AN IN SITU TEMPERATURE CALIBRATION OF A GUARDED HOT PLATE APPARATUS

by

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This paper presents design, development, and testing of a custom-made calibration environment for the calibration of temperature sensors applied in a guarded hot plate apparatus for thermal conductivity measurements. Description of an in situ calibration principle and realization are introduced. Results of numerical simulation, as well as those of experimental validation are given in a separate section of the paper. According to the results, proposed in situ calibration by using the applied guarded hot plate apparatus elements improves both accuracy and traceability the of thermal conductivity measurement

Key words: *guarded hot plate method, in situ temperature calibration*

Introduction

The guarded hot plate (GHP) method is a reference technique for the measurement of thermal conductivity of insulating and poorly conductive materials. It is based on an application of the Fourier's law of heat conduction. A stationary and homogeneous heat flux through tested specimens is generated in a specifically designed heater, which produces a measurable temperature difference between the specimen surfaces. By measuring the flux, *i. e.*, power consumption of the heat source, as well as the temperature difference, specimen area and specimen thickness, one can evaluate the specimen thermal conductivity.

Any version of apparatus which operates according to this method needs to provide, at a highest possible extent, a stationary and 1-D heat flux through the cross-section of a plane-parallel homogeneous specimen with a finite thickness. In practice, however, major contribution to the uncertainty of thermal conductivity values comes from temperature measurement, hence achieving its high accuracy is required. Knowing that the uncertainty of temperature measurement is always greater than the calibration uncertainty of applied temperature sensors due to different effects of particular measurement conditions, it is recommended [1], to calibrate temperature sensors *in situ*, *i. e.* under the conditions similar to those of the actual measurement. In order to meet this requirement on the basis of solutions proposed in [2] and procedures described in relevant standards [3, 4], also harmonized with other major apparatus improvements [5], a custom-made environment for an *in situ* calibration of the temperature sensors used for the thermal conductivity measurement in the Vinca Institute of Nuclear Sciences, has been developed. Description of this environment and results of its validation are presented in this work.

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Apparatus and measurement

A GHP apparatus developed in the Institute Vinča [6, 7] consists of a central measuring section, power and control unit, measurement system, and supporting mechanical construction with an isolation chamber. A schematic representation of the GHP apparatus is shown in fig. 1(a). A heat source, marked as GHP, is placed between two identical specimens of the material under investigation and a pair of massive flat aluminum coolers in contact with the opposite specimen sides. The temperature of the coolers is maintained by circulating fluid from a thermostatic bath.

The hot plate, as shown in fig. 1(b), consists of two separated, co-planar, and centrally symmetrical electrical heaters. The inner, or main heater, generates a heat flux which flows by conduction through the specimens in the direction toward the coolers. The value of this flux is determined by measuring the voltage over the main heater wire and the current through related standard resistor. The outer, or guard heater, provides an auxiliary heat flux in order to ensure 1-D heat flux in the central specimen area. The both heaters were made of resistance heating wires and winded as a squared spiral with related spatially chosen nodes, marked A to D in fig. 1(b), for connecting a stabilized power source and a precise multichannel voltmeter. The wires were tightly placed between two isolation layers, and finally enclosed by two square 500×500 mm aluminum plates. A 250 mm squared gap and 1.2 mm in width was made in the both plates for a thermal separation between the central and guard heater surfaces, as seen in fig. 1(b). The free surfaces of the plates were coated with high emissivity graphite paint in order to minimize the heat exchange due to reflected radiation through the specimens. As an important measure of deviation from the 1-D heat flow, the temperature difference between the main and guard heater is determined by using a specially designed thermopile fixed below the aluminum plates over the gap and electrically isolated by thin layers of polytetrafluor oethylene coated glass fabric.
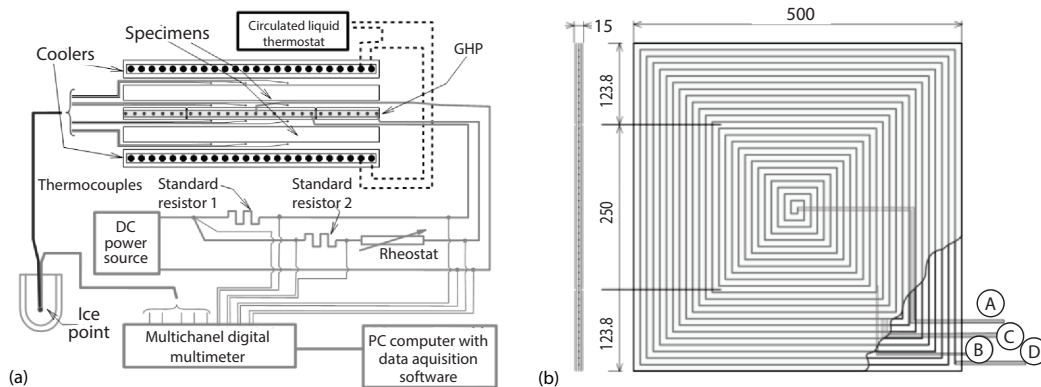

