uncertainty was evaluated to be $\rho_F = 0.1$ %.

The relative uncertainty ρ_{KM} of the comparative calorimetric method can be expressed according to the Gaussian uncertainty propagation law (19)

$$\rho_{KM} = \sqrt{\rho_{autoB}^2 + \rho_{autoY}^2 + \rho_M^2 + \rho_F^2} \tag{19}$$

In the illustrated case $\rho_{KM} = 0.35$ %.

7. Combined relative uncertainty of the result of measurement of specific thermal capacity

Evaluation of the combined relative uncertainty ρ_{CCB} of the result of indirect measurement of the specific thermal capacity of c_{cB} of the investigated thermal insulation concrete includes, according to the Gaussian uncertainty propagation law (20), also the relative uncertainty ρ_{CB} of the result of the measurement of the specific thermal capacity and the relative

uncertainty ρ_{KM} of the applied comparative calorimetric method

$$\rho_{CCB} = \sqrt{\rho_{CB}^2 + \rho_{KM}^2} \tag{20}$$

In the illustrated case, ρ_{CCB} = 3.08 %.

The final expanded combined relative uncertainty ρ_{RCCB} in relation to the combined relative uncertainty ρ_{CCB} can be defined by the expansion coefficient k. In technical practice, a convention is commonly used for a roughly estimated probability of coverage of the result based on the parallels with the standard Gaussian distribution. For a probability of coverage of approximately 95 %, an expansion coefficient k = 2 (21)

$$\rho_{RCCB} = k \cdot \rho_{CCB} \tag{21}$$

In the illustrated case ρ_{CCB} = 6.16 %. The specific thermal capacity is conventionally measured calorimetrically under the stationary heat flow relatively laboriously, but very accurately. At present, non-stationary pulse transient methods are used and developed. They are based in principle on the fact that the planar source of heat generates a thermal impulse and the thermal response to this thermal impulse is recorded by a thermocouple placed separately from the heat source, it is analyzed and adequately interpreted [17]. The commercial measuring instrument Isomet 2114 uses the same principle, i.e. a non-stationary method of measuring the surface of the investigated sample in order to evaluate its material parameters. Thermal impulses are transmitted onto the surface of the material, and the time dependence of the temperature response of the material is then measured. The measurement and evaluation of results performed by the Isomet are time-saving, the steps of measurement are fully automated, and they are manually relatively unexacting. A combination of 36 direct measurements was performed on the sample of the thermal insulating concrete, both on the material surface and inside the material (on the surface of the sample section), and then the mean values of the respective pairs of results were determined by indirect measurements. The results of the measurement of the specific thermal capacity by the given calorimetric chamber were compared with the results of measurements of the specific thermal capacity performed with the use of the commercial instrument Isomet 2114. Since the instrument Isomet 2114 declares only the mean values of the results of the measurements of the specific thermal capacity [18], the measured evaluated results had to be recalculated to the results that would be comparable by dimensions. In the illustrated case, the mean values of the specific thermal capacity varied within the order of 1 %. However, a higher relative uncertainty of the measurement result is declared by the manufacturer for evaluation of the material bulk thermal capacity, namely of 15 % of the measured range ($+10^3$ J·m^{-3}·K^{-1}), to assess the specific volume heat capacity of the material. Although the principle of the method of measurement of material parameters with the use of the Isomet is incomparable with the principle of measurement of material parameters by a comparative calorimetric method, the mean values of the measurement results can be compared in a satisfactory manner.

8. CONCLUSIONS

The paper presents a method for evaluation of the combined relative uncertainty of the result of measurements of the specific thermal capacity of a selected sample of thermal insulating concrete by a comparative calorimetric method carried out in the prototype semi-automated calorimetric chamber. The final result of the evaluation is 453 J·kg^{-1}·K^{-1} with a combined relative uncertainty of the measurement result of 6.2 %. Within the frame of evaluation of the overall relative uncertainty of the result of the measurement of the given material parameter, a conventionally acceptable method was proposed for evaluation of the combined relative uncertainty of the step temperature measurement performed on the opposing walls of the investigated sample and on the opposing walls of the comparative etalon. We proceeded during evaluation in particular by statistical processing of the partial relative systematic uncertainties of the results of the direct temperature measurements and consistently according to the Gaussian uncertainty propagation law.

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