Bulgarian Society for NDT
International Journal "NDT Days"
Volume 1, Issue 4, Year 2018 ISSN: 2603-4018

Calorimetric Quality Control of Heat Insulating Materials

Vitalii BABAK[1], Zinaida BUROVA[2], Oleg DEKUSHA[1]

[1] Institute of Engineering Thermophysics NAS of Ukraine, Kyiv, Ukraine
e-mail: vdoe@ukr.net
[2] National University of Life and Environmental Sciences of Ukraine, Kyiv, Ukraine
e-mail: zinaburova@nubip.edu.ua

Abstract
A set of devices for the heat insulating materials thermal conductivity and thermal resistance measuring using a probe express method for monitoring their quality in full-scale verification tests and a stationary plate method for certification tests is presented. The results of studies the most common types of modern heat-insulating materials of the world's leading manufacturers in a wide temperature range are analyzed.

Keywords: thermophysical properties, heat insulating materials, thermal conductivity, heat flow meter, stationary mode, probe express-method

1. Introduction

The problems of energy saving and resources economy are especially acute in the housing and communal services of Ukraine. At the same time, priority social tasks are the creation of optimal microclimatic conditions in the premises of buildings and structures by applying in the construction of modern heat insulating materials and thermo-modernization of the old housing stock. To address these challenges regulatory documents and requirements for mandatory energy certification of buildings have been developed in Ukraine. This led to the need for energy audit of the exploited residential, public and administrative buildings in order to accurately assess the state of the heat-protective qualities of fencing structures to decide on their further thermo-modernization.
In order to carry out the thermo-modernization and energy-saving technologies it is necessary to use high quality modern heat insulating materials first of all. The main efficiency indicator of materials or products, the main purpose of which is to reduce heat losses and increase energy efficiency of envelope building and structures, are their thermophysical characteristics: thermal resistance and effective thermal conductivity.
The heat insulators market in Ukraine is constantly expanding at the expense of both imported and domestic producers offering a wide range of products. It should be noted that materials with a thermal conductivity of 0,02 to 0,2 W/(m·K) are considered to be heat insulating.

2. Analysis of publications

There are many international normative documents regulating methods for determining the thermal conductivity of various building materials and heat insulators [1 – 3]. To assess the quality and certification of heat-insulating building materials and products according to their thermophysical characteristics, the leading experts consider it necessary to apply methods for measuring thermal conductivity in a stationary thermal mode [4, 5]. Using of dynamic (impulse) methods is not recommended since building materials and, in particular, effective heat insulation materials are not isotropic in their thermal properties and the determination the thermal conductivity of such materials by impulse methods is characterized by a significant systematic error, namely, the values obtained are overestimated. However, impulse methods have an advantage in the express obtaining final result and can be applied for a preliminary evaluation of the heat insulator quality.

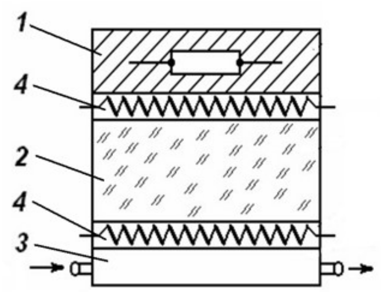
3. Purpose of the study

The aim of the work is the development, metrological certification and implementation a set of specialized devices for quantitative evaluation the heat insulating building materials quality by calorimetric methods in laboratory and in-situ conditions including non-destructive technique.

4. Apparatus and methods of research

4.1 Stationary plate method and device for the thermal conductivity research

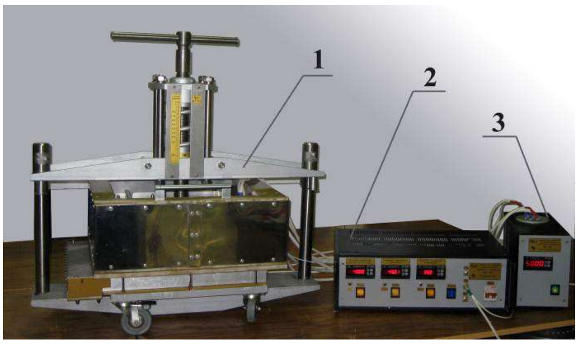
For certification tests the IT-7 type measuring devices [6] were developed in Institute of Engineering Thermophysics National Academy of Sciences (NAS) of Ukraine to measure the effective thermal conductivity and thermal resistance of a wide range of building and insulating materials in accordance with the applicable standards [2, 3]. In these devices a symmetrical scheme of the plate thermometric method is implemented [7] using two identical primary heat flow sensors [8]. A schematic chart of the method is shown in Figure 1.