Figure 1. Schematic of the GHP apparatus; (a) central measuring section with electrical circuit, (b) cross-section of the GHP (locations for temperature measurements are marked in circles)

The measurement system of the apparatus consists of the multichannel digital voltmeter with a set of K-type thermocouple sensors and a computer with an in-house developed software for acquisition, display, and data storage [8]. The thermocouples, 0.1 mm in diameter, are used with cold junctions kept at constant ice-point temperature. Thermal electromotive force (EMF) values are recorded by the voltmeter and converted to temperature according to standard polynomials. Voltage drops over the contacts of the main and guard heaters, as well as over the two standard resistors for the measurement of related electrical currents are also measured and recorded by the multichannel voltmeter, as shown in fig. 1(a).

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At the beginning of a measurement procedure, the pair of specimens is placed between the GHP and two coolers and the volume of the isolation chamber is filled with a granular, thermal insulating material. The temperatures of the thermostatic bath and the two heaters are set to those values which provide the steady-state 1-D heat flux through the specimens. The evolution of all measured signals is monitored until the steady-state is reached. The stabilization time is usually 10-40 hours, depending on the operating temperature, initial state, as well as the properties of specimens' material. Having the stationary values of the voltages measured over the main heater and relating standard resistor which give the value of the heat flux, P, and the resulting mean temperature difference between the *hot* and *cold* surfaces of the specimens in the steady-state, ΔT, the effective thermal conductivity value of the specimens under test, λ_{eff}, is determined from $\lambda_{\text{eff}} = (P/2A)(d/\Delta T)$, where d is the mean specimen thickness and A the cross-sectional area of the main heater.

The specimen dimensions suitable for this apparatus are 500×500 mm, with a thickness 5-80 mm. A typical heat flux through the specimens may vary 1-10 W, while the average temperature difference across the specimen surfaces should lie in the range 5-15 °C, depending on the specimen thickness and its thermal conductivity. The mean specimens' temperature can be set in the range about 15-85 °C.

Following instructions in [9], the measurement uncertainty of the thermal conductivity is the combined uncertainty of the main heater power, mean specimen thickness, total main heater surface, and measured temperature difference [10]. In most cases, the uncertainties related to the values of heater surface and specimen thickness do not significantly contribute to the total measurement uncertainty of final results. On the other hand, regardless the typical low uncertainty of voltage measurements, determination of the related heat flux value is far more uncertain. In fact, effects of undesired heat exchanges give a significant contribution to this uncertainty, especially in the cases of a low heat flux (main heater power of 1 W or less) [11]. Corrections due to these effects are usually performed by temperature measurements in several additional points of interest [9-11].

In the application of this apparatus, as in the GHP method in general, the greatest contribution to the total uncertainty comes from the temperature measurements, *i. e.*, from the value of temperature difference, ΔT. The ASTM standard [3] recommends that this value should be in the aforementioned range from 5-15 °C, but with a relative extended uncertainty less than 1%. The standard also sets a condition that the surface temperature should be measured at several locations over the hot plate, that is, in the case of the actual apparatus, in the five measuring locations per surface, see fig. 1(b), or 20 temperature points in total. Moreover, additional sensors may be placed into other locations in order to monitor the measurement conditions and perform appropriate corrections due to the non-uniformity of temperature profile. Having the described apparatus and specific conditions for temperature measurement, as well as the recommendations regarding the precision, the applied temperature sensors connected to the measurement device should be calibrated under appropriate *in situ* environment [1, 2].

