VERIFICATION AND VALIDATION OF AN ADVANCED GUARDED HOT PLATE FOR DETERMINATION OF THERMAL CONDUCTIVITY

by

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The guarded hot plate (GHP) method is a widely used technique to measure thermal conductivity of thermal insulation specimens in steady-state conditions. In this paper, an advance of GHP has been developed in the Laboratory for Thermal Technique and Fire Protection in Institute IMS. The innovative GHP has been applied for measuring thermal conductivity using additional heat flux meters. The design of this GHP is similar to the design of the plate for GHP apparatus, however, it has different design: smaller width of thermal barrier, which is filled with insulation glue. Heaters inside hot and guard plates are built from wire for thermocouples, which is a unique type of heater. Geometry of heater has been optimized inside plates to achieve uniform temperature distribution along the specimen surface. Temperature uniformity of GHP and energy balance were experimentally determined. The verification and validation results of improved GHP have been shown. The test method was validated comparing test results of thermal conductivity with results of the round-robin test. Four national companies participated in the round-robin comparison on thermal conductivity measurement by GHP method. The measurement was performed on the same specimen of thermal insulation material (expanded polystyrene) according to SRPS EN 12667 at temperatures ranging between 10-40 °C. The measured thermal conductivity of all participants in the round-robin test was input data for statistical processing according to SRPS ISO 5725-2 and ISO 13528. To evaluate the performance of the participants, the "z" score has been used. Measurements were conducted successively for all participants. Since 2020, the Accreditation Body of Serbia also approved this test method.

Keywords: *thermal conductivity, heat plate, SRPS EN 12667, GHP apparatus with heat fluxmeters, method verification and validation*

Introduction

The development of urban construction promotes rapid expansion of advanced building and materials industry [1]. The structural and thermal properties of multilayer build-

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ing components play a key role not only in hygrothermal and acoustic comfort but also in energy performance of building's envelope [2]. Heat gains and heat losses of buildings mostly depend on the thermal and physical properties of structures and materials [3]. An excellent thermal insulation of thermal storage systems is a prerequisite to reduce heat losses to a minimum [4]. As in many cases, energy consumption is related to the use of heat in different circumstances and environments, knowledge of thermal characteristics of materials and systematization of available and new data, which is of great importance for both industry and research in science and engineering [5]. In an effort to save energy and improve the thermal efficiency of buildings, the use of an insulator with good thermal performance and optimal thickness is important when constructing industrial, commercial, and residential buildings [6].

Thermal conductivity is one of the most important thermodynamical properties of materials. Heat transfer through solid bodies is conductive heat transfer and it depends on thermal conductivity of a material. Determination of thermal conductivity is a crucial parameter, because it gives possibility for calculation of thermal transmittance according to algebraic Fourier equation [7]. Fourier's law can be treated as fundamental theory of heat conduction for single-phase material in thermal equilibrium, and it may not be automatically applicable for multi-phase heat transfer problems [8]. Determination of thermal conductivity is based on using Fourier equation, where temperature difference between hot and cold plate, thickness of specimen and density of heat flow rate are measured in steady-state.

The GHP method is a reference technique for measurement of thermal conductivity of insulating and poorly conductive materials [9]. Determination of thermal conductivity in steady-state can be achieved using standardized test methods. Steady-state heat conduction through the specimens is achieved in equilibrium state when the heat flux becomes constant between the heater and to the coolers [10]. Thermal conductivity of insulation materials can be measured using standardized stationary methods with heat flow meters, ISO 8301 [11], and with GHP apparatus according to ISO 8302 [12]. The first method uses density of measured heat flow rate for calculation of thermal conductivity. The second method (GHP apparatus) is based on the measurement of electrical power from an electrical heater installed on the hot plate. Hot and guard plates need to obtain uniform temperature along the surface of a specimen.

In this paper, an advance GHP has been applied for measuring thermal conductivity using heat flow meter. The design of this GHP is similar to the design of plate for GHP apparatus, however, it has two new characteristics: different width of thermal barrier and the gap between hot and guard plates have been filled with insulation glue. Heaters inside hot and guard plates are built from wire for thermocouples and their geometry is optimized inside plates to achieve uniform temperature distribution along the specimen surface.