1 – heater
2– test sample
3 – chiller
4 – heat flow meter (sensor)

Figure 1. The heat flow meter apparatus symmetric scheme with one sample [2, 3].

The essence of this calorimetric method is to create a stationary heat flow through a flat sample directed perpendicular to its facial (largest) surfaces. A heat flow generated by heater 1 and chiller 3 is one directed and pierces simultaneously the central zone of test sample 2 and the sensitive zone of two identical heat flow meters 4. The thermal conductivity coefficient is determined from the results of measurements the sample thickness, the temperature difference between its working surfaces and the heat flow density through the sample provided that it is unidirectional and uniform.
The appearance of the IT-7 device is shown in Figure 2.



1 – thermal unit
2 – electronic unit
3 – stabilisation thermostat

Figure 2. The IT-7 device for certification tests building and insulating materials

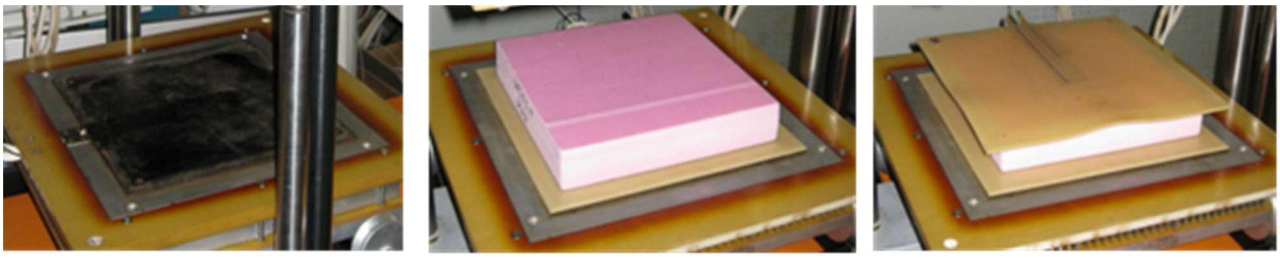
It consists of a thermal unit 1 in which a sample of the experimental material is placed and provides the necessary temperature and thermal conditions, functionally connected to the electronic unit 2, which contains regulators of thermal conditions and means for receiving and processing primary measurement information and transferring it to a personal computer for further processing, and a thermostat 3 to stabilize the reference junctions of the thermocouples. When conducting studies at a temperature below room temperature unit is placed in a climatic chamber.

Basic technical characteristics of the IT-7 device are:

- thermal conductivity coefficient measurements range is from 0,02 to 3,0 W/(m·K);
- main relative error is ± 3%;
- operating temperature from range is from – 40 to +180 ºC;
- sample size is 300×300×(10…120) mm.

The indirect method of metrological certification based on metrological guarantied quantities [6] allows certificating the produced devices without application of thermal conductivity reference materials with a permissible relative error ± 3% according to requirements of functional standards. It is advantageous to distinguish the created device from analogues.

Samples should be prepared for examination in accordance with recommendations [2, 3]: solid samples are ground to ensure that the working surfaces are plane-parallel and for soft and fibrous make additional dividers or a bounding box. Before placing into the measuring cell of a thermal unit, the samples are measured and weighed to calculate their density. The test sample is placed in a measuring cell between two identical heat flow sensors as it shown in Figure 3. In the case of testing solid samples with non-polished surfaces two thin elastic gaskets with built-in thermocouples are positioned between the sensors and the sample, orienting their junctions to the surface of the sample (Fig. 3, b, c).



a) b) c)

Figure 3. The process of placing the sample into the devices measuring cell:
a) the measuring cell is opened;
b) a lower elastic gasket and a sample are installed;
c) the upper elastic gasket is installed

Measurements should be made on a minimum of three samples of the same material in a dry state at an average temperature of +10ºC and +25ºC or throughout the operating temperature range depending on the purpose of the experiment. The design formula for the thermal conductivity is:

$$\lambda = h \cdot \left(\Delta T / \overline{q} - R_K \right)^{-1}, \quad (1)$$

where h is the sample thickness;

$\Delta T = T_H - T_L$ – the temperature difference between the upper and lower working surfaces of the sample;

$\overline{q} = 0{,}5 \cdot \left(q_H + q_L \right)$ – the average value of the heat flow density passing through the upper and the lower working surfaces of the sample;

R_K – the correction for the total contact thermal resistance of the measuring cell determined during device calibrating.