In situ calibration environment and simulation example

The main requirement for any temperature calibration environment is to provide a good heat exchange between a reference and a thermometer under calibration. A stirred fluid, such as water or silicone oil, is an excellent calibration environment because heat is transferred efficiently in all directions. In dry calibration environments, however, the heat exchange between the reference thermometer and its surroundings should be closely analyzed. As the perfect temperature homogeneity of the calibration environment cannot be achieved, corresponding
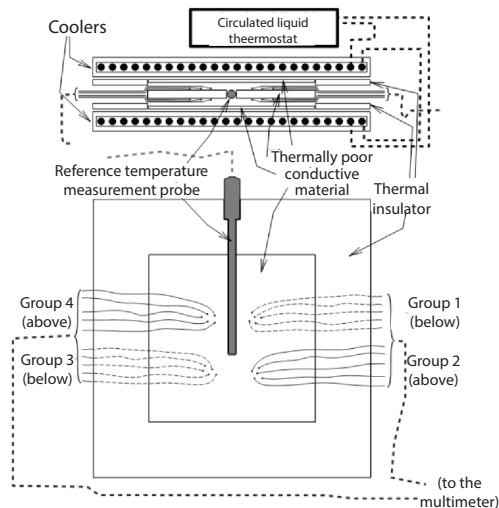
Figure 2. An *in situ* calibration environment of the apparatus

temperature deviations should be estimated and included in the final uncertainty budget.

An *in situ* calibration set-up developed and applied in this work is shown in fig. 2. A reference thermometer is placed into a tightly tailored groove previously cut in a thin plate made of an elastic, dielectric, and thermally poorly conductive material that serves as a calibration medium. Temperature sensors under calibration (thermocouples in this case) are located at the both surfaces of the same calibration medium. The medium with an additional thin elastic layer for the reduction of thermal contact resistance and air gaps that surround thermocouple wires is then pressed between the two coolers of the apparatus.

In the case of a solid calibration medium, such as that presented in fig. 2, heat exchange through the lateral surfaces of the three-layered structure may be significant, so the dimensions of plates are reduced to make a frame of thermal insulation material whose outer edges fit to the cross-section dimensions of the coolers. As the intention is to simulate actual operating conditions to the maximal possible extent, entire construction shown in fig. 2 is therefore closed within the insulation chamber whose volume is filled with the same granulated thermal insulation as in a usual apparatus operation.

Silicon rubber, in form of sheets 1 mm and 3 mm in thickness, has been chosen for the material of the calibration medium due to its thermal stability and mechanical flexibility. Thermal conductivity of the material was determined experimentally by using the axial guarded heat flow apparatus [11]. The thermal insulating frame that can be easily cut and installed into the apparatus was made of two polystyrene sheets, 3 mm in thickness.

In order to verify the concept of the described calibration environment, a 3-D model was made and related heat transfer was simulated by a FEM analysis. The coolers were modelled according to their original design – 12 mm wide and 9 mm deep grooves carved into the 15 mm thick AlMg4.5Mn block, covered with 3 mm silicone rubber sheets and 5 mm thick AlMg4.5Mn plates. A reference temperature probe was modelled as a stainless steel rod with 3 mm in diameter. The diameter of the probes under calibration (thermocouple wires) was taken to be 10 μm and thickness of its PTFE insulation 15 μm. The values of thermophysical properties of the applied materials, except for the thermal conductivity of the silicon rubber sheets, were taken from literature [12]. The set-up was considered to be surrounded by air.