Temperature uniformity on the surface of plates and specimen was checked and experimentally validated. To regulate lateral heat transfer between two plates, athermopile across perimeter of gap between hot and guard platewas used. In addition, the energy balance analysis was performed. All results were approved according to standards SRPS EN 12667 [13] and SRPS EN 1946-3 [14].

Round-robin test program was performed to validate test results for thermal conductivity obtained using this new design of GHP. Good correlation was found between the research results and results from other laboratories. In addition to that, this test method was accredited by the Accreditation Body of Serbia (ATS) in 2020.

Development of guarded hot plate

A new design of GHP was developed in the Laboratory for thermal technique and fire protection at the Institute IMS (Institute for testing of materials in Serbia). The project started in 2018. A GHP was designed for temperature ranges from 10-40 ℃. The idea was to replace old standard type of GHP with a new one. This new design of plate is predicted to determine thermal conductivity according to the Serbian national standard SRPS U.A2.020 [15].

Old design of GHP was developed according to withdrawn DIN standards [11, 15]. The control system is not certain for today measuring requirements for thermal conductivity. Obtaining stationary state with this design was too long and data analysis with non-computed acquisition was hard to achieve and the uncertainty was large. There is a constant demand on Serbian market for this type of examinations. An advanced system was needed with reduced time for obtaining steady-state and with lower measurement uncertainty. Consequently, a new design of plate was proposed, different from standard designs [12-14]. Major advantage of this design is a small width of thermal barrier, as well as the fact that the space between hot and guard plate has been filled with insulation glue. Temperature difference between these two plates is controlled by using thermopile. It is placed along the perimeter of the thermal barrier. Electrical heaters of hot and guard plates are milled into the plate and material of this heater is thermocouple *K*-type. The thermocouple *K*-type represents the shielded wire made from chromel-alumel, with measurement range –200 ℃ to 1260 ℃. Heaters are connected on the standard plug for thermocouple *K*-type, which is fixed to the guard plate. These two heaters are connected on the two-channel voltage source, which is controlled by computer. Cold plates were not developed. Instead of developing new cold plates, old cold plates from standard laboratory GHP were used. These plates were improved using PID regulated cooling bath with temperature fluctuation of 0.1 ℃ on the surface of cold plates. An additional requirement of project was to develop a test desk for entire equipment system.

To obtain steady-state with GHP apparatus is more time-consuming because it is necessary to have zero heat flow rate between hot and guard plate, so the stabilization of electrical power lasts longer. Methods with heat flow meter take a shorter time to obtain reading of heat flux in the steady-state where heat flux is constant. The temperature difference should also be constant in the steady-state. During development of new GHP, these factors were improved. The thermal conductivity has been measured faster and more precise, with heat flux meters using design of new GHP. These factors were numerically approved and after this, on the surfaces of hot and cold plates were placed thin heat flux meters. The old plate was installed according to the standards[11, 15] while new designed plates used two thin flux meters and measuring method was tested according to the standard [13].

Technical description of guarded hot plate

The GHP is designed for standard measurement of thermal conductivity where specimens are placed below and above the heat plate (symmetric design). Two cold plates were placed on the specimens. Scheme of apparatus for determination of thermal conductivity is shown on fig. 1.

Heat plate with dimensions 750 mm × 750 mm, with thickness of 16 mm is produced using two aluminum plates, which are milled, before placement of heaters. Both plates are glued to each other with high thermal conductivity glue. Heat plate consists of two parts: inside (hot) and outer (guard) plate. Hot plate has dimensions 500 mm × 500 mm, while the rest of the heat plate is guard plate. Both plates are separated with thermal barrier. This break is two-component insulation glue with width of 1 mm. Detail of the thermal barrier is shown in fig. 2 (cross section with the special *stair*). This *stair* is an important construction detail. It makes hot and guard plates inseparable. This was not the case in old plates, where the thermal barrier is wider.