The thermal conductivity of the material may be calculated as the average value for the investigated samples, taking into account the measurement error δ = ± 3% by the formula:

$$\overline{\lambda}_T = \frac{1}{N}\sum_{i=1}^{N}\lambda_{T_i} + \delta\,, \qquad (2)$$

where $\overline{\lambda}_T$ is the thermal conductivity average value at a temperature T;

λ_{T_i} – the thermal conductivity value of the i-th sample at a temperature T;

N – the number of samples.

In table 1 the results of research the most common types of modern insulation materials on the IT-7 device are presented.

Table 1. The thermal conductivity value of effective heat insulators

Material name	Density, kg/m^3	Thermal conductivity, W/(m·K) (T = 10…25 ºC)
Isophenic IPN-nano	32 … 35	0,019… 0,021
Foam polyurethane	40 … 45	0,024…0,026
Mineral wool	10 … 13	0,035…0,040
Expanded polystyrene	35 … 38	0,043…0,046

The IT-7 device wide temperature range allows for the examination materials throughout the range of operating temperature values with normalized accuracy. Research results of modern heat insulators are shown in Figure 4.

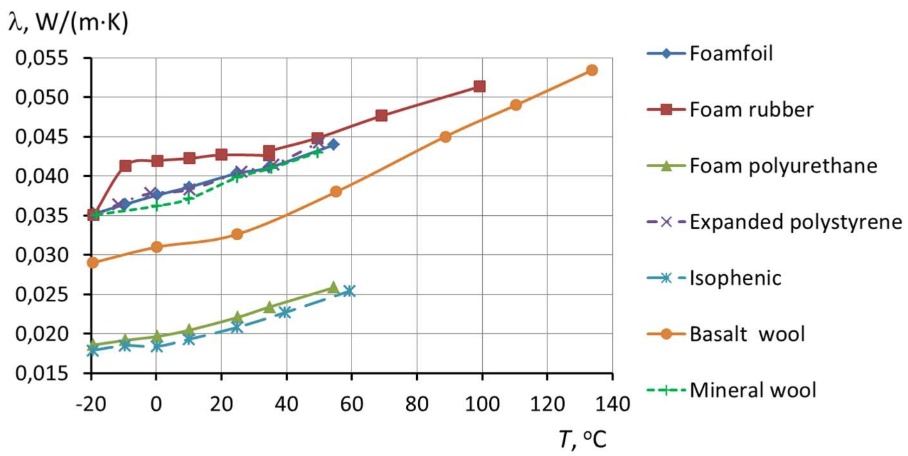
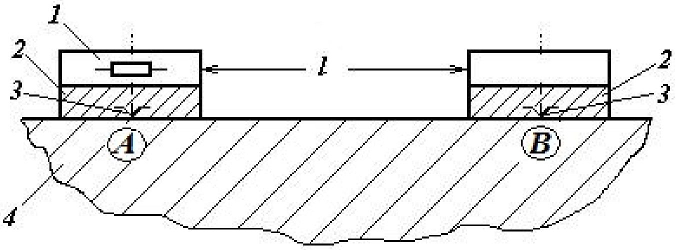


Figure 4. The temperature dependences of modern insulators thermal conductivity value

Analyzing the results obtained at the IT-7 device (Table 1, Figure 4) it is possible to select the material that best suits the requirements for the buildings or structures exploitation and also to predict the change of its heat protection qualities at significant temperature gradients.