Numerical simulations performed under various boundary conditions, based upon previous measuring experience on the apparatus, and at different operating temperatures, showed that the temperature distribution of the calibration medium depended rather on the assumed boundary condition than on the operating temperature. For example, for a boundary condition of first kind, results of the temperature distribution at upper and lower surface of the calibration medium (3 mm silicon rubber plate) are shown in fig. 3. The temperature of the lower and upper coolers was set to 40.0 °C and 40.3 °C, respectively, while the temperature of all lateral surfaces, fig. 2, was set to 38 °C, corresponding to a real situation when the isolation chamber was filled with the granular thermal insulator.

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According to figs. 3(a) and 3(b), it can be noticed that the surface temperature near the reference probe (a tiny line from the left hand side at $y = 0.25$ m) slightly differs from the temperature in its vicinity, but that difference may usually be neglected in comparison to the overall temperature distribution. Also, the influence of the probes under calibration to the temperature distribution at the surfaces of the calibration medium was negligible in all simulations.
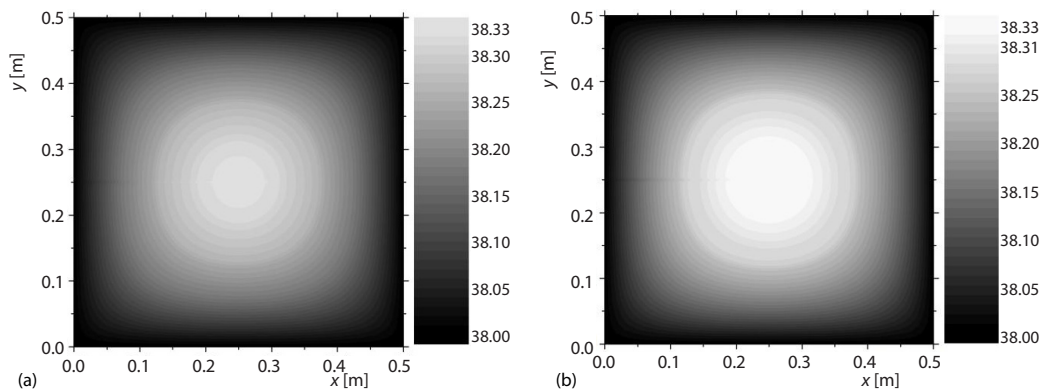

Figure 3. Simulated temperature distribution; (a) upper and (b) lower surface of the calibration medium

Temperature profiles in direction parallel and perpendicular to the reference probe for $y = 0.25$ m and $x = 0.25$ m, respectively, and in the range of interest (0.1-0.4 m for the both co-ordinates) are given in fig. 4. Figure 4(a) shows that the reference probe locally disturbs temperature field at the extent of only 0.01 °C when the air temperature is 2 °C below the thermostat temperature, while the temperature difference of 0.3 °C of the thermostat liquid causes the temperature drop of 0.02 °C through 3 mm silicone rubber sheet. In perpendicular direction, temperature perturbation due to the reference probe is also negligible, *i. e.*, less than 0.01 °C, while the overall surfaces temperature variations are about 0.13 °C in the range of interest. If we take into consideration that the best extended calibration uncertainty for this type of sensors is about ±0.08 °C in a liquid calibration medium, the computed variation of the described temperature profile is within this limit. However, the results of simulations suggested that potential usage of the proposed in situ environment has been possible within certain regions within calibration medium.
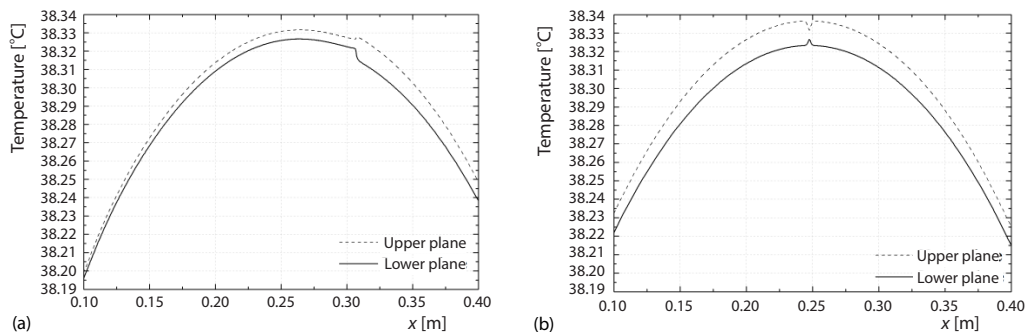