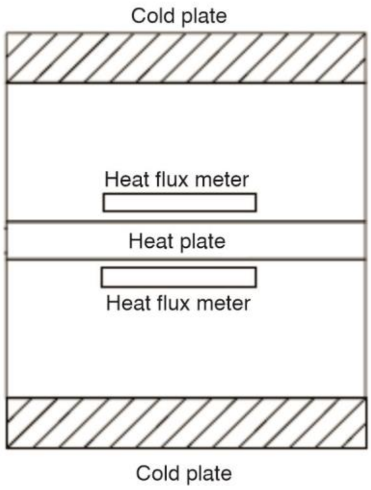

Figure 1. Scheme of apparatus for determination of thermal conductivity

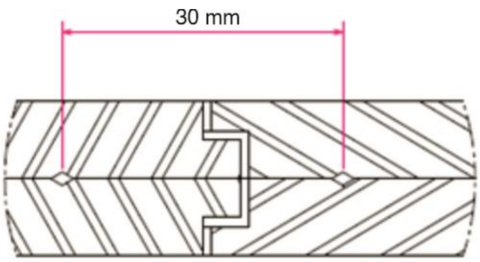

Figure 2. Cross-section of thermal barrier and distance between heaters inside hot and guard plate

Heat plate consists of two segments. Each segment consists of hot and guard plates, which are glued to each other. Two segments are connected using glue with high thermal conductivity value. Before fixing plates to each other, the grooves carved into plates for electrical heater. Electrical heaters are made from the thermocouple wire (*K*-type), with wire thickness of 0.8 mm. In the grooves carved into plates, which are with isosceles triangle geometry, are installed electrical heaters. Air space between heater and plates is filled with glue to prevent convection heat transfer in these gaps.

Distance between two wires is 30 mm and this distance is optimized using FEM with goals to reach steady and uniform temperature on the surfaces of heat plates.

Configuration of heaters installed on the heat plate is shown in fig. 3. Five standardized plugs for thermocouples were used. Heaters were connected in these plugs, while on plug is for measuring voltage on the thermopile. Heaters are supplied with two channels voltage source.

Experimental determination of heat-plate properties

General

Experimental determination of heat plate properties developed by Laboratory for Thermal Technique and Fire Protection (Institute IMS) is demonstrated. Properties such as temperature uniformity during the entire working interval and during the steady-state, uniformity of heat transfer through the specimens above and below, were measured. Lateral heat transfer through particular design of thermal barriers between hot and guard plate was evaluated using thermopile across the perimeter of the thermal barrier. Intensity of measured voltage on thermopile was proportional to the temperature differences, which are being calculated.

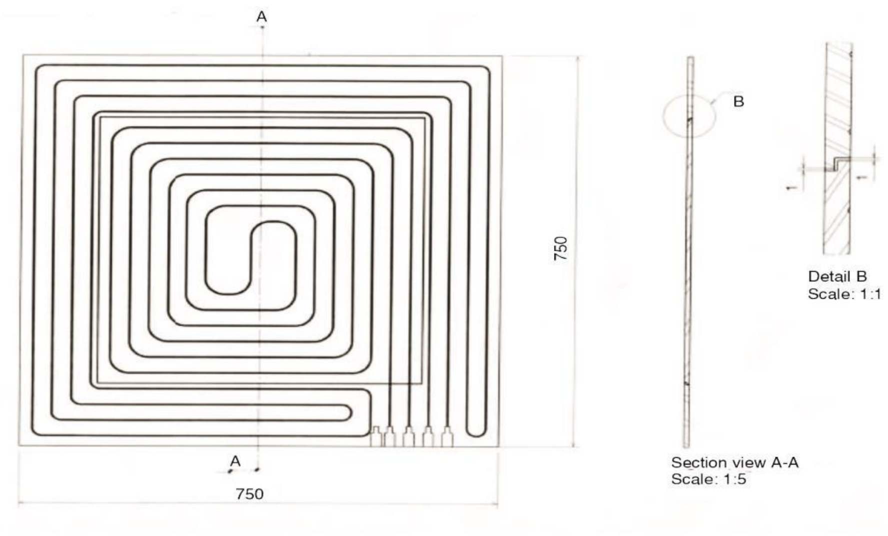

Figure 3. Configuration of installed heaters on the plate

Limits for the equipment performances for measuring of thermal conductivity are provided by the standard SRPS EN 12667 [13]. Standard criteria dealing only with heat plate was checked.