4.2 Probe express-method and device for the on-site thermal conductivity research

In addition to calorimetric testing of building materials samples in steady-state laboratory conditions it is necessary to make express measurements of thermal conductivity in the production process in order to control the quality of the product before sending it to the facility or directly to the installation site for discarding non-standard elements that do not correspond to the established ones normative requirements to their thermophysical characteristics. In this purpose the compact portable probe type devices for the building and heat-insulating materials thermal conductivity express-measurements were developed in Institute of Engineering Thermophysics NAS of Ukraine [9, 11].
These devices are based on the method of local heat influence on a limited area of a controlled material flat surface with a differential measuring the difference in heat flow densities and temperatures in the heating zone and the unperturbed zone [10, 11]. The principle of the method consists in bringing a constant density heat flow to the research material sample through a limited area of its surface that contacts the probe. In this case the thermal conductivity value is determined in the steady state from the measurements results of thermal flow density and the difference in temperature values at the center of the thermal action section and at the peripheral point of the surface where the thermal effect may be taken as zero. Schematic diagram of the method is shown in Figure 5.



A – local heat influence zone
B – unperturbed zone

1 – heater
2 – heat flow sensor
3 – temperature sensor
4 – sample

Figure 5. The differential method of local thermal impact

To implement the method a probe device is placed on a flat body surface 4, the temperature of which is equal to the ambient temperature. The heater 1 is such small dimensions so that the body can be considered a semi-infinite array. At zone *A* through the contact area to the surface of the body a constant density thermal flow generated by heater 1 and measured by sensors 2 and 3 (q_A, T_A) acts. The body temperature field undergoes distortions and ceases to be uniform. At the same time thermal losses occur from the body surface outside the contact site due to convective radiation heat transfer. The temperature field at zone *B* that occurs as a result of thermal action is fixed by equal sensors 2, 3 (q_B, T_B) and algebraically added to the initial one [9]. The thermal conductivity is calculated by formula:

$$\lambda = \varphi \cdot \left(\frac{q_A - q_B}{T_A - T_B} \right), \tag{3}$$

where φ is a piecewise polynomial approximation function determined during device calibrating.
The advantage of this method is the possibility of thermal conductivity determining on samples of irregular shape (including on finished products) having one flat surface.
The developed probe express-method is implemented in the IT-8 device shown in Figure 6.

1 – box;
2 – working probe;
3 – the probe-referent;
4 – temperature probe;
5 – limiter

a)

b)

c)

d)

Figure 6. The IT-8M device (a) and sample mounting options (b, c, d)

The IT-8 device (Fig. 6, a) is designed to operation with the computer which facilitates the automation of sensors measurement, processing and documentation processes, and differs according to the design. In this model the heat block and the elements of the electronic unit are combined in the general box 1 and the measurement information from the sensors embedded in the working probe 2, the probe-referent 3 and the temperature probe 4 is transmitted via the USB interface to the laptop where the thermal conductivity is calculated. The horizontal surface of the case 1 is equipped with limiters 5.
The test material sample or finished product may be of an arbitrary shape, but must have one flat surface or allow it to be prepared. The dimensions of this surface must be at least 200×200 mm, and the thickness of the sample is not less than 100 mm. The IT-8 device can work in two positions: "Sample below" (Fig. 6, b) – used for research of porous and homogeneous materials, and "Sample above" (Fig. 6, c, d) – for research of samples of friable, porous and homogeneous materials. In addition such a device can be installed directly on the insulation layer of industrial equipment, creating the necessary conditions for the correct conduct of the experiment and to assess the quality of its thermal insulation in a non-destructive way.
Basic technical characteristics of the IT-8 device:

- thermal conductivity range is from 0,02 to 1,5 W/(m·K);
- main relative error is ± 8%;
- operating temperature range is from 10 to 40 ºC;
- time to reach the stationary mode is about 25 minutes;
- measuring time is 5 minutes;
- diameter of contact spot with sample surface is 20 mm;
- overall dimensions 250×250×85 mm;
- weight is 3,5 kg.

For a qualitative evaluation of the probe express-method the results of expanded polystyrene samples thermal conductivity measurements on the devices IT-7 and IT-8 were compared. The maximum deviation of the results obtained on the IT-8 device does not exceed 2% of those obtained on IT-7 as shown in Figure 7.