Figure 4. Simulated temperature profiles in direction; (a) parallel and (b) perpendicular to the reference probe, related to the example shown in fig. 3

According to the performed simulations for different boundary conditions and temperatures, therefore one can define zones in the calibration medium where the temperature difference between the reference probe and sensors under calibration is less than a pre-defined limit, for example, 0.05 °C. Then, within these zones, locations of the sensors and corresponding corrections to the surface temperature distribution can be straightforwardly determined and corrected or, alternatively, included to the calibration uncertainty budget as the contribution due to a non-uniformity of temperature calibration medium.

Validation

The validation of the proposed *in situ* calibration environment was performed by comparing the data obtained by this calibration to those obtained by placing the same sensors in a calibration bath environment. A digital thermometer FLUKE, model 1502a, with a platinum resistance Pt-100 probe, model 5618B, was used as the reference thermometer in the both calibration cases. A JULABO bath, model F34-ED, was used in the stirred liquid, as well as in the in situ calibration. The multichannel voltmeter used for readouts of the twenty thermocouples under calibration was KEITLEY, model 2700, with a multiplexer slot, model 7700, connected to a PC with an in-house developed software for data acquisition and display [8]. The thermocouples are labelled from T01 to T05 (Group 1), T06 to T10 (Group 2), T11 to T15 (Group 3), and T16 to T20 (Group 4). For the *in situ* calibration groups of thermocouples 1 and 3 were located at the silicone rubber surface closer to the cooler and below the reference probe and the remaining two groups were on the opposite side, above the reference probe, as depicted in fig. 2. All the groups were positioned at distance of about 5 cm from the platinum probe's sensitive volume and, within each group, individual thermocouples were
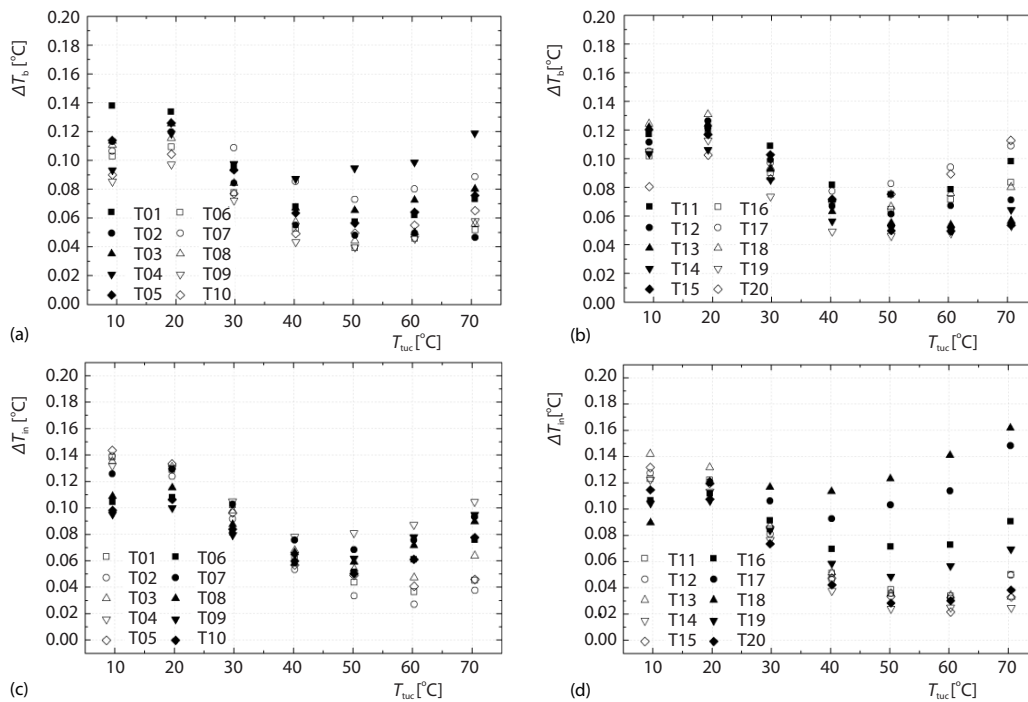

Figure 5. Temperature corrections of thermocouples calibrated in the bath (the upper row) and the *in situ* calibration environment (the lower row)

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distributed in such manner that their leads did not cross and their ends were at a mutual distance of less than 1 cm.