Energy balance of heat plate

The major requirement for heat plate is temperature uniformity on the heat plate surfaces. Beside this requirement, it is necessary to obtain temperature uniformity on both cold plates, because workflow of heat plate depends on the working conditions of the cold plates. If these requirements were fulfilled, then the uniform heat transfer through both specimens would be satisfied. Two heat flux meters were installed on the surfaces of heat plate above and below. Positions of heat flux meters are shown in fig. 1. Based on measured heat flow density in the steady-state, it is possible to check uniformity of heat transfer through both specimens. Measured values of heat flux in the steady-state on both sides of heat plate are shown in fig. 4. Averaging of measured heat fluxes is during time interval of 500 minutes. The heat flux fluctuation $q_{\mathrm{hot(up)}}$ is lower because we had in this place cold plate. Temperature fluctuations on cold plate are much lower because it is cooled by fluid, while hot plate is heated by the electrical heater, so fluctuations are higher.

Energy balance analysis of heat plate was performed on specimens of mineral wool with thickness of 5 cm. Surface of specimens is rough and there is increased surface heat resistances. These values of surface heat resistances above and below the heat plate are not the same. Relative difference between measured heat fluxes is less than 6%, which is satisfied congruence between measured results. Allowed relative difference between these measured heat fluxes is not any requirement in test standards, because this relative difference depends of surface roughness and material composition. On the solid materials, this value is lower, for example, measured relative difference was less than 3% on extruded polystyrene. A limit for heat flux fluctuation in the standard is 2%. Based on this standard, heat flux fluctuation is not acceptable as shown in fig. 4.

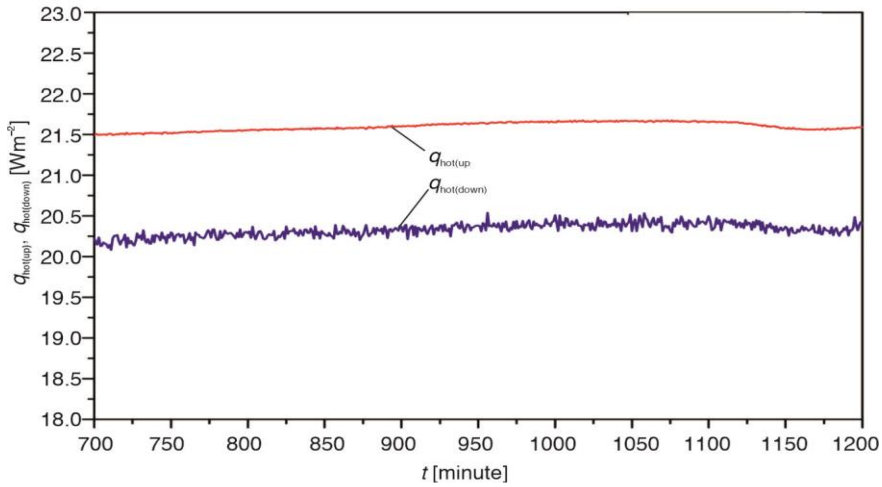

Figure 4. Measured heat fluxes above and below $q_{hot(up)}$, $q_{hot(down)}$of heat plate

Relative difference between measured heat fluxes depends on the material composition, but this relative difference is a low value, so it can be concluded that energy balance of heat plate is experimentally approved.

Checking of temperature uniformity and stability of heat plate

Five thermocouples are placed on both sides of hot plate, while four thermocouples are placed on the guard plate. Thermocouples are placed according to standards [13, 14]. Figure 5 shows measured temperatures on the hot plate. During steady-state, there was no increase of standard deviation between any measured temperatures on the hot plate. Allowed deviation between any two measuring points at every moment is less than 0.2 K, which is one of major requirements for GHP verification [13], and this requirement is experimentally checked.