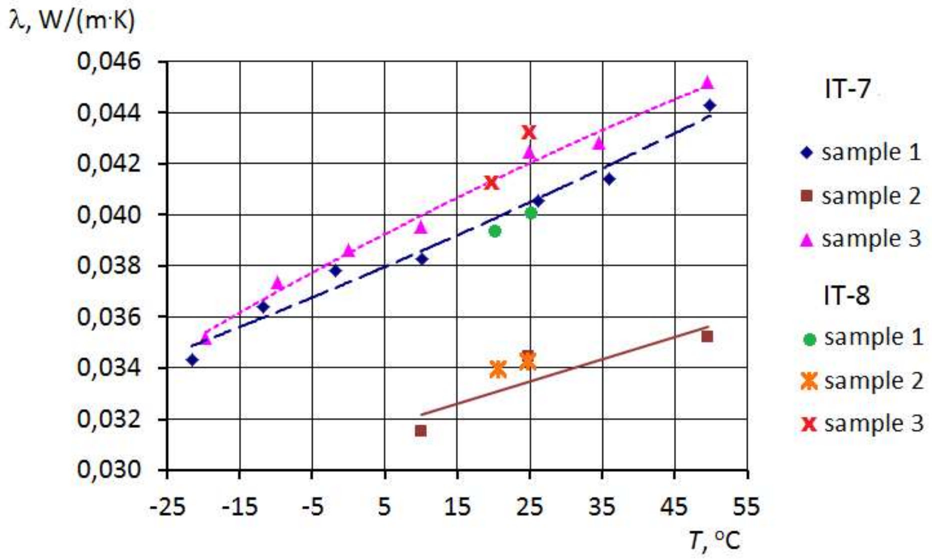


Figure 7. The thermal conductivity temperature dependences of expanded polystyrene samples obtained experimentally on IT-7 and IT-8 devices

5. Conclusion

Certification tests of modern insulating materials with a standardized error should be carried out on device IT-7 type in a steady-state laboratory conditions. However, in order to quickly obtain the final result it is worthwhile using a probe-type mini-device such as IT-8 which makes it possible to investigate the thermal conductivity on arbitrary shape samples with sufficient accuracy.

The set of calorimetric devices created by the Institute of Engineering Thermophysics NAS of Ukraine specialists is implemented in the leading certification laboratories as the State Enterprises "Ukrmetrteststandard" and "State Research Institute of Building Structures", in the building enterprises and municipal power engineering research and testing laboratories of Ukraine as non-standardized metrological certified instruments for thermophysical quantities measuring.

References

1. Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus: Active Standard ASTM C518-10. Developed by Subcommittee: C16.30. Annual Book of ASTM Standards Vol 04.06. doi:10.1520/C0518-10.
2. Thermal insulation. Determination of steady-state thermal resistance and related properties. Heat flow meter apparatus (ISO 8301:1991, IDT): DSTU ISO 8301:2007.
3. Building materials and products. Method of determination of steady-state thermal conductivity and thermal resistance: DSTU BV.2.7-105-2000 (GOST 7076-99).
4. Zarr R.R. A History of Testing Heat Insulators at the National Institute of Standards and Technology. ASHRAE Transactions, 2001, Vol 107, Pt 2, pp.661-671.
5. Hulstrom L.C., R.P. Tye, S.E. Smith. Round robin testing of thermal conductivity reference materials. Proc. XIX Thermal Conductivity Conference, 1986, pp 17-29.
6. Burova Z., L. Vorobyov, L. Dekusha, O. Dekusha. Complex IT-7C for building materials thermal conductivity measurements. Metrology and Devices, Kharkov, 2009, Vol. 6, pp.9-15.

7. Burova Z. Methods and Devices for Insulation Materials Heat-Conducting Determination. SWorld Journal, 2016, Vol.4, Issue 11, pp.26-28.
8. Teplometry: theory, metrology, practice. Book 1. Methods and resources of heat flow measuring. Ed. T Grischehko, NAS of Ukraine, 2017, 440 p., ISBN 978-966-02-8199-8
9. Hardware-software for monitoring the generation, transportation and consumption of thermal energy: Monograph. Ed. V. Babak, NASU, 2016, 298 p., ISBN 978-966-02-7967-4
10. Hotra O., O. Dekusha. A device for thermal conductivity measurement based on the method of local heat influence. Przegląd Elektrotechniczny (Electrical Review), R. 88 NR 5a/2012, pp 223-226, ISSN 0033-2097
11. Theoretical and applied bases of economic, ecological and technological functioning of energy objects : Monograph. Ed. A Zaporozhets, T Bilan, NASU, 2017, 312 p., ISBN 978-966-02-8331-2