The results of calibration obtained in the temperature range from 10-70 °C in the calibration bath and the proposed in situ calibration environment are shown in fig. 5. The results are presented as temperature corrections, ΔT_b and ΔT_{in}, *i. e.*, differences between real temperature (measured by the reference thermometer) and related indicating temperatures T_{tuc} (measured by the applied sensors connected to the voltmeter), in the bath and *in situ*, respectively.

According to these data, temperature corrections show similar behavior in the entire temperature range, deviating ±0.07 °C or less, as compared to their corresponding group averages, and seemingly no relation among the groups or the values of individual thermocouples can be established. It can be noticed that the temperature corrections for thermocouples in Group 4 (T16 to T20) differ from the other groups, but these differences are still small in comparison to measurement uncertainty values for thermocouples.

An extract of calibration results for the twenty thermocouples when the thermostat temperature was set to 40 °C is given in tab. 1, where T_b and σ_{Tb} are the mean steady-state temperature and its standard deviation for the calibration in the bath, while T_{in} and σ_{Tin} are the same for the *in situ* calibration. The mean temperatures have been corrected to the related reference temperature, $\Delta T_b = T_{ref,b} - T_b$ and $\Delta T_{in} = T_{ref,in} - T_{in}$, and the differences between the calibration corrections, $\Delta T_b - \Delta T_{in}$, have been computed. Finally, for comparison purposes, the extended total calibration uncertainties ($k = 2$) in the bath calibration, U_{Tb}, are given in the last column. All the values in tab. 1 are in °C.

Table 1. Calibration results obtained at 40 °C

Label		T_b	σ_{Tb}	ΔT_b	T_{in}	σ_{Tin}	ΔT_{in}	$\Delta T_b - \Delta T_{in}$	U_{Tb}
Group 1	T01	40.27	0.006	0.07	40.22	0.007	0.06	0.01	0.12
	T02	40.29	0.005	0.05	40.23	0.006	0.05	0.00	0.08
	T03	40.27	0.004	0.07	40.22	0.006	0.07	0.00	0.11
	T04	40.25	0.004	0.09	40.20	0.005	0.08	0.01	0.11
	T05	40.28	0.006	0.06	40.22	0.006	0.06	0.00	0.12
Group 2	T06	40.29	0.005	0.05	40.22	0.005	0.06	-0.01	0.11
	T07	40.26	0.003	0.08	40.20	0.006	0.08	0.00	0.11
	T08	40.28	0.007	0.06	40.21	0.006	0.07	-0.01	0.12
	T09	40.30	0.004	0.04	40.22	0.006	0.06	-0.02	0.11
	T10	40.29	0.008	0.05	40.22	0.005	0.06	0.02	0.12
Group 3	T11	40.26	0.004	0.08	40.23	0.006	0.05	0.03	0.11
	T12	40.27	0.008	0.07	40.23	0.007	0.05	0.02	0.08
	T13	40.28	0.005	0.06	40.23	0.005	0.05	0.01	0.11
	T14	40.28	0.006	0.06	40.24	0.005	0.04	0.02	0.11
	T15	40.27	0.004	0.07	40.23	0.008	0.05	0.02	0.11
Group 4	T16	40.27	0.008	0.07	40.21	0.005	0.07	0.00	0.12
	T17	40.26	0.006	0.08	40.19	0.005	0.09	−0.01	0.12
	T18	40.27	0.004	0.07	40.17	0.006	0.11	−0.04	0.11
	T19	40.29	0.004	0.05	40.22	0.005	0.06	−0.01	0.11
	T20	40.27	0.006	0.07	40.24	0.007	0.04	0.03	0.12