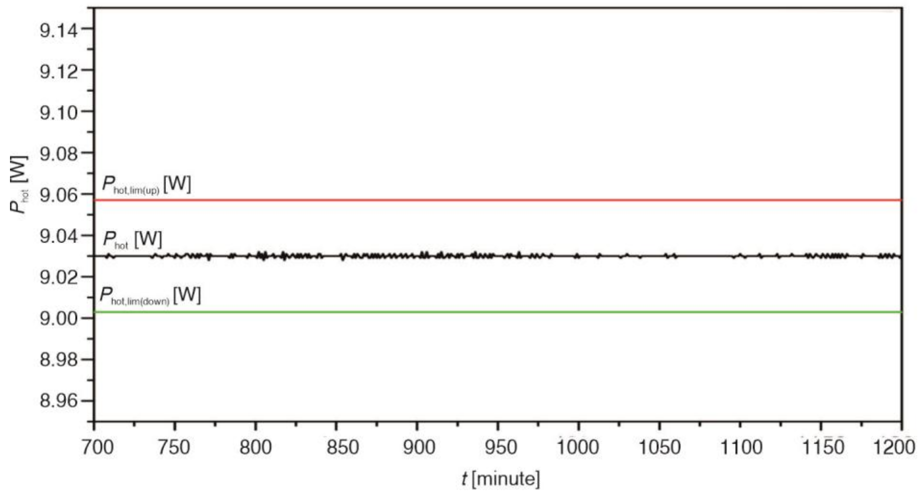

Figure 5. Fluctuation of input power of hot plate

Temperature stability is shown in fig.5. It can be concluded that the temperature stability is approved. Same analysis is performed on the other side of the hot plate and similar results were obtained.

According to the standard for GHP verification [13], limited fluctuations of input power, is less than 0.3%. The fluctuation of input power is in permitted limits, which is shown in fig. 5. In the fig.6, the temperature difference on specimen during the steady-state time has been given. The maximum deviation during the stationary period has been 0.82%, while allowed limit of fluctuation temperature difference is 1%.

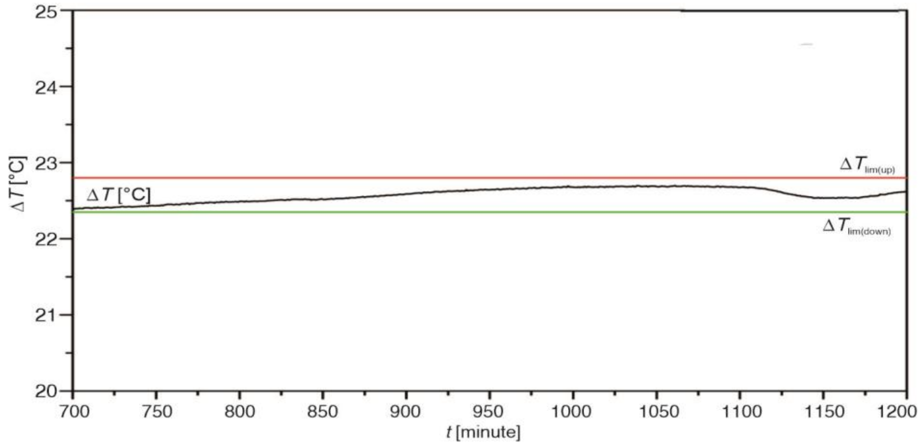

Figure 6.Temperature difference between average hot and average cold plate

Figure 7 shows a temperature difference between the average temperatures on the hot plate surfaces above and below.

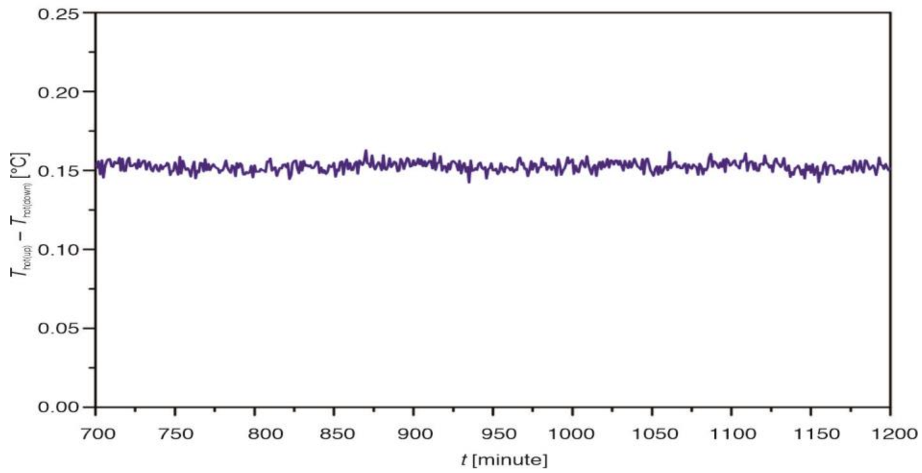

Figure 7. Temperature difference between average temperatures on the hot plate surfaces above and below

Allowed temperature difference between temperatures on the hot plate surfaces above and below is 0.2 K [13], while measured temperature difference is approximately 0.15 K. Heating uniformity of hot plate was approved using energy balance and temperature analysis.