The differences $\Delta T_b - \Delta T_{in}$ for all twenty thermocouples at all calibration temperatures are shown in fig. 6. According to them, the differences did not exceed the maximum extended calibration uncertainty in the bath calibration, *i. e.*, ±0.12 °C, thus justifying the application of the

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proposed *in situ* calibration environment. On the other hand, calibration corrections obviously depended on the calibration temperature, fig. 5, and that might be related to various sources, such as the temperature distribution at the contact surfaces of the cold plates, longitudinal thermal profile and homogeneity of thermocouple wires, thermal contact resistance, and heat exchange with surroundings. In order to distinguish the influence of these sources, which was not under the scope of this work, an analysis of repeatability and reproducibility of an extended number of measurements under various boundary and temperature conditions should be additionally performed.
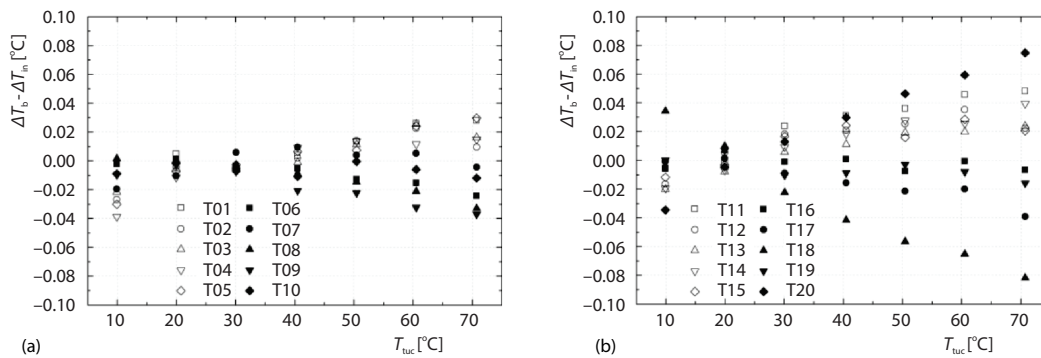

Figure 6. Differences of the two temperature calibration corrections

Conclusion

By developing custom calibration environment for an *in situ* temperature calibration of the GHP apparatus, greater reliability of temperature measurements in terms of both accuracy and traceability has been achieved. According to the presented validation, the differences in temperature calibration corrections, which include the effects of mounting the temperature sensors into the apparatus, as well as the properties of calibration medium, are comparable to the best possible calibration uncertainty of the applied temperature sensors obtained by the conventional method that include the properties of calibration medium only. As the result, the proposed in situ calibration represents a better solution for the calibration of temperature sensors used in the described GHP apparatus and, in general, offers an example for possible developments of similar solutions in other apparatuses for thermal conductivity measurement.

Nomenclature

A – cross-sectional area of the main heater [m^2]
d – mean specimen thickness, [m]
k – coverage factor for expanded uncertainty
P – main heater heat flux, [W]
T01-T20 – labels for temperature sensors
T_b – temperature of sensors, measured in the bath, mean value, [°C]
$T_{ref,b}$ – temperature of reference probe, measured in bath, mean value, [°C]
T_{in} – temperature measured *in situ*, mean value, [°C]
$T_{ref,in}$ – temperature of reference probe, measured *in situ*, mean value, [°C]
T_{tuc} – temperature of the sensor under calibration, mean value [°C]

ΔT – temperature difference between the *hot* and *cold* surfaces of the specimens, [°C]
ΔT_b – corrections to T_b according to $T_{ref,b}$, [°C]
ΔT_{in} – corrections to T_{in} according to $T_{ref,in}$, [°C]
U_{Tb} – expanded calibration uncertainty ($k = 2$) in bath, [°C]
x, y – planar co-ordinates, [m]

Greek symbols

λ_{eff} – effective thermal conductivity, [$Wm^{-1}\,°C^{-1}$]
σ_{Tkb} – standard deviaton of T_b, [°C]
σ_{Tin} – standard deviaton of T_{in}, [°C]

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