Conduction heat transfer through thermal barrier between hot and guard plate

The heat plate consists of two parts, hot and guard plate. Space between these plates is filled with thermal insulation glue, which is a thermal barrier. Major goal of thermal barrier is to prevent conductive heat transfer between these two plates. If temperature difference on these plates is equal to zero then there is no conductive heat transfer through a thermal barrier.

Thermal performance of thermal barrier was checked by placing thermopile across perimeter of a thermal barrier. Voltmeter was connected to the end of thermopile wires. The measured voltage is proportional to the temperature difference, so it is possible to calculate average temperature difference across perimeter of thermal barrier. Voltage signal, which was measured, is shown in fig. 8. First, it is necessary to perform some approximation about voltage drop on thermopile. Voltage drop on each of the thermocouples of thermopile is equal. There were 56 thermocouples in series and after dividing of voltage signal with number of thermocouples then electric signal on one thermocouple is calculated. Averaged value of measured voltage for 500 minutes is 0.135 mV. Dividing this value with number of thermocouples average voltage signal on every thermocouple is 2.41 μV. Two thermocouples in arrow represent differential thermocouple connection, so measured voltage in this case is 4.82 μV. At this voltage value, temperature difference is 0.12 °C. In addition to the calculated temperature difference using thermocouples in the differential connection (thermopile), the temperature difference between the hot and guard plate was determined using thermocouples located on the plates. Figure 9 shows obtained temperature difference that is relatively similar to the obtained temperature difference using thermocouples in the differential connection. Since the temperature differences between the hot and guard plate are negligible, a high level of uniformity of the entire heat plate can be stated.

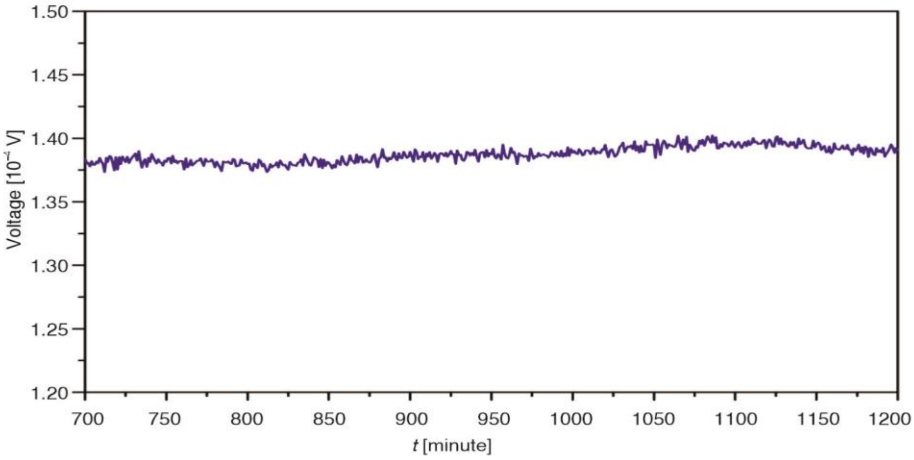

Figure 8. Measured voltage signal on thermopile

Additional requirements for hot guard plate apparatus

Assessment of GHP apparatus validity needs a check for working condition of cold plates. This analysis was performed on cold plates. The basic requirements for cold plates are uniform temperature distribution on the surface of cold plates and the proximity of average operating temperatures on both cold plates. Five thermocouples are placed on the cold plates, and these positions are defined by the standard [13]. The maximum deviation during the stationary period has been 0.82 %. Each of the measured temperatures at any time during the stationary period did not deviate above 1%, so it has been confirmed that the cold plates have uniform temperature distribution.

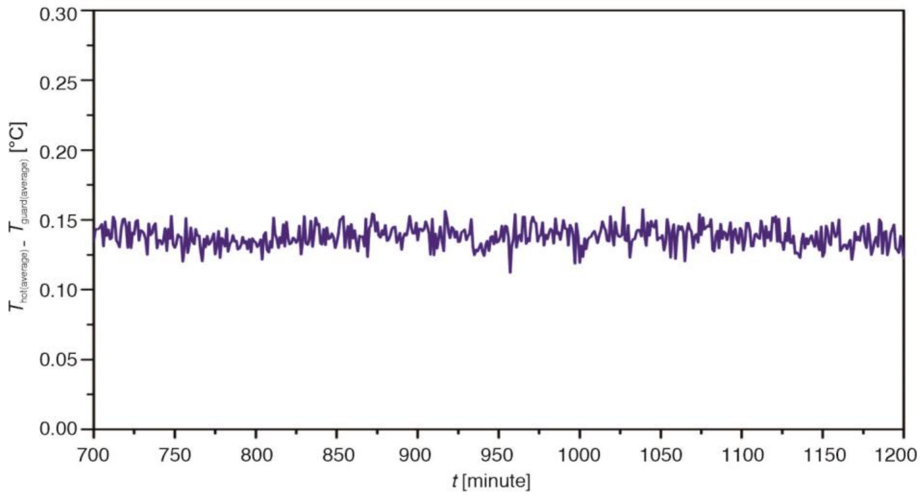

Figure 9. Temperature difference between hot and guard plate

An additional design requirement of the device itself is the prevention of lateral losses through the specimens. Lateral heat losses through the specimen are not directly related to the operation of the hot guard plate, but they were analyzed in this paper to confirm the correct operation of the test method. Estimation of lateral losses through the specimen is determined using the edge temperature ratio, *e*, according to the standard SRPS EN 12667 [13]. The edge temperature ratio is defined by the following formula:

$$e = \frac{T_e - T_2}{T_1 - T_2} \tag{1}$$

where T_1 and T_2 are average temperatures on hot and cold plate, while T_e is the edge temperature of the specimen.

Lateral heat losses are theoretically equal to zero if the mean temperature of the specimen edge corresponds to the mean operating temperature, *i.e.* when the edge temperature coefficient has a value of 0.5.

The value of lateral heat losses through the specimen itself directly depends on the temperature operating regimes, *i.e.* if the measurement is performed at an elevated mean temperature, and then the lateral losses are also increased because the temperature difference between the edge of the specimen and the environment is increased. In order to analyze the effect of heat lateral losses, the measurement of thermal conductivity at higher operating temperature regimes was performed in order to analyze the operation of the device in other conditions when higher lateral losses are expected. In this way, the insulating properties of the device housing were checked.

The mean temperature of the hot plate in the stationary time interval is ≈34 °C, while the mean temperature of the cold plates is ≈11 °C. Ambient temperature was controlled in the temperature interval 23 ±1 °C. Two thermocouples were previously placed on the edges of the specimen and it was determined by measurement that $T_e \approx T_{amb}$. Using the previous values, the value of the edge temperature ratio for the previous operating parameters was calculated and amounts to 0.52. Based on the obtained value of 0.52 (≈0.50), it was concluded that the lateral losses are negligible, *i.e.* the construction of the housing is also suitable for operating modes with elevated average temperatures.

Validation of the heat plate using round-robin test results

During 2019, the Laboratory for Thermal Technique and Fire Protection (as a part of the IMS Institute) proposed to manufacturers of thermal insulation materials that have devices for measuring thermal conductivity within their factories, which is a requirement of harmonized production standards, to join the interlaboratory testing program (round-robin). Interlaboratory comparison is an important activity of accredited laboratories and designated institutes [16]. The purpose of this program was to determine the differences in the measured values of thermal conductivity and statistical processing of the obtained values on the same specimen using the test standard SRPS EN 12667 [13] at average temperature of 10 °C.

Three large manufacturers in Serbia and the Laboratory joined the program. The task of the Laboratory as an organizer was to select a specimen and deliver the same specimen to the participants of the round-robin testing program. A specimen was extruded polystyrene, two plates with dimensions of 750 mm × 750 mm, were selected. The reason why the specimens were cut to these dimensions was measuring of the thermal conductivity on the *new* hot plate. The measurement was performed at an average temperature of 29.05 °C and the obtained thermal conductivity was subdued to a thermal conductivity of 0.03425 W/mK at 10 °C using the standard SRPS EN 10456 [17].

The device with the *new* hot plate did not directly participate in the round-robin program because it was required that all program participants have the same specimens dimensions 500 mm × 500 mm and the measurement was performed at the same average specimen temperature.

After measuring on a specimen with dimensions 750 mm × 750 mm, the specimen with dimensions 500 mm × 500 mm was produced. The measurement was then performed on another device of the testing laboratory of the IMS Institute, which was a participant in the round-robin program, at an average temperature of 10 ˚°C. After that, the specimen was sent for measurement to other participants in the program.

After the measurement was completed, statistical report GFT-6538/19-MLP [18] was written, where all data were statistically processed where all participants received a passing grade. The statistical report has been done in accordance with the standards SRPS ISO 5725-2 [19] and ISO 13528 [20]. To evaluate the performance of the participants the *z* score has been used. In the fig. 10 the obtained results of thermal conductivity at 10 °C has been shown. With the numbers 1-4 the values of thermal conductivity for four participants in the round-robin program has been presented [18]. The obtained value of thermal conductivity on the device with the innovative hot plate has been marked with a number 5.The value of thermal conductivity, which uses a GHP with two heat flux meters, is 0.03425 W/mK at 10 °C.

In the tab. 1, based on the obtained values of measured thermal conductivity and after of application of the methodology for evaluating the performance of participants in round-robin testing in accordance with the standards [19, 20], the obtained values of *z* score has been presented. The absolute value of *z* score ≤2 is assessed as satisfactory.

The obtained value is certainly in the satisfactory domain of measured values, which can be seen in fig. 10. Validity of obtained result on this heat plate is in agreement with other results in fig.10.

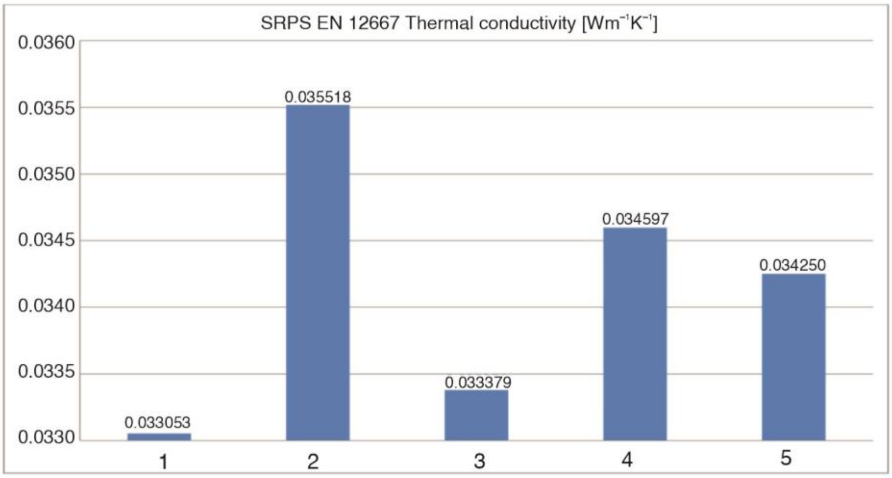

Figure 10. Interlaboratory results for thermal conductivity measurements [18]

Table 1. The obtained values of *z* score

Participants	SRPS ISO 5725-2	ISO 13528
1	-1,0	-0,7
2	1,2	0,9
3	-0,7	-0,5
4	0,4	0,3
5	0.1	0.1

Conclusions

This paper presents an advanced GHP for measuring the thermal conductivity of insulation materials with higher precision and at the faster rate than the old equipment. The specific design of the thermal barrier between the hot and guard plate is made of thermal insulation glue and is improved compared to competing heat plates where GHP usually consist of two separate units. The design of this new plate is composed as one functional unit. Innovative solutions are also presented through the use of advanced heaters, which are wires for thermocouples. In addition to improved structural characteristics, the heat plate was experimentally tested in order to demonstrate the temperature uniformity and the energy balance of the heat plate.

The obtained results of measured temperatures and heat flux were evaluated according to the standard requirements. The results achieved by the measurement are more certain than the requirements prescribed in the standards, which means that this concept and design of the heat plate can be effectively used in the practice for measurement of thermal conductivity.

The results of the thermal conductivity test with the new design of GHP were validated through the results of the interlaboratory tests. After the detailed analysis of the test method by the technical experts of the Accreditation Body of Serbia, the accreditation of the test method was approved at the beginning of 2020.

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Nomenclature

e – edge temperature ratio [–]
T_e – edge temperature on specimen [°C]
T_1 – average temperature on hot plate [°C]
T_2 – edge temperature on cold plate [°C]